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Stress Relaxation and Creep Recovery Tests Performed on Wheat Kernels Versus Doughs: Influence of Glutenins on Rheological and Quality Properties[1]

J. D. C. Figueroa[2] and Z. J. E. Hernández
Centro de Investigación y de Estudios Avanzados
Querétaro, Mexico

P. Rayas-Duarte
Robert M. Kerr Food & Agricultural Products Center, Oklahoma State University
Stillwater, OK, U.S.A.

R. J. Peña
Centro Internacional de Mejoramiento de Maíz y Trigo
El Batán, Mexico

Researchers have shown that the overall elastic-plastic deformation of agricultural products can be explained by the theory of viscoelasticity. The method employed to measure viscoelasticity compares the behavior of the product under load with the response of an idealized viscoelastic or rheological model. To perform this type of analysis it is necessary to have some knowledge of stress distribution in loaded materials. Such information provides an explanation of how the structure of a material is able to support applied loads and explains the mechanisms of failure.

Stress Relaxation Tests

In stress relaxation tests, a constant strain is applied, and the stress required to maintain the deformation is measured as a function of time. The measured relaxation time indicates how fast the material dissipates stress after sudden deformation. Because mechanical damage to agricultural products usually results from compressive loads, more data can be found for compression tests than for tensile tests (17). When a stress relaxation test is performed, different behaviors are observed: ideal elastic materials do not immediately begin to relax, whereas ideal viscous materials instantaneously begin to relax. Viscoelastic solids gradually relax and reach an equilibrium stress greater than zero, whereas the residual stress for viscoelastic fluids is zero (4,21). Stress relaxation can be examined using models to assist in describing what is happening to structures at the molecular level.

To describe the viscoelastic behavior of a wheat kernel, a massless, mechanical, generalized Maxwell model was used to describe the behavior at the molecular level. This model is composed of springs (representing ideal solids, they account for the pure elastic behavior of viscoelastic materials) and dashpots (representing ideal fluids, they account for the viscous behavior of viscoelastic materials) combined in many different ways that can explain the viscoelastic behavior (Fig. 1).

Early research by Matsumoto et al. (16), who worked with structural relaxation of dough using an extensigraph, indicated that the behavior of dough could not be represented by a single Maxwell model, but rather by a large number of models distributed among a range of relaxation times between 1 and 1,000 sec.

In this study, the model consisted of four elements with a spring in parallel (Fig. 1). Under the application of constant strain or deformation, the viscoelastic function exhibited by the model is described by the following exponential equation:

$$\sigma(t) = \sigma_1 e^{-t/\tau_1} + \sigma_2 e^{-t/\tau_2} + \sigma_3 e^{-t/\tau_3} + \sigma_4 e^{-t/\tau_4} + \sigma_0 \quad \text{(eq. 1)}$$

$$\text{and, } \tau_i = \eta_i/\sigma_i \quad \text{(eq. 2)}$$

where σ = stress, t = time, τ = relaxation time, and η = viscosity coefficient.

A set of wheat genotypes representing wide variability in grain hardness and gluten quality-related parameters was used to determine the relationship between the viscoelastic properties of wheat kernels and the physicochemical and rheological parameters of dough and breadmaking quality.

Analysis of data from stress relaxation experiments on wheat kernels suggests two relaxation mechanisms dissipate applied stress (Fig. 2): one at a shorter time of 1 to 10 sec (τ_1 and τ_2) and one at a longer time of ≈50 to 450 sec (τ_3 and τ_4). Several researchers have reported similar relaxation times in wheat kernels (6,7), barley kernels (13,20), gluten (1,12), and wheat dough (15,16) using different instruments.

Wheat Kernel and Dough Properties and Breadmaking Quality

Viscoelasticity and Physicochemical Characteristics. Wheat kernels with slow (long) relaxation times, especially τ_2, τ_3, and τ_4, were positively correlated with better breadmaking quality compared with short relaxation times, except for ash content, which had a highly significant negative correlation. Many researchers studying dough and gluten characteristics have indicated that a slower relaxation time was associated with good baking

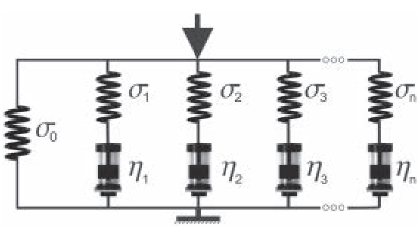

Fig. 1. Representation of a generalized Maxwell model consisting of a single spring constant (σ_0) in parallel with Maxwell models composed of dashpots (η_1 to η_n), to account for viscous behavior, and springs (relaxation constant σ_1 to σ_n), to account for elastic behavior. (Adapted from Hernández-Estrada et al. [9])

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[2] Corresponding author. Centro de Investigación y de Estudios Avanzados, Libramiento Norponiente 2000, Fracc. Real de Juriquilla, Querétaro, C.P. 76230 Qro., Mexico. E-mail: jfigueroa@qro.cinvestav.mx

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quality. For gluten, rapid relaxation times were associated with small polymer molecules that relax rapidly, whereas longer relaxation times were associated with high molecular weight (HMW) polymers (1,5).

Rheological Properties and Breadmaking Quality. There were some significant and highly linear correlation coefficients between specific breadmaking quality descriptors and the viscoelastic properties of wheat kernels. A plot of the generalized Maxwell model for good breadmaking quality and soft wheat kernels shows good separation among profiles for different wheat types (Fig. 3). Deformation of soft wheat kernels (poor breadmaking quality) requires less stress compared with harder wheat kernels (good breadmaking quality) (19). In addition to differences in σ_0 (Fig. 3), another important feature is that a quasi equilibrium state was reached at ≈180–200 sec at a moisture content of 12%.

Relaxation times of 40 to 60 sec were associated with quality parameters and rheological properties of wheat kernels; meanwhile, shorter relaxation times (1–10 sec) were correlated with mechanical properties of kernels and dough mixing times. To corroborate this information, the first derivative of the stress relaxation curves obtained directly from intact wheat kernels is presented. The curve shown in Figure 4 resembles the curve for dough-mixing properties obtained using a farinograph. The derivative curve generated by the stress-relaxation test suggests that relaxation times τ_1 and τ_2 (≈50 sec) are related to structure reformation mainly due to hydrogen and other noncovalent bonding; farinograms show that dough formation (explained by covalent bonds, such as disulfide bonds, as well as noncovalent bonds) is a function of mixing time. Relaxation times τ_3 and τ_4 (ranging from >50 to 1,000 sec) can be measures of quality, similar to dough formation properties shown in farinograms.

Dough Versus Rheological Properties and Breadmaking Quality. Li et al. (12) reported two relaxation processes for gluten, one at <1 sec and another at 10–1,000 sec, which indicated that in gluten this behavior corresponds to a network structure attributable to entanglements and physical cross-links of polymers. In the wheat kernel, gluten proteins are intrinsically disordered proteins with limited regular secondary structure elements. Thus, the majority of gluten proteins exist as randomly oriented chains with hydrogen bond interactions between water and amino acid side chains. In dough these proteins are stretched by tension during mixing into a network. A large number of interactions between the chains as a result of the alignment of the molecules leads to an increase in dough strength. This is probably the reason that the relaxation time required for dough, as reported by Li et al. (12), is shorter than τ_1 and longer than τ_4 observed for wheat kernels. We investigated the stress relaxation of dough samples using the same compression technique used with kernels. Figure 5 shows the equation and plot describing the location of the relaxation time of the dough at 0.45 sec (first decay), 3.2 sec (second decay), and 24 sec (third decay), at which time the dough reached a quasi steady state, which was similar to the values reported by Li et al. (12) of <1 to 10 sec of relaxation.

Wheat Kernel Viscoelasticity, Stress Relaxation, and Hardness

The data discussed above suggest that the specific Maxwell model elements describe mainly protein-like structures. The question is whether the specific proteins with high or low molecular weight glutenin subunit (HMW-GS or LMW-GS, respectively) compositions reported by other researchers (14,18) are associated with the elastic or plastic characteristics indicated by the Maxwell model elements.

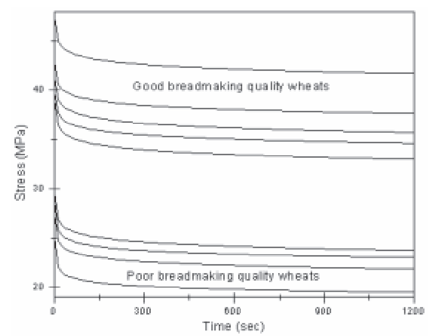


Fig. 3. Means of stress as a function of time for different wheat kernel types. (Adapted from Figueroa et al. [7])

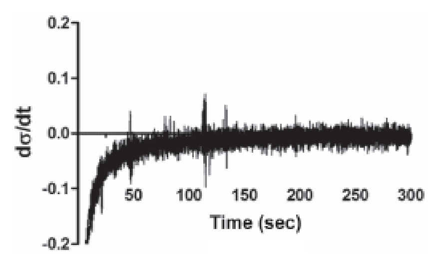


Fig. 4. First derivative of the stress-relaxation curve of a wheat kernel. σ = stress; *t* = time.

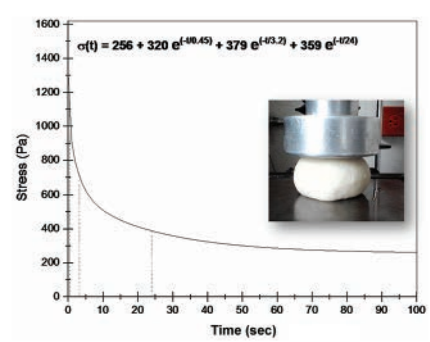


Fig. 5. Stress relaxation of wheat dough as a function of time obtained using a texture analyzer (TA.XTPlus) with 0.40 of strain. Relaxation times are indicated by vertical dotted lines. σ = stress; *t* = time.

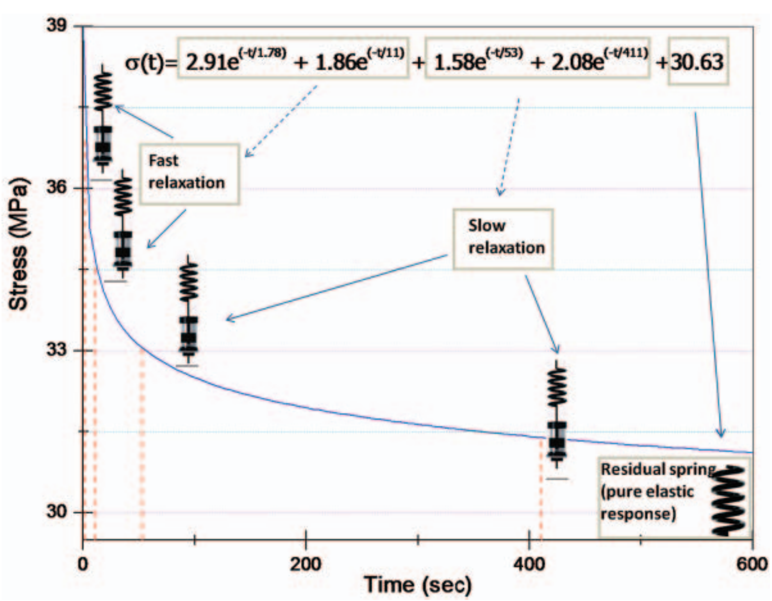


Fig. 2. Stress relaxation curve of a wheat kernel from regressed equation data that illustrate where to locate relaxation times (vertical dotted lines) and stresses (horizontal black marks). σ = stress; *t* = time.

It appears that not only is the total amount of protein important but also the amount and type of individual GS. However, despite considerable previous research into the rheological properties of the HMW-GS and LMW-GS of wheat, basic information and data on the mechanical and viscoelastic aspects of these GS in wheat kernels is very limited (8). Wheat kernels, like other grains, are subjected to a series of static and dynamic loads during harvesting, handling, transport, processing, storage, conditioning, and milling. Such loads cause significant damage to the kernels, which leads to a decrease in the intrinsic quality associated with specific HMW-GS and LMW-GS composition and an increase in susceptibility to deterioration during storage.

Hernández-Estrada et al. (9) reported correlations between the stress relaxation of wheat kernels, HMW-GS and LMW-GS composition, and breadmaking quality, suggesting that some interactions were stronger with high stresses in GS with good breadmaking quality, i.e., *Glu-A1* 2* and *Glu-A1* 5+10 compared with *Glu-A1*-null (Fig. 6). LMW-GS also have a very important influence on wheat quality, especially the *Glu-A3* and *Glu-B3* loci where the *Glu-A3 e* (null) alleles also showed short relaxation times (τ_3 and τ_4), suggesting they form poor protein interactions compared with *Glu-A3 b* and *Glu-B3 d* (Fig. 7).

The generalized Maxwell model suggests that the differences can primarily be explained as the effect of a combination of HMW-GS and LMW-GS stresses (Figs. 6 and 7), as well as other nonprotein polymers such as pentosans and β-glucans that act as plasticizers (viscous behavior) and dissipate stress in the kernel (9).

In addition to moisture content, which was kept constant in the sample wheat kernels, genotype and specific alleles in intact kernels and HMW-GS and LMW-GS appeared to be predominant factors affecting relaxation stress constants and times for wheat kernels.

Single spring stress (σ_0) affects the mechanical, rheological, and breadmaking properties of wheat kernels. If one is interested only in mechanical properties, it may be that determining spring stress related to hardness is enough; however, to examine viscoelastic properties other regressed coefficients need to be determined. If no mathematical software is available, a simple procedure can be used to screen grains and other biological materials. Force relaxation curves can be obtained by averaging the data (six observations) using simple plotters without applying mathematical models. This seems to be a practical alternative for rapidly selecting cereal cultivars in breeding programs using a nondestructive method. The selected kernels can then be planted, controlling the desired quality using the pedigree method.

Creep and Creep Recovery Tests

In creep tests stress is applied and held constant, and deformation is measured as a function of time. In a practical situation, creep is analogous to in-bin storage, where the lower layer of kernels is exposed to a dead load from a bed of kernels above (3). If the kernel layer is deep enough, some kernels at the bottom of the layer may break.

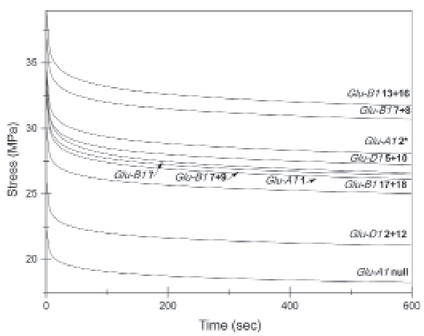


Fig. 6. Stress relaxation curves of wheat kernels with different high molecular weight glutenin subunit compositions.

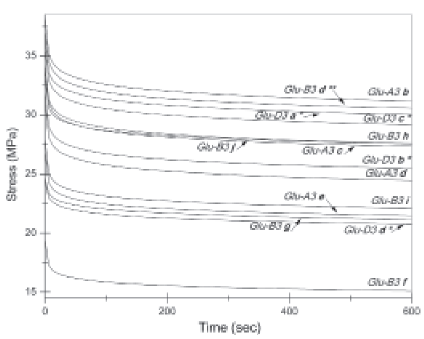


Fig. 7. Stress relaxation curves of wheat kernels with different low molecular weight glutenin subunit compositions. *Glu-A3 e* = null; *Glu-B3 j* = IB/IR rye translocation.

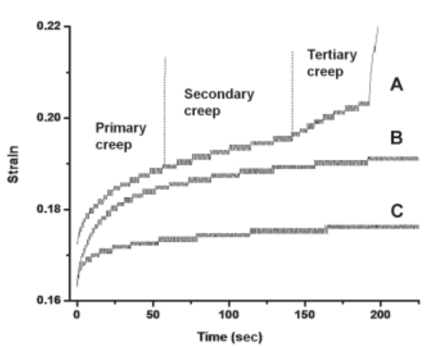


Fig. 8. Creep curves of wheat kernels with 12% moisture content and 25°C temperature. **A,** Wheat kernel sample with tertiary creep or broken kernel at 80 N; **B** and **C,** Curves with some secondary creep that reaches steady state.

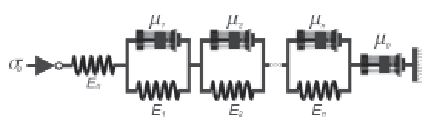
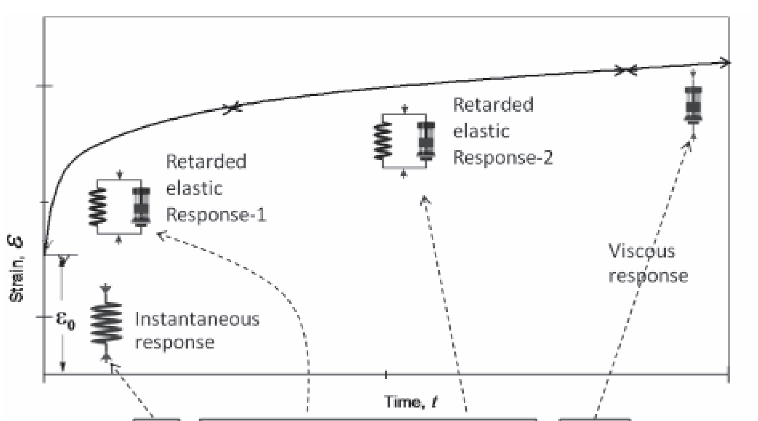


Fig. 9. Mechanical analogue representation of generalized Kelvin-Voigt model with six elements. σ = stress; *E* = elastic modulus; and μ = viscosity coefficient. (Adapted from Hernández-Estrada et al. [10])



$$\varepsilon(t)=\frac{\sigma_0}{E_0}+\frac{\sigma_0}{E_1}\left(1-e^{-t/\lambda_1}\right)+\frac{\sigma_0}{E_2}\left(1-e^{-t/\lambda_2}\right)+\frac{\sigma_0}{\mu_0}t$$

$$\lambda_i=\frac{\mu_i}{E_i}$$

$$D(t)=\frac{\varepsilon(t)}{\sigma_0}=D_0+D_1\left(1-e^{-t/\lambda_1}\right)+D_2\left(1-e^{-t/\lambda_2}\right)+\frac{1}{\mu_0}t$$

Fig. 10. Typical curve and mechanical representation of each component of the creep and compliance equation for compression stress. ε = strain; *t* = time; σ = stress; *E* = elastic modulus; μ = viscosity coefficient; *D* = compliance; and λ = retardation time.

Creep Conditions and Generalized Kelvin-Voigt Model. A typical creep curve (Fig. 8) may exhibit three regions: a primary creep in which the curve is concave down, a secondary creep in which deformation is proportional to time (steady state), and a tertiary creep in which deformation accelerates until creep rupture occurs. In many cases, the test terminates with the failure of the specimen. A mechanical analogue representing the generalized Kelvin-Voigt model is shown in Figure 9. Figure 10 shows that tertiary creep can be avoided by regulating the magnitude of the imposed stress.

Burger and Generalized Kelvin-Voigt Model. The viscoelastic behavior of wheat kernels was studied using mechanical analogues composed of springs and dashpots. The system is illustrated in Figure 9 and comprises a combination of Hookean (springs) and fluid (dashpots) bodies to describe the experimental data. The six element model (a spring in a series with a Kelvin-Voigt model) used in a creep test (10) can be described as shown in Figure 10 and the following equation:

$$\varepsilon(t)=\frac{\sigma_0}{E_0}+\frac{\sigma_0}{E_1}(1-e^{-t/\lambda_1})+\frac{\sigma_0}{E_2}(1-e^{-t/\lambda_2})+\frac{\sigma_0}{\mu_0}t \quad \text{(eq. 3)}$$

$$\text{and, } \lambda_1=\frac{\mu_1}{E_1};\ \lambda_2=\frac{\mu_2}{E_2}$$

where ε = strain, t = time, σ = stress, E = elastic modulus, λ = retardation time, and μ = viscosity coefficient.

In terms of uniaxial creep compliance (17,21)

$$D(t)=\frac{\varepsilon(t)}{\sigma_0}=D_0+D_1(1-e^{-t/\lambda_1})+D_2(1-e^{-t/\lambda_2})+\frac{t}{\mu_0} \quad \text{(eq. 4)}$$

where D = compliance, t = time, ε = strain, σ = stress, λ = retardation time, and μ = viscosity coefficient.

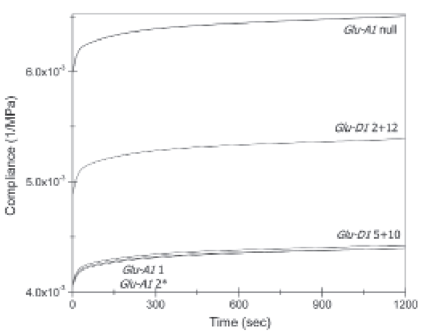


Fig. 11. Typical creep compliance curves calculated from averaged regressed values for wheat kernel samples with different high molecular weight glutenin subunit compositions. (Adapted from Hernández-Estrada et al. [10])

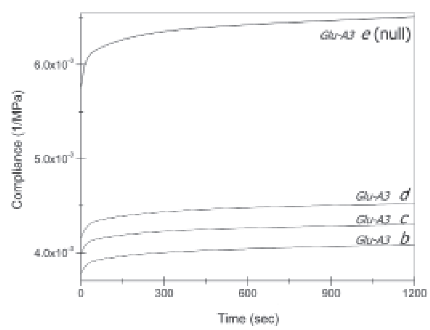


Fig. 12. Typical creep compliance curves calculated from averaged regressed values for wheat kernel samples with different low molecular weight glutenin subunit compositions. *Glu-A3 e* = null; *Glu-B3 j* = IB/IR rye translocation. Cultivars possessing *Glu-B3 f* and *Glu-D3 f* had poor viscoelastic performance due to the combination of *Glu-A3 e* silent and *Glu-A1* null, respectively. (Adapted from Hernández-Estrada et al. [10])

Effect of HMW-GS and LMW-GS Composition. Most of the wheat cultivars possessing good breadmaking quality alleles showed steady-state times of ≈300 sec under an applied stress of 38 MPa, whereas wheat kernels from poor breadmaking quality genotypes continued to deform (Figs. 11 and 12). This seems to be an advantage because creep evaluations can be completed in 4 to 5 min.

At the *Glu-A1* locus the null subunits presented lower moduli of elasticity compared with *Glu-A1* alleles 1 and 2* (Fig. 11). As expected the viscosity coefficients associated with retardation time (λ_1 and λ_2) were also lower. Also as expected, the *Glu-D1* locus showed higher elasticity moduli and higher viscosity values for the 5+10 alleles and lower values for compliances compared with samples containing the 2+12 allele.

Measurement of Creep Compliance of Wheat Kernels Versus Wheat Dough. Several authors have indicated that the nomenclature for creep compliance under uniaxial compression (or tension) should be $D(t)$, instead of $J(t)$ or $G(t)$, which are creep compliance under shear (2,17,21). Viscoelastic properties of doughs from near-isogenic wheat lines were measured for creep compliance under shear to evaluate compositional differences in HMW-GS (11). The study reported that 5+10 allele lines displayed higher viscosity and viscoelasticity (lower compliance) than 2+12 allele lines. Similar trends were

Table I. Wheat kernel creep measured using a texture analyzer[a]

Genotypic Group / Glutenin Subunit	Modulus of Elasticity (MPa) and Retardation Time (sec): E_0	E_1 (×10^3)	λ_1	E_2 (×10^3)	λ_2	Coefficient of Viscosity (MPa·sec): μ_0 (×10^7)	μ_1 (×10^5)	μ_2 (×10^6)	Compliance (1/MPa): D_0 (×10^{-3})	D_1 (×10^{-4})	D_2 (×10^{-4})
Glu-D1											
5+10	245 a	7.52 a	13.4 a	7.58 a	177 a	1.56 a	1.02 a	1.36 a	4.36 b	1.90 b	1.72 b
2+12	205 b	4.58 b	13.0 a	5.16 b	161 a	1.19 a	0.69 a	0.90 b	6.64 a	7.94 a	3.68 a

[a] Kernels were under constant compression at 70 N for 1,200 sec. Means followed by different letters within a column are significantly different ($P < 0.05$). E_0 = instantaneous modulus of elasticity; E_1 and E_2 = retarded elastic moduli at λ_i = ith retardation time; μ_0 = viscosity coefficient; μ_1 and μ_2 = viscosity coefficients; D_0 = instantaneous compliance; and D_1 and D_2 = retarded compliances.

Table II. Dough creep measured using a rheometer[a]

Baking Performance	Loaf Vol (cm^3)	Modulus of Elasticity (Pa) and Retardation Time (sec): G_0	G_1 (×10^3)	λ_1	G_2 (×10^3)	λ_2	Coefficient of Viscosity (Pa·sec): η_0 (×10^5)	η_1 (×10^3)	η_2 (×10^4)	Compliance (1/Pa): J_0 (×10^{-4})	J_1 (×10^{-4})	J_2 (×10^{-4})
Good	1,200	9.98 a	4.87 a	0.4 b	2.49 a	8.8 b	1.78 a	1.79 a	2.2 a	1.0 b	2.1 b	4.0 b
Poor	900	6.97 b	2.52 b	0.5 a	1.12 b	10.7 a	0.71 b	1.23 b	1.2 b	1.4 a	4.0 a	8.9 a
DMCT[b]		6.82	6.30	4.1	2.63	13.7	4.0	25	3.6	1.5	1.6	3.8

[a] Dough was under 100 Pa of shear stress for 100 sec of creep and 100 sec of recovery. Means followed by different letters within a column are significantly different ($P < 0.05$). G_0 = instantaneous modulus of elasticity; G_1 and G_2 = retarded elastic moduli at λ_i = ith retardation time; η_0 = viscosity coefficient; η_1 and η_2 = viscosity coefficients; J_0 = instantaneous compliance; and J_1 and J_2 = retarded compliances in creep phase.

[b] Dough measured using compression with a texture analyzer (TA.XT2); the appropriate nomenclature is E for modulus of elasticity and D for compliance.

observed for compliance of wheat kernels under uniaxial compression when evaluating the same HMW-GS studied by Hernández-Estrada et al. (10) (Table I). The data on wheat dough provided in Tables II and III showed creep and regressed value trends similar to those reported by Van Bockstaele et al. (22) and Lefebvre and Mahmoudi (11).

Kernel recovery was not estimated because the texture analyzers (TA.XT2 and TA.XTPlus) used required some modifications to perform the recovery test. When using a rheometer to measure the creep recovery of dough, the data were quite similar in both creep and creep recovery tests (Tables II and III; Figs. 13 and 14). The creep compliance values in Table I were obtained using wheat kernels. Tables II and III show similar tendencies regarding viscoelastic properties, although the magnitude of creep compliance values for wheat kernels was approximately six-fold higher than the values for dough.

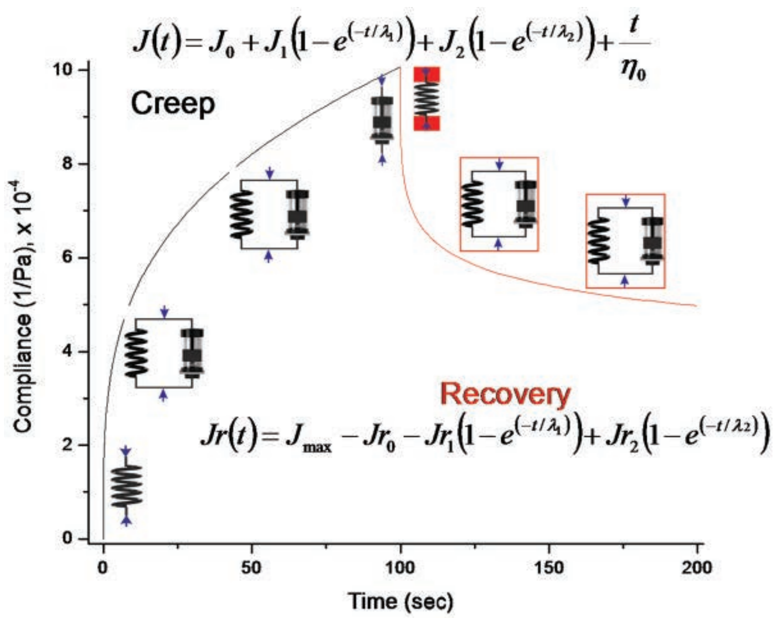


Fig. 13. Typical curve and mechanical representations of each component of the creep and compliance equation for shear stress of dough. J and Jr = compliance; t = time; λ = retardation time; and η = viscosity coefficient.

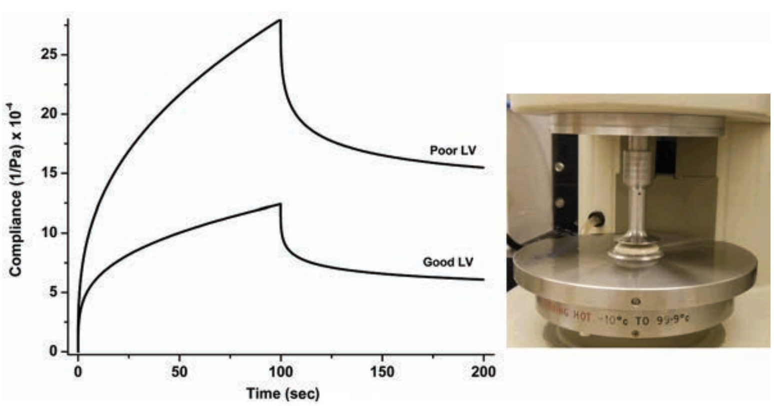


Fig. 14. Creep and creep recovery curves for wheat dough with different loaf volumes (LV).

Table III. Dough creep recovery measured using a rheometer[a]

Baking Performance	Loaf Vol (cm^3)	Modulus of Elasticity (Pa) and Retardation Time (sec)					Coefficient of Viscosity (Pa·sec)[b]		Compliance (1/Pa)		
		Gr_0	Gr_1 ($\times10^3$)	λr_1	Gr_2 ($\times10^3$)	λr_2	ηr_1 ($\times10^3$)	ηr_2 ($\times10^4$)	Jr_0 ($\times10^{-4}$)	Jr_1 ($\times10^{-4}$)	Jr_2 ($\times10^{-4}$)
Good	1,200	9.13 a	5.02 a	0.55 b	3.40 a	16.0 b	2.77 a	5.42 a	1.10 b	1.99 a	2.96 a
Poor	900	6.40 b	2.59 b	0.72 a	1.53 b	17.3 a	1.87 b	2.65 b	1.57 a	3.87 b	6.56 b

[a] Dough was under 100 Pa of shear stress for 100 sec of creep and 100 sec of recovery. Means followed by different letters within a column are significantly different ($P < 0.05$). Gr_0 = instantaneous modulus of elasticity; Gr_1 and Gr_2 = retarded elastic moduli at λr_i = ith retardation time; ηr_1 and ηr_2 = viscosity coefficients; Jr_0 = instantaneous compliance; and Jr_1 and Jr_2 = retarded compliances in recovery phase.

[b] The recovery model did not have ηr_0 ($\times10^7$).

Conclusions

We have summarized examples of fundamental measurements of viscoelasticity of wheat kernels and doughs using stress relaxation, creep, and creep recovery tests. Often, the only method for determining quality accepted by plant breeders, millers, and bakers is a baking test. Fundamental rheological tests are frequently applied for more basic studies, but their use is not widespread enough for the characterization of a large number of cultivars. In addition the relationships between fundamental rheology and baking quality have not been sufficiently examined.

The methods reported here offer an option for examining the responses of structures and separating them into groups that appear to be simple and related to fundamental properties. The fundamental rheological methods described are well-suited for the characterization of wheat kernels and functional dough properties. The methodology for testing wheat kernels is reliable, easy, rapid, and nondestructive. As a result, the characterization of individual kernels could be useful in breeding programs as well as the bread industry, complementing the information obtained from the characterization tests that are currently performed.

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Juan de Dios Figueroa works with the Bio-organic Materials Group at CINVESTAV-IPN (Center for Advanced Studies and Research of the National Polytechnic Institute) in Querétaro, Mexico. His areas of interest are the Maxwell (stress relaxation) and Kelvin-Voigt (creep) generalized models for studying viscoelastic properties and high-fiber, low-fat tortillas to prevent obesity, diabetes, and cardiovascular diseases. Juan has published more than 150 scientific articles, among them 21 patents. He developed dry nixtamalization, wet ecological nixtamalization, and ohmic processes among other technologies. In 2012 he received the Texture Technologies Quality Research Award for Best Presentation from AACC International. He received the Dios Yum Kaax Award at the 2012 4th International Nixtamalization Congress for the development of an ecological nixtamalization process to produce whole grain corn tortillas. Juan also received the First Place of Premio Alejandrina 2012 for participation in the elaboration of "intelligent nanoparticles for encapsulation, and controlled release of bioactive compounds with beneficial properties for human health." Juan is an AACCI member and can be reached at jfigueroa@qro.cinvestav.mx.

Zorba Josué Hernández Estrada is a Ph.D. student in materials science working with the Bio-organic Materials Group at CINVESTAV-IPN (Center for Advanced Studies and Research of the National Polytechnic Institute)in Querétaro, Mexico. His area of expertise is cereal rheology and technology, and his research is focused on the viscoelastic characterization of wheat grain and dough and its relation to functional quality. Zorba earned a master's degree in materials science from CINVESTAV investigating the "Effect of Microwave Radiation on the Physicochemical and Elastic Properties of Wheat Kernels." He has received scholarships from CONACYT (National Council of Science and Technology of Mexico). He has authored several publications on the use of stress relaxation and creep recovery models for evaluating the effects of glutenins of high and low molecular weight on the viscoelastic properties of wheat kernels and doughs and β-glucans in barley. Zorba is an AACCI member and can be reached at zhernandez@qro.cinvestav.mx or heeszorba@hotmail.com.

Patricia Rayas-Duarte is a professor and cereal chemist in the Department of Biochemistry and Molecular Biology with duties at the Robert M. Kerr Food & Agricultural Products Center at Oklahoma State University, where she explores the basic biochemical principles that govern the functionality of grains in industrial applications. Patricia's research focus is on evaluating specialized biochemical and rheological properties of gluten proteins and their role in key processing and quality parameters of interest to the baking industry. She is also interested in the improvement of methodologies for the interpretation and prediction of processing properties relevant to present demands of customers and processors. She is a graduate of the University of Nebraska-Lincoln (M.S. and Ph.D. degrees in food science and technology) and the Universidad de Sonora (B.S. degree). Patricia is an AACCI member and can be reached at pat.rayas_duarte@okstate.edu.

Roberto J. Peña is a cereal chemist with a bachelor's degree from the National University of Mexico, Mexico; a master's degree from Kansas State University, U.S.A.; and a Ph.D. degree from the University of Manitoba, Canada. Roberto is a principal scientist and head of the Wheat Chemistry and Quality Laboratory of CIMMYT (Centro Internacional de Mejoramiento de Maíz y Trigo) in El Batán, Mexico. Roberto's main area of research is improvement of grain compositional factors that determine wheat processing quality. He has spent more than 28 years with CIMMYT addressing wheat quality improvement related to the genetic control of key quality traits and the expression and stability of quality under diverse agroclimatic conditions. He has authored or co-authored more than 100 publications in refereed journals and book chapters.