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Chapter 12

Use of the Stress-Relaxation and Dynamic Tests to Evaluate the Viscoelastic Properties of Dough from Soft Wheat Cultivars

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1. Introduction

Wheat is the only cereal capable of produce strong and cohesive dough, qualities responsible for the wide variety of food products made from it. In Mexico, wheat is classified into five groups based on functionality; those wheat varieties in group three are called soft, and they are used in products such as cakes and cookies.

Wheat dough is classified as viscoelastic material (Faubion & Hoseney, 1990) which has elastic and viscous characteristics conferred by gluten. Gluten network is formed by hydrophobic interactions between water and protein polymers of the flour (mainly glutenins and gliadins). The nature of the glutenin mainly influences the functional properties of strength and elasticity, while the gliadin fraction provides extensibility and viscosity to the dough (Lu & Grant, 1999). Studies have been conducted in search of those factors that influence dough's viscoelastic properties to better understand the behavior of dough, so it can be handled and utilized properly.

Smith et al. (1970) utilized the dynamic test in a gluten-starch-water system and found that high protein content is reflected in high values of the storage module (G´) and loss module (G´´). Besides protein content, it has been found that the soluble protein fraction is a determinant factor for the rheological properties of dough by acting as a lubricating agent (Rouille et al., 2005). The importance of the total gliadin fraction in the structure of gluten was observed by Lee & Mulvaney (2003). Gupta et al. (1993) utilized an extensograph to evaluate doughs and found a high correlation between unextractable proteins and maximum resistance (Rmax), which led them to believe that a high molecular weight fraction of glutenin contributes to the resistance of the dough.

Another factor affecting the rheological properties of dough is starch. It has been seen that damage to starch and the presence of other minor components affect rheological properties (Dexter et al., 1994; Lynn & Stark, 1995). Campos et al. (1997) and Chiotelli et al. (2004) suggested that the rheological changes observed in dough subjected to low heating tests may be due to starch molecules present. Zeng et al. (1997) found that 80% of the variation in the viscosity of starch paste of 13 cultivars of wheat was due to the concentration of amylose and amylopectin, and Morris et al. (1997) observed that this variation is due to the presence of isoenzymes synthesized in relation to the starch granule. Georgopulus (2006) observed that the rheological properties of dough are affected more by the removal of the native lipids of the flour than for its gluten.

Rheological properties of wheat flour are determinants of the desired characteristics in the final product, as well as for the design of equipment and processes. Therefore, it is necessary to find reliable rheological tests that are a useful tool for characterizing dough of wheat flours (Safari-Ardi & Phan-Thien, 1998). Rheology of wheat dough is broad, and the tests utilized are diverse. Initially, empirical tests were more used than the fundamental tests, but disadvantages were seen such as: dependence on the instrument, the form and quantity of the sample utilized and lack of theoretical basis (Faubion & Hoseney, 1990), which led to the development of fundamental tests. The most important fundamental viscoelastic tests utilized for wheat dough are the force-deformation ratio, creep test, the dynamic test and stress-relaxation test (Mohsenin, 1978).

Recently, the dynamic test and the stress relaxation test have been used to characterize the dough viscoelasticity. Safari-Ardi & Phan-Thien (1998), utilizing the dynamic test observed that there were no differences among dough elaborated with different flours, probably due to the small deformation used (<1%). Therefore, it was decided to evaluate viscoelastic properties of dough applying a stress-relaxation test. In this technique, the dough is rapidly deformed at a predetermined level and the stress is measured over time, where deformations are >1% (Faubion & Hoseney, 1990; Mohsenin, 1978; Rao et al., 2000).

Several researches have utilized and recommended the stress-relaxation test (Lee and Mulvaney, 2003; Rao et al., 2000; Uthayakumuran et al., 2002; Wikström & Eliasson,, 1998; Yadav et al., 2005). Safari-Ardi et al. (1997) found when using the stress-relaxation test, it was possible to differentiate dough from distinct wheat flours, and demonstrated that besides the method being consistent, the data of the stress-relaxation test obtained at high amplitudes (1-15% deformation in $3x10^3$ s) were very precise (Safari-Ardi & Phan-Thien, 1998). Utilizing the stress-relaxation test, it has been found that the distribution of the quantity of protein (high molecular weight glutenin) and its molecular weights are related to the relaxation time (Rao et al., 2000; Uthayakumaran et al., 2002). In addition, it has been shown that moisture content and strain affect the relaxation characteristics of dough by doing them no linear (Yadav et al., 2005). Similarly, Smith et al. (1970) observed that the relaxation time increases with mixing, and Rao et al., (2000) concluded that this parameter is less for strong dough than that moderately strong.

The study of viscoelastic properties of flour from different wheat cultivars has been reported for different authors (Larsson & Eliasson, 1996a, 1996b; Wikström & Eliasson, 1998; Safari-Ardi & Phan-Thien, 1997, 1998; Van Bockstaele et al., 2008), nevertheless, none of them specifically study the viscoelastic characteristics of soft wheat, and this could be important for the cookies and baked industry. The purpose of this research was to evaluate the rheological properties of soft wheat dough from some cultivars in order to determine in the most appropriate manner its final use and in particular its quality. The viscoelastic properties of soft wheat dough were evaluated by applying the stress-relaxation and the dynamic test, and relate them with empirical rheological measurements and physicochemical characteristics.

2. Experimental procedure

2.1. Raw material

Samples of soft wheat from four cultivars were used: Barcenas, Cortazar, Salamanca and Saturno. Samples were obtained in the Central part of Mexico (El Bajío Zone), and they were sent to the Departamento de Investigación y Posgrado en Alimentos from the University of Sonora at Hermosillo Sonora, México, where was carried out this study.

2.2. Flours elaboration

Wheat samples of the four cultivars were cleaned (Blount/Ferrell-Ross, model M2BC), and placed in plastic bags, which were stored at refrigeration temperature (4°C) until use. Wheat samples were conditioned based on the international approved method number 26-95 (American Asociation of Cereal Chemists, [A.A.C.C], 2000), at a moisture content of 16%, utilizing a conditioner (Chopin Instruments, Villeneuve-La-Garenne, France). Samples were allowed to stand for a period of 24 h before preparation of flours. The conditioned samples were milled using the approved method number 26-10 (A.A.C.C., 2000), and an experimental mill (Brabender, model Quadrumat Senior, South Hackensack, NJ). For maturation, flours were allowed to stand for a period of 15 days.

2.3. Flours quality analysis

For proximate chemical analysis of flours, the official methods of the A.A.C.C. International (2000) were used, and the following determinations were made: protein content (approved method number 46-13) using a nitrogen analyzer (LECO, model FP-528, MI, USA), and the protein factor was N x 5.7; ash content (approved method number 08-03); and moisture content (approved method number 44-40).

Wet gluten content was determined utilizing the approved method number 38-11 (A.A.C.C., 2000) and the apparatus Glutomatic (Falling Number, model 2100, Huddinge, Sweden). Sedimentation volume was determined using the approved method number 56-61A (A.A.C.C., 2000). The falling number was measured utilizing approved method number 56-81b (A.A.C.C., 2000) and the apparatus Falling Number (model 1400, Huddinge, Sweden).

2.4. Rheological tests

2.4.1. Empirical test

The farinographic parameters of water absorption, stability and time of development were obtained based on the farinographic approved method number 54-21 (A.A.C.C., 2000), and utilizing a farinograph (Brabender Instruments, type 810143, South Hackensack, NJ).

To characterized flour-water dough for extensibility and resistance to extension, it was used approved method number 54-30A (A.A.C.C., 2000), and the Alveograph (Chopin Instruments, Villeneuve-La-Garenne, France).

2.4.2. Fundamental test

Sample preparation. Dough was prepared with 100 g of flour of each cultivar, and adding distilled water corresponding to the absorption obtained from the farinograph. Flour was placed and mixed in a mixer with 300 g capacity (National MFG, Lincoln, NE, USA) without the distilled water. Then it was mixed for 1 min with one afterward; the dough was allowed to stand for 30 min in a proofing chamber (National MFG, Lincoln, NE, USA) with a controlled environment (30°C, 95% relative humidity). Next, samples of dough weighing individually 2.7 g were placed in plastic bag, hermetically sealed, and left at room temperature (25°C) until use in the rheometer.

Dynamic method. Oscillatory measurements were conducted at 0.1% strain within the linear viscoelastic regime (previously carried out), over a frequency range of 0.1 to 100 rad/s. Frequency sweep experiments were conducted in a Rheometer (Rheometrics Scientific, model RSF III, Piscataway, NJ, USA). The rheometer was equipped with 25-mm diameter parallel plates that were maintained at 25° C. A dough sample of 2.7 g was round by hand and placed between the plates of the rheometer. Sample was gently flattened to fit the plate geometry using a Teflon-coated spatula. The upper plate was lowered to a fixed gap between plates of 2 mm. The exposed edges of dough were trimmed, and to avoid drying, petroleum jelly was injected around the plates. Sample was allowed to rest for a period of 20 min before the test. The rheometer was run in the frequency scanning in oscillatory mode, and at a controlled temperature. The viscoelastic parameters determined were: the storage modulus (G′, Pa), the loss modulus (G′′, Pa) and tangent of phase angle (δ=G′′/G′). These parameters were obtained using the software analysis program of the rheometer (RSI Orchestrator, Rheometrics Scientific) (Magaña-Barajas et al., 2011).

Stress-relaxation test. Sample of dough was prepared and placed in the rheometer in manner similar to that utilized in the dynamic test. After placing the sample in the rheometer, it was allowed to rest for a period of 20 min before the test. Based on preliminary tests and some researches (Li et al., 2003; Rao et al., 2000; and Safari-Ardi & Phan-Thien 1998), stress relxation was measured at intervals of 0.1 s applying a 15% shear strain for 30 min at a controlled temperature of 25ºC operating in shear stress mode. The parameters determined were maximum stress at 15% strain (G_0) and the relaxation time (τ).

2.5. Experimental design and statistical analysis

A completely randomized experimental design was performed, where the factor was the wheat cultivar: (Barcenas, Cortazar, Salamanca and Saturno). For the purpose of determining the effect of the factor on the different determinations, analysis of variance (ANOVA) was used with a 95% confidence interval. In addition, simple correlations (r) between different determinations were carried out. ANOVA was carried out with the Statistical Analytical System Software (SAS Institute, Inc. Cary, NC, 2002).

3. Results and discussion

3.1. Flours quality

Analysis of variance showed that the wheat cultivar affected significantly ($p<0.05$) the proximate chemical analysis of flours. Table 1 presents mean values for protein, moisture, ash and wet gluten content, sedimentation volume, and falling number each flour from the soft wheat cultivars. Cultivar Barcenas showed the highest values of protein content, ash content, wet gluten, sedimentation volume and falling number. Moisture content was similar for all the cultivars.

In general, it has been observed that wheat cultivar affected the physical and chemical properties of flours (Lin & Czuchajowska, 1997). In some investigations (Carrillo et al., 1990; Chiotelli et al., 2004; Yamamoto et al., 1996), marked physicochemical differences have been observed in cultivars of soft wheat, and that falling number has been seen to influence the rheological properties of dough. Sedimentation volume indicates the quality of the proteins presents; in hard wheat the close values to 70 mL indicates high baking quality (A.A.C.C., 2000; Pomeranz, 1988). Falling number values greater than 400 s have been reported that the amylolytic activity is low. Therefore, wheat is considered that has been exposed to low rainfall during growth up to harvest time, consequently resulting in healthy grains (Carcea et al., 2006; Pomeranz, 1988). In this study, for all the flours, the Falling number was greater than 400 s, which indicate that flours were obtained from healthy soft wheat cultivars.

3.2. Rheological tests

3.2.1. Empirical test

Table 2 shows the mean values for water absorption, stability and developing time each flour. Salamanca had the highest values of water absorption and developing time, while cultivar Barcenas had the greatest stability. Values for water absorption, stability and developing time are directly proportional with protein content. In addition, water absorption is directly proportional to the diameter of the cookie (Bloksma, 1990; Bloksma & Bushuk, 1988; Farrand, 1969; Yamamoto et al., 1996).

Table 2 shows also the alveographic parameters for flours from the soft wheat cultivars. Cultivar Salamanca showed the highest values of W, P and P/L. These high values probably were due to its protein content, a factor that also results in high water absorption (Unbehend et al., 2004; Yamamoto et al., 1996).

Cultivar	Moisture Content (%)	Protein Content[a] (%)	Ash Content[a] (%)	Wet Gluten[a] (%)	Sedimentation Volume[a] (mL)	Falling Number (s)
Barcenas	14.08 ± 0.33[b]	11.81 ± 0.50	0.84 ± 0.18	34.87 ± 4.82	32.03 ± 1.62	476.60 ± 31.73
Cortazar	14.06 ± 0.23	10.58 ± 0.31	0.69 ± 0.21	31.77 ± 2.47	22.04 ± 1.18	422.51 ± 9.51
Salamanca	14.10 ± 0.45	11.27 ± 0.49	0.76 ± 0.18	33.00 ± 1.02	28.92 ± 8.66	449.67 ± 27.54
Saturno	14.05 ± 0.61	10.32 ± 0.41	0.55 ± 0.04	30.86 ± 2.46	25.46 ± 1.94	409.98 ± 13.85

[a] Dry basis [b] Standard Deviation

Table 1. Physicochemical characteristics of flour from soft wheat cultivars

Cultivar	Farinograph			Alveograph		
	Water Absorption (%)	Stability (min)	Developing Time (min)	W[b] (10^{-4}J)	P[c] (mm H_20)	P/L[d]
Barcenas	54.04 ± 3.84[a]	4.28 ± 2.01	3.70 ± 0.66	114.75 ± 14.25	49.76 ± 5.04	0.46 ± 0.11
Cortazar	56.16 ± 1.48	1.96 ± 0.59	3.39 ± 0.39	85.50 ± 10.22	52.63 ± 7.54	0.57 ± 0.05
Salamanca	56.39 ± 1.70	3.75 ± 2.14	4.54 ± 1.67	139.62 ± 66.88	67.01 ± 16.41	0.70 ± 0.15
Saturno	53.28 ± 1.51	3.51 ± 0.68	3.14 ± 0.54	106.33 ± 17.58	48.69 ± 4.13	0.52 ± 0.10

[a] Standard Deviation; [b]W= Deformation energy of dough; [c]P= Maximum overpressure, tenacity; [d]P/L= Curve configuration ratio, extensibility

Table 2. Rheological characteristics of flour from soft wheat cultivars

3.2.2. Fundamental test

Two techniques were used to characterize the viscoelasticity of dough from soft wheat cultivars: a) the dynamic method; and b) the stress-relaxation test, which were performed in a rheometer.

Dynamich method. The viscoelastic properties of dough obtained from the soft wheat cultivars were the storage modulus (G′), loss modulus (G′′) and tangent of angle phase (Tan δ) (Figures 1, 2 y 3, respectively). To evaluate if there were statistically differences among the viscoelastic properties of dough from the different wheat cultivars, an analysis of variance was performed on the G′, G′′ and Tan δ values at a frequency of 5 rad/s, where the dough viscoelasticity was in the linear viscoelastic domain (Figures 1, 2 and 3). According to the ANOVA, G′, G′′ and Tan δ obtained with the dynamic test at a frequency of 5 rad/s had similar values ($p>0.05$), indicating that dough from the soft wheat cultivars were in the same values of viscoelasticity (Table 3). For all the soft wheat cultivars, the G′ values were greater than those for G′′, and Tan δ was greater than 0.50.

This indicates that in soft wheat cultivars dough prevailed the viscous behavior over the elastic behavior.

Figures 1 and 2 show the dependence of G´ and G´´ with frequency (Hibber & Wallace, 1966). In both figures, values of G´ and G´´ are increasing, which could indicate that dough hardness increase, and it could be due to content of gluten (Chiotelli et al., 2004) and protein quality, resulting in the gluten of wheat dough not as weak as expected (since soft wheat flour contains a weak gluten). Several authors (Huebner et al., 1999; Yamamoto et al., 1996) agreed that content and quality of protein and gluten content are factors affecting positively these viscoelastic parameters.

In Figure 3 is presented the behavior of Tan δ with frequency for dough from all soft wheat cultivars. Initially, Tan δ shows a slight decrease; at intermediate levels (1-10 rad/s) values at a given frequency are similar; and at high frequencies this parameter shows the same initial phenomenon that at low frequency values. Tan δ for all the soft wheat cultivars is higher than 0.5, which indicates that viscous behavior is higher than the elastic behavior.

Stress-relaxation test. Dough was subjected to a deformations much greater than that in the dynamic test (>1%). The parameters evaluated from this test were the initial (maximum) stress at 15% strain (G_0), and the relaxation time (τ). This last parameter is related to the process of flow occurring when dough is relaxed, and is defined as the time required for the force to fall 1/e times, or by 36.8% of its original value (Smith et al., 1970).

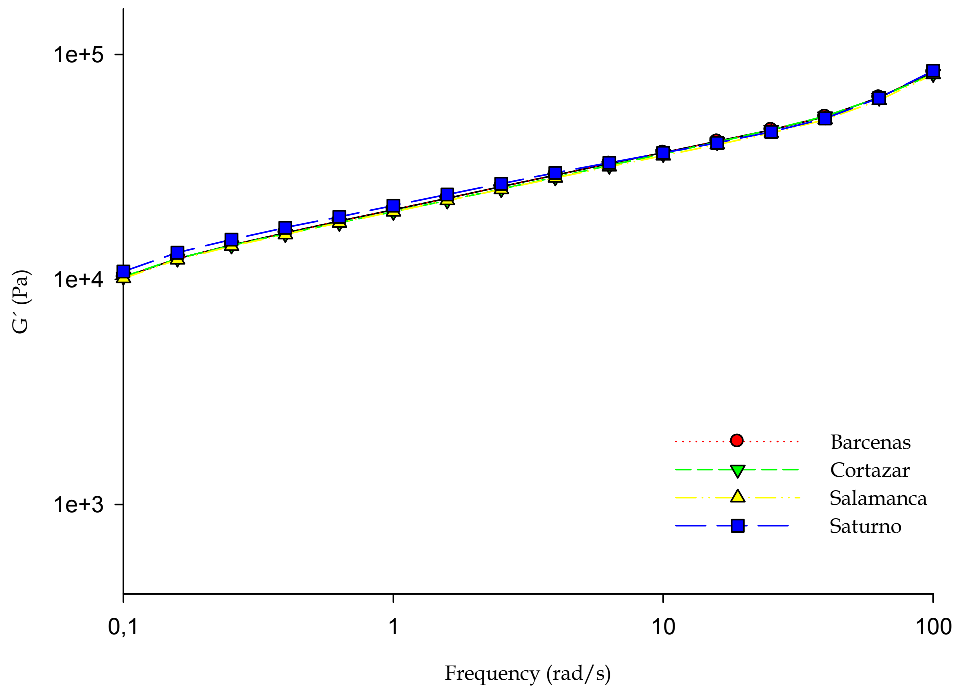

Figure 1. Storage modulus (G´) evaluated with the dynamic test for dough made of soft wheat cultivars.

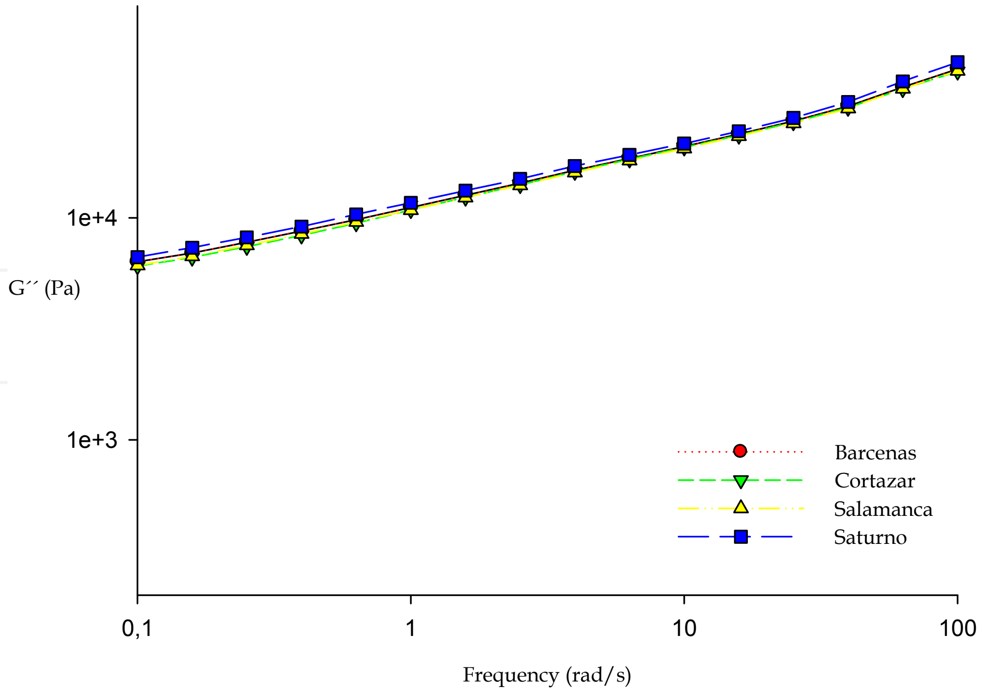

Figure 2. Storage modulus (G´) evaluated with the dynamic test for dough made of soft wheat cultivars.

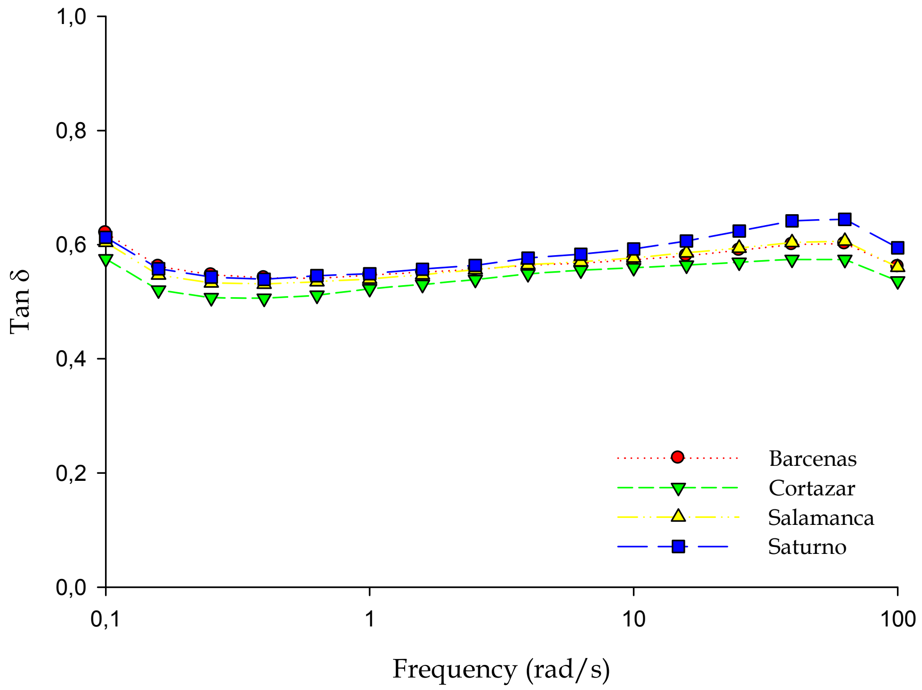

Figure 3. Tangent of angle phase (Tan δ) evaluated with the dynamic test for dough made of soft wheat cultivars.

An ANOVA was carried out to see if there were differences between the viscoelastic properties of the different soft wheat cultivars determined with the stress-relaxation test. It was shown that the soft wheat cultivar affected only to τ ($p<0.05$).

Figure 4 shows the behavior and mean values for the viscoelastic parameters of τ and initial maximum stress (G_0) of dough from the soft wheat cultivars, obtained in the stress-relaxation test. Initially, dough was subjected a high deformation (15%) yielding in response a high initial stress, and as time progress, dough relaxed and stress diminished. It is observed that all curves present a single maximum stress coinciding with Rao et al. (2000). Li et al (2003) found that relaxation process occur in two steps: the first, correspond at the increase of distribution of protein polymers short chain (gliadin and glutenins of low molecular weight). The second peak corresponds at the protein polymers of large chain like glutenins of high molecular weight. In this case, the soft wheat cultivars evaluated had slow protein content (9.9%) and the content of protein polymers short chain (44.22% of gliandins, of total) is highest than the content of protein polymers short chain (data not showed), changing the ratio glutenins/gliadins.

Cultivar	Dynamic Test[g]			Stress Relaxation Test[h]	
	G'^{b} (Pa)	G''^{c} (Pa)	Tan δ^{d}	G_0^{e} (Pa)	τ^{f} (s)
Barcenas	29080 ± 3384[a]	16378 ± 1926	0.56 ± 0.03	510.42 ± 119.61	0.36 ± 0.07
Cortazar	28445 ± 11489	15621 ± 1114	0.54 ± 0.02	466.00 ± 55.61	0.38 ± 0.02
Salamanca	28246 ± 4174	16007 ± 2714	0.56 ± 0.01	484.28 ± 130.50	0.33 ± 0.07
Saturno	29750 ± 2967	17121 ± 1524	0.057 ± 0.02	462.45 ± 56.50	0.41 ± 0.02

[a] Standard Deviation; [b] G´: Storage modulus; [c]G´´: Loss modulus; [d]Tan δ: Tangent of phase angle; [e]G_0: Initial maximum stress; τ^f: Relaxation time; [g]: the viscoelastic parameters were evaluated at frequency of 5 rad/s; [h]: the viscoelastic parameters were measured at 15% shear strain

Table 3. Viscoelastic characteristics of dough made from soft wheat cultivars evaluated with the dynamic and stress relaxation tests

In Table 3 is presented G_0 and τ values for all flours. The wheat cultivar Saturno had the highest value of τ (0.41 s). The high values of τ indicates that the recovery of deformed dough structure is slow, reflecting a weak gluten. That wheat cultivar presents the lowest values of moisture, protein content, ash content, wet gluten and falling number, which probably caused a lesser strength in gluten. This could explain the strength of the dough. Dough of cultivars Salamanca and Barcenas had the lowest τ values (0.33 y 0.36 s, respectively). These varieties have high values in almost all the physicochemical properties. Low values of τ correspond at strong gluten. For this reason when dough is subject to a deformation quickly recovers its original form. In general, variation of values of τ (range 0.33 s to 0.41 s) was probably attributed at the content and type of protein, and moisture content of the wheat cultivar. This has been observed in several investigations, where it has been found that moisture and gluten properties (such as gluten strength) affect the parameters of this test (Fu et al., 1997; Larsson & Eliasson, 1996a; Li et al., 2003; Yadav et al., 2005).

Results obtained show an inverse relationship between the values of G_0 and τ. Applying a high strain to dough with three-dimensional network structure of strong gluten, values of G_0 are obtained almost immediately after deformation.

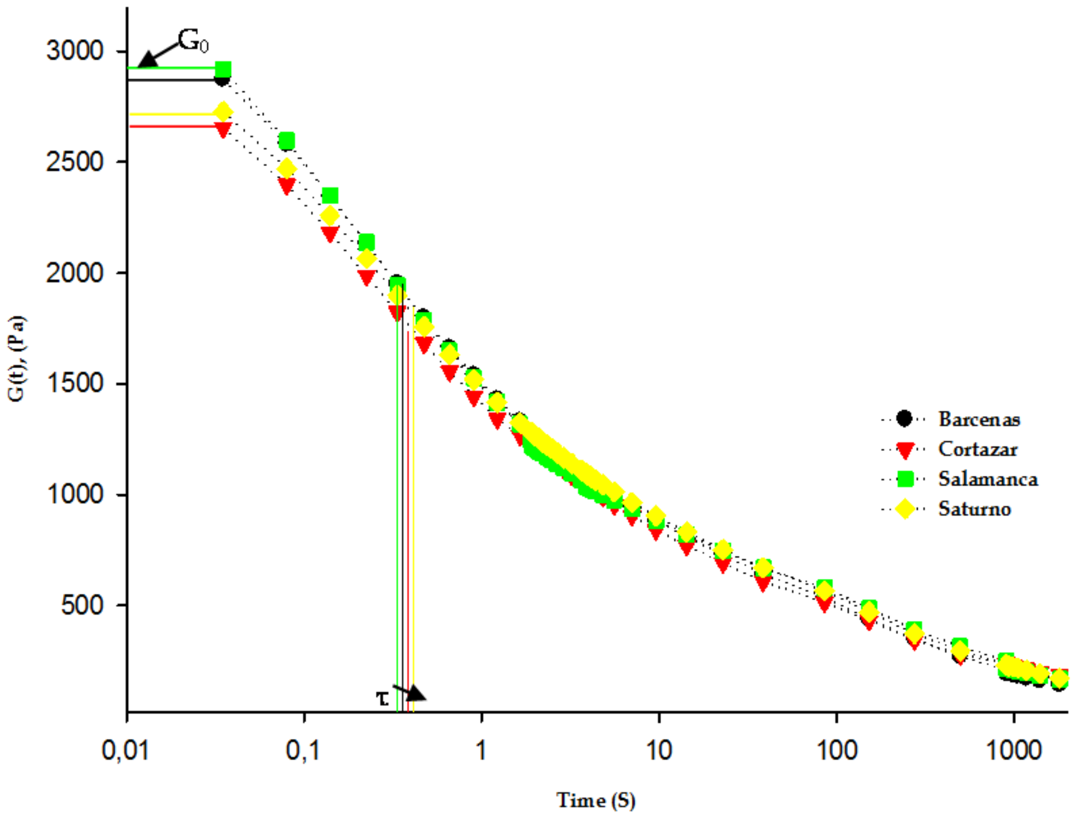

Figure 4. Relaxation modulus, G (t) vs time of dough from soft wheat cultivars, at 15% of deformation. τ: relaxation time.

The relaxation time was the parameter showing the greatest difference among the soft wheat cultivars. However, the differences obtained in τ among dough from the soft wheat cultivars were less than estimated. This despite to the range of low protein content of the soft wheat cultivars (10 to 12%, approximately), and besides, the fraction of gliadin and glutenin were of low molecular weight and approximately similar in all the samples (70% gliadin and 30% glutenin of low molecular weight, data not shown). This could indicate that tests used to evaluate the viscoelasticity in dough were high sensibility being capable to characterize dough from different soft wheat cultivars. Characterization and differentiation of dough was better with the stress-relaxation test than with the dynamic test, coinciding with others authors (Lee and Mulvaney, 2003; Rao et al., 2000; Uthayakumuran et al., 2002; Wikström & Eliasson,, 1998; Yadav et al., 2005; Safari-Ardi et al., 1997).

3.2.3. Correlations between viscoelastic characteristics and other determinations

To determine if there were significant relations among the viscoelastic properties of dough from soft wheat cultivars, and physicochemical and rheological evaluations in flours, simple correlations *(r)* were performed.

Five significant correlations were found among the viscoelastic characteristics and physicochemical determinations. Regarding to the stress-relaxation test, there were four highly significant correlations (p<0.01). The relaxation time (τ) showed a strong correlation with developing time (farinograph) (r=-0.959), which indicates indirectly a high protein content and strong gluten, resulting in a rapid response upon deformation, as a consequence lower τ values. G_0 showed a strong correlation with protein (r=0.984), which could be reflected in the greater strength of gluten, thereby a high resistance upon deformation. A second correlation of G_0 was with wet gluten (r=0.987), which probably is due to the fact that a greater amount of gluten causes greater resistance to deformation, and this is in agreement the study carried out by (Li et al., (2003). Finally, the last correlation of G_0 was with falling number (r=0.986).

The dynamic test only yielded a highly significant negative correlation ($p<0.01$) for the viscoelastic parameter of G´ (storage modulus) with water absorption (r=-0.975), which could be explained by the fact that high water absorption can give indirect evidence of a high moisture content, which in turn has a negative effect on G´ (Faubion & Hoseney, 1990). No correlations were found between the stress-relaxation test and dynamic test utilized to determine the viscoelastic properties of the dough of soft wheat cultivars.

4. Conclusions

The viscoelastic characterization of dough made from flours of soft wheat cultivars was achieved by fundamental tests. The soft wheat cultivar showed to be a determinant factor in all characteristics of the flour. Apparently, the strength of the gluten network is critical in the rheological and viscoelastic characteristics of dough. All the cultivars presents more viscous behavior than elastic behavior (Tan δ>0.5). Wheat varieties evaluated showed a range τ of between 0.33 to 0.41 s.

The stress-relaxation test was better than the dynamic test in differentiating the viscoelastic characteristics of the soft wheat dough. This is confirmed by the highest number of correlations relative to the stress relaxation test, compared with the dynamic method. In our case, τ offers more information than that of G_0, making it an important parameter for characterizing dough. Parameters most affecting τ was content and quality of the protein moisture content and falling number in flours. The stress-relaxation test is a simple and rapid method to perform viscoelastic evaluations, and it is believed to be a technique suitable for the characterization and differentiation of viscoelastic characteristic among dough made from soft wheat cultivars, which is important for determining the final use.

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