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Viscoelastic properties of tablets from Osborne fractions, pentosans, flour and bread evaluated by creep tests

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A b s t r a c t. Little attention has been given to the influence of non-gluten components on the viscoelastic properties of wheat flour dough, bread making process and their products. The aim of this study was to evaluate by creep tests the viscoelastic properties of tablets manufactured from Osborne solubility fractions (globulins, gliadins, glutenins, albumins and residue), pentosans, flour and bread. Hard and soft wheat cultivars were used to prepare the reconstituted tablets. Sintered tablets (except flour and bread) showed similar values to those obtained from the sum of the regression coefficients of the fractions. Gliadins and albumins accounted for about 54% of the total elasticity. Gliadins contributed with almost half of the total viscosity (45.7%), and showed the highest value for the viscosity coefficient of the viscous element. When the effect of dilution was evaluated, the residue showed the highest instantaneous elastic modulus (788.2 MPa). Retardation times of the first element (λ_1 ~ 3.5 s) were about 10 times lower than the second element (λ_2 ~ 39.3 s). The analysis of compliance of data corrected by protein content in flour showed that the residue fraction presented the highest values. An important contribution of non-gluten components (starch, albumins and globulins) on the viscoelastic performance of sintered tablets from Osborne fractions, flour and bread was found.

K e y w o r d s: viscoelasticity, creep test, Osborne fractions, fluor, bread

INTRODUCTION

Different investigations have reported that the unique viscoelastic properties of wheat grains reside primarily in the gluten forming storage proteins of its endosperm (Gianibelli *et al*., 2001; Greenwood and Ewart, 1975; Lindsay and Skerritt, 1999). Many parameters such as the quality of flour protein, the amount of gluten, proportion of gluten proteins, and the type of subunits present in the flour are evaluated to predict the quality of dough and bakery products (Saxena *et al*., 1997). However, little attention has been given to the evaluation and influence of non-gluten components, such as pentosans and starch, on the viscoelastic properties of dough, the bread making process and their products.

D'Appolonia and Gilles (1971) mentioned that pentosans are closely associated with proteins, especially with the gluten fraction of wheat flour, hence these components could be related with the viscoelastic properties during the bread making process. For indirect measurement of wheat properties associated with gluten and non-gluten components, Hernández-Estrada *et al.* (2012) proposed the creep

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test and the use of the generalised Kelvin-Voight model methodology that allowed the separation of rheological parameters like elastic, viscoelastic and viscous flow characteristics. The plastic work of wheat kernels may be related to water, lipids, pentosans and other carbohydrates that are important for dough extensibility (Figueroa *et al*., 2009). Non-gluten components of dough, like albumins, globulins, starch and pentosans, showed lower elastic moduli than dough (Hernández-Estrada *et al*., 2014, 2017). In general, viscoelastic behaviour of wheat kernels and their doughs are most likely explained by the interactions of all of their gluten and non-gluten components that form unique systems (Ponce-García *et al*., 2016). Thus, the study of viscoelastic properties of other wheat components besides gluten alone justifies their evaluation using different methods that would complement the body of knowledge in this field.

Figueroa *et al*. (2016) indicated that starch granules, water soluble pentosans and non-gluten components may not be considered as inert filler, since it has been clearly shown that those components are also responsible for the viscoelastic behaviour of sintered tablets made from wheat products such as flour, dough and bread.

The aim of this study was to evaluate the viscoelastic properties of sintered tablets of proteins obtained from Osborne solubility fractions (globulins, gliadins, glutenins, albumins and residue), pentosans, flour and bread from two wheat classes, evaluated by creep tests.

MATERIALS AND METHODS

Samples of four cultivars of hard red spring wheat (HRS): Len, Butte, Era and Coteau, and of two cultivars of soft red wheat (SRW): Adder and Fairfield, were tested in this study.

The grain samples were tempered at room temperature on the lab bench in plastic bottles. After measuring the moisture of the wheat with NIR using a FOSS Infratec 1 241 grain analyser (FOSS, Eden Prairie, MN), additional water was added to bring the moisture to 12%. The samples were shaken every hour for 3 h and then rested at room temperature for approximately 16 h prior to milling. Tempered wheat samples were milled using a Brabender Quadrumat Jr. (C.W. Brabender, South Hackensack, NJ) laboratory mill. Straight grade flour was blended and screened through an 84 SS sieve to remove foreign material. The bread was prepared using a 2 h fermentation schedule to avoid overfermentation (AACCI, 2011). Bread loaves were sliced to 10 mm thick and placed in a single layer on wire drying racks. The bread was dried in two stages – 1) overnight on the laboratory bench at approximately 25 °C and 35-40% relative humidity, followed by grinding in a food chopper, and 2) freeze dried (Labconco Freezone 4.5, serial 061162977S, Kansas, Missouri) during 72 h at -53°C and 0.050 mBar, to reduce the moisture content to about 1 %.

Solvent solubility extraction was conducted according to the modified Osborne fractionation procedure of Chen and Bushuk (1970) in the wheat flour. Samples of flour with NaCl (0.5 M) were stirred and centrifuged, the supernatant (albumins and globulins) was dialysed with water to precipitate the globulin fraction. The pellet left in the tube (sediment) was mixed with 70% aqueous ethanol, stirred and centrifuged; the gliadin fraction was obtained in the supernatant by dialysis to remove ethanol. The pellet left on the tube was stirred with acetic acid (0.05 M) and centrifuged, the supernatant contained the acid soluble glutenin fraction. The pellet contained the residue, mostly starch containing acid insoluble glutenin. All the Osborne protein fractionation was carried out at 4°C, and all the centrifugations were at 5 000 x *g* for 30 min.

Fractions were freeze dried (Labconco Freezone 4.5, serial 061162977S, Kansas, Missouri) during 72 h, at -53°C and 0.050 mBar, and stored in tightly closed containers at 5°C until needed for analysis (approximately two weeks). Water soluble pentosans (WSP) were calculated by difference using the recovery of the fractions (Figueroa *et al*., 2016).

Tablets of 4.2 mm height and 8.07 mm diameter (contact area of 51.52 mm^2) were sintered from Osborne protein fractions, flour and bread. For each tablet, 300 mg of sample was weighed into a 10 ml beaker sealed with parafilm and placed in a controlled temperature chamber at 20°C for 2 h. The tablet-forming die of hardened steel had an inside diameter of 7.95 mm, and a length of 31.69 mm. Two hardened steel dowels were used to apply the pressure necessary to form the tablets. The lower dowel (5.54 mm long) was inserted into a loose-fitting cylinder, 31.69 mm high, and the die was placed on top of this cylinder with the dowel of 43.23 mm long positioned into the die. The sample was transferred into the die, the upper dowel placed into the lower die and the set positioned onto a table with a hydraulic press (Trupper 501, 50 t capacity). The load on the die was gradually increased to reach 25 t and maintained for 5 min before removing the tablet from the die. Reconstituted tablets were also prepared, containing the same percentage (w/w) of the fractions obtained. Six tablets were obtained for each fraction.

In creep experiments, the material is subjected to dead loads under constant strain and the deformation as a function of time is observed (Mohsenin, 1986). Once the tablets were obtained and before the test began, the height and diameter were measured using a caliper (Model CD-6 CS, Mitutoyo Corp., Japan) in order to calculate the applied strain and the contact area of each tablet.

The method used to measure viscoelastic properties by creep test was according to Hernández-Estrada *et al.* (2012) with some modifications. Briefly, before the test, 4 tablets with about 4% moisture content were placed in a controlled temperature chamber at 25°C for 2 h to reduce variance in the results. A Texture Analyzer (Texture Technologies

Corp. TA-XT2) with a 10 mm diameter stainless steel probe (TA-510) was used to measure the tablets deformation. The tablets were loaded in compression until reaching 70 N with a constant loading rate of 0.1 mm s^{-1}. Load (N) and displacement (mm) data were acquired at 10 points/s, the stress was held constant for 300 s, and the displacement data was used to calculate strain dividing the deformation by the initial height of the tablets. The test was performed once on each tablet to obtain four curves and data were extracted for calculations.

Mechanical models consisting of springs and dashpots are used to explain and interpret the rheological behaviour of linear viscoelastic materials. The spring obeys the Hooke law and the dashpot the Newton law. In the Kelvin-Voigt model, the elements are forced to move together at constant rate (Mohsenin, 1986), a three elements model that follows the Eq. (1):

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

where: $\varepsilon(t)$ is the strain (%) as a function of time t (s), σ_0 is the applied stress, E_0 (MPa) is the elastic modulus of the Hookean body; the retardation time ($\lambda_1=\mu_1/E_1$) that in the case of creep is (1-1/e) or about 63% of the time for total strain; E_1 is the elastic modulus and μ_1 (MPa s^{-1}) is the viscous coefficient of the dashpot in the Kelvin-Voigt element.

A four-element Burger model resulted of a combination of the Maxwell and Kelvin-Voigt models and is expressed in Eq. (2):

$$\varepsilon(t) = \frac{\sigma_0}{E_0} + \frac{\sigma_0}{E_1}\left(1 - e^{-t/\lambda_1}\right) + \frac{\sigma_0}{\mu_0}t. \quad (2)$$

The first term of Eq. (2) represents the instantaneous elastic deformation of the sample, the second term is related to the combined effect of viscoelasticity, and the third term expresses the viscous effect and the viscosity coefficient (μ_0, MPa s^{-1}).

According to Mohsenin (1986), to obtain a 6-element model, another Kelvin-Voigt unit must be added to the Burger model (Fig. 1).

To obtain the results in terms of uniaxial creep compliance, the equation suggested by Hernández-Estrada *et al.* (2012) was applied (Eq. (3):

$$D(t) = \frac{\varepsilon(t)}{\sigma_0} = D_0 + D_1\left(1 - e^{-t/\lambda_1}\right) + D_2\left(1 - e^{-t/\lambda_2}\right) + \frac{t}{\mu_0}, \quad (3)$$

where: ε – strain (%), t – time (s), σ – stress (MPa), λ – retardation time (s), and D – compliance (1/Pa), D_0 – instantaneous compliance or compliance at zero time (1/Pa), D_1, D_2 – compliance of 1st and 2nd retarded elastic element (1/Pa).

Analysis of variance (ANOVA) was performed using the General Linear Model procedure of SAS Statistical Software, version 9.3. Multiple comparison of the means was performed using the Duncan Multiple Range test at $\alpha=0.05$ level. Nonlinear regression analyses were performed with OriginPro 9 (OriginLab Corporation, Northampton, MA, USA).

RESULTS AND DISCUSSION

The evaluated protein fractions reached a *quasi*-steady state at 100 s after the application of constant stress. Usually, creep test performed on intact wheat kernels required more time (up to 200-1200 s) to reach the *quasi* equilibrium. The coefficients obtained by the creep curves fitted with the generalised Kelvin-Voigt model with six elements showed that gliadins and albumins represent about 54% of the total elasticity, however, gliadins showed the highest elastic modulus (E_0) among all the evaluated fractions (Table 1). Glutenins contributed with 17% of the total elasticity (E %) showing an E_0 of 1391.2 MPa, significantly higher than those reported by Hernández-Estrada *et al*. (2012). Those authors found an average value of 242 MPa for the elastic moduli of HMW-GS and LMW-GS from thirty-four wheat lines,

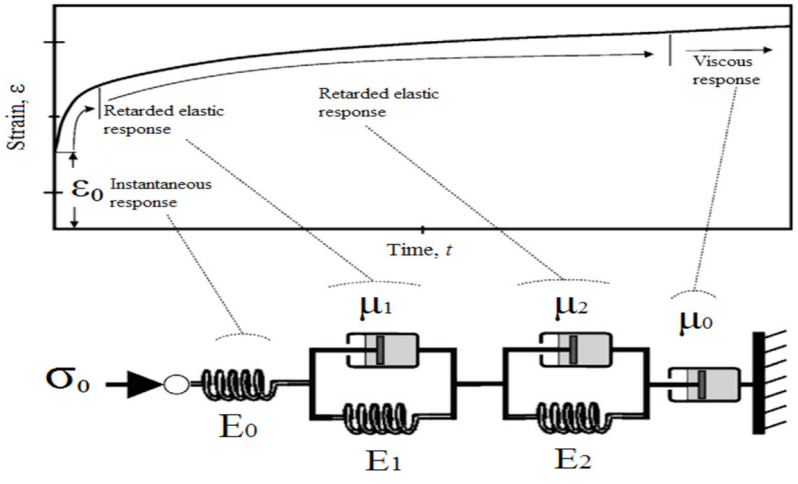

Fig. 1. Analogous mechanical representation of the generalised Kelvin-Voigt model with 6 elements (Hernández-Estrada *et al*., 2012).

T a b l e 1. Viscoelastic properties of Osborne solubility fractions, water soluble pentosans, flour and bread sintered tablets produced from hard red spring and soft red winter wheat cultivars

Sample	Modulus of elasticity and retardation time							Coefficient of viscosity			Compliance		
	E (%)	E_0	E_1	λ_1 (s)	E_2 (MPa)	λ_2 (s)	μ (%)	μ_0	μ_1	μ_2	D_0	D_1	D_2
		(MPa)						(MPa s^{-1})			(1/MPa)		
Albumins	23.60	1411.2b	$1.39x10^5$ab	3.47a	$9.87x10^4$b	42.00a	21.12	$1.04x10^9$b	$4.92x10^5$ab	$4.22x10^6$ba	$7.19x10^{-4}$cb	$7.43x10^{-6}$dc	$1.05x10^{-5}$c
Globulins	8.85	1235.6c	$5.11x10^4$d	4.14a	$3.74x10^4$d	39.04a	6.11	$3.01x10^8$c	$2.03x10^5$c	$1.44x10^6$c	$8.09x10^{-4}$b	$1.97x10^{-5}$a	$2.67x10^{-5}$a
Gliadins	31.05	1572.6a	$1.78x10^5$a	3.34a	$1.35x10^5$a	39.43a	45.73	$2.25x10^9$a	$5.19x10^5$a	$4.77x10^6$a	$6.39x10^{-4}$c	$6.21x10^{-6}$d	$7.71x10^{-6}$c
Glutenins	17.03	1391.2cb	$1.02x10^5$bc	3.74a	$6.92x10^4$c	40.74a	15.52	$7.62x10^8$cb	$3.08x10^5$bc	2.73x106bc	$7.39x10^{-4}$cb	$1.20x10^{-5}$bc	$1.63x10^{-5}$b
Residue[b]	9.85	937.4d	$5.49x10^4$d	3.69a	$4.40x10^4$d	39.95a	5.96	$2.92x10^8$c	2.24x105c	$1.81x10^6$c	$1.08x10^{-3}$a	$2.01x10^{-5}$a	$2.48x10^{-5}$a
Pentosansc	9.59	1256.3cb	$6.14x10^4$dc	3.09a	$3.45x10^4$d	34.53a	5.56	$2.75x10^8$c	$2.31x10^5$c	$1.44x10^6$c	$8.04x10^{-4}$b	$1.65x10^{-5}$ba	$2.91x10^{-5}$a
Average		1300.7	$5.86x10^5$	3.57	$6.98x10^4$	39.28		$8.20x10^8$	$3.29x10^5$	$2.73x10^6$	$7.98x10^{-4}$	$1.36x10^{-5}$	$1.91x10^{-5}$
All fractions[d]		1294.7	$7.24x10^4$	5.34	$5.50x10^4$	69.70		$3.82x10^9$	$4.01x10^5$	$3.84x10^6$	$7.85x10^{-4}$	$1.45x10^{-5}$	$1.92x10^{-5}$
Starch	18.64	1160.95	$1.19x10^5$	4.29	$6.87x10^4$	43.72	12.97	$6.37x10^8$	$5.15x10^5$	$3.00x10^6$	$8.65x10^{-4}$	$8.57x10^{-6}$	$1.46x10^{-5}$
Non-gluten	51.89						38.75						

a – Means (n= 6) followed by the same letter in the same column are not significant different at $p < 0.05$; b – residue is mainly composed by starch 68%, protein 4.4% and water insoluble pentosans about 3% (Jelaca and Hlynka, 1971); c – water soluble pentosans; d – sintered tablets include the percentage in weight of all fractions including water soluble pentosans; d – E is expressed as a percent of each fraction, where $E = E_0 + E_1 + E_2$; e – μ is expressed as a percent of each fraction, where $\mu = \mu_0 + \mu_1 + \mu_2$.

this value being lower than that found in this study. Perhaps there was an additive effect of the modulus from the different glutenin subunits that was expressed in the complete fraction. However, when starch was evaluated separately, higher values were showed for the instantaneous elastic moduli, similar to those obtained by albumins and gliadins. Staniforth and Patel (1989) evaluated the creep compliance behaviour of direct compression excipients from different materials, suggesting that starch undergoes a high degree of elastic deformation at different stress levels.

The retarded modulus of elasticity of the second element (E_2) showed lower values in almost all fractions when compared with E_1 (Table 1). Gliadins showed the highest values for both moduli (E_1 and E_2). Retardation times λ_1 (~ 3.5 s) were lower than λ_2 (~ 39.3 s), but no significant differences were detected among the fractions. Shorter retardation times were detected in all Osborne fractions and water soluble pentosans, when compared with those reported by Figueroa *et al.* (2011) and Hernandez-Estrada *et al*. (2012) on intact wheat kernels and glutenin subunits, respectively. The latter means that differences between materials can be measured using this methodology, applying rheological models that detect differences among samples. Thus, wheat proteins behave in a different way depending of their molecular structure, which could be related with the quality parameters of their products.

Dobraszczyk and Morgenstern (2003), using the results obtained by Bloksma and Bushuk (1988), described the experimental measurements for stress relaxation tests in different doughs. The curves did not show the exponential decay typical for a single relaxation time, indicating a wide range of relaxation times. Their results indicated that molecular mechanisms are involved for the relaxation process and deformation processes within dough, which can be related to the wide molecular weight distribution of gluten.

In this study, gliadins contributed with almost half of the total viscosity (45.7%), and showed the highest value for the viscosity coefficient of the viscous element (μ_0, dashpot). With respect to the first and second retarded elastic deformation elements (μ_1 and μ_2), albumins and gliadins showed the highest values (Table 1). The viscosity coefficients displayed wide variation, there were reductions by factors of 1000 for μ_1, and 100 for μ_2. Gliadins showed the highest values for μ_0, μ_1, and μ_2, which describes the plasticiser effect of gliadins and its function as a viscous material promoting the extensibility of gluten (Belton, 1999). Plastic components seem to be more related to non-gluten factors (Maucher *et al*., 2009). Ponce-García *et al.* (2016) noted that some low molecular weight glutenin subunits (LMW-GS), gliadins, lipids, pentosans and other carbohydrates act as plasticisers for dough extensibility. It was remarkable that the albumin fraction also showed higher viscosity coefficients (μ_1, μ_2), significantly contributing to the viscosity of the system. Glutenins showed 1/3 of the viscosity when compared with gliadins, in fact, the glutenin fraction showed similar values for E % and μ %.

The residue showed the highest instantaneous compliance (D_0). When D_1 and D_2 (compliance of the first and second retarded elastic elements) were evaluated, globulins, residue and water soluble pentosans presented the highest values (Table 1). According to Hernández *et al.* (2012), compliances obtained from creep test in kernels are correlated to some quality parameters such as SDS, SDS index, mixing time, alveograph deformation energy of dough, tenacity-extensibility ratio and tenacity.

The estimated parameters of the Kelvin-Voigt model for creep test coefficients from the Osborne solubility fractions and water soluble pentosans were corrected by weight (Table 2). The correction was made in order to compare the parameters and to observe the dilution factor effect that could exist among the fractions. When the extraction was performed, the residue showed the highest weight, as expected (86.1%), of all the fractions, followed by albumins and gliadins. It can be seen that the sum of gliadins and glutenins accounted for only 6.9% from the total flour weight.

In sintered tablets, gliadins, albumins and glutenins showed the highest instantaneous elastic moduli (Table 1). Nevertheless, when the effect of dilution was evaluated (Table 2), the residue showed the highest instantaneous elastic modulus E_0 (788.2 MPa), with similar values in Tables 1 and 2, perhaps by the largest proportion obtained on the Osborne fractionation. In addition, the residue fraction presented the highest values of elastic moduli (E_1 and E_2) when compared with the other fractions. In creep-compliance curves, starch shows a region representing a higher elastic deformation during compression, represented by spring stretching in the Maxwell unit (Stainforth and Patel, 1989). The dough strength has been attributed to glutenin fractions (Khatkar and Schofield, 1997), however, in their study the water effect on each fraction was not evaluated. It may be seen that the low contribution of glutenins to the elasticity and the low values of the elastic components were a consequence of their characteristics, such as glutenins subunit size, polarity and number of cysteine residues, influencing the capability of disulphide bonds formation, which are necessary to conform the glutenin polymer structure (Žilić *et al*., 2011). The estimated retardation times (λ_1 and λ_2) were not statistically different among the fractions.

When viscosity coefficients were analysed, the residue fraction showed the highest values for μ_0, μ_1 and μ_2, contributing with more than a half of the total viscosity (58.5%), followed by gliadins (26.5%) and albumins (9.3%) fractions (Table 2). The coefficient of viscosity μ_0, representing the viscous response of the sample, was also the highest in the residue fraction.

T a b l e 2. Viscoelastic properties of sintered tablets from Osborne solubility fractions and water soluble pentosans corrected by weight, flour and bread sintered tablets produced from hard red spring and soft red winter wheat cultivars

Sample	Modulus of elasticity and retardation time					Coefficient of viscosity				Compliance		
	Weight (%)	E (%)	E_0	E_1	E_2	μ (%)	μ_0	μ_1	μ_2	D_0	D_1	D_2
			(MPa)				(MPa s^{-1})			(1/MPa)		
Albumins	3.82b	7.97c	53.56b	$5.37x10^3$b	$3.82x10^3$b	9.32c	$1.73x10^4$b	$1.36x10^5$b	$3.90x10^7$b	$2.70x10^{-5}$b	$2.80x10^{-7}$b	$3.90x10^{-7}$b
Globulins	0.79d	0.56e	9.74b	$3.63x10^2$b	$2.84x10^2$b	0.67e	$2.46x10^3$b	$1.67x10^4$b	$2.82x10^6$b	$6.56x10^{-6}$b	$1.80x10^{-7}$b	$2.20x10^{-7}$b
Gliadins	5.22b	14.12b	79.50b	$9.32x10^3$b	$6.99x10^3$b	26.49b	$2.50x10^4$b	$2.28x10^5$b	$1.11x10^8$b	$3.18x10^{-5}$b	$2.90x10^{-7}$b	$3.80x10^{-7}$b
Glutenins	1.67dc	2.69d	23.01b	$1.87x10^3$b	$1.23x10^3$b	3.15d	$5.18x10^3$b	$4.71x10^4$b	$1.32x10^7$b	$1.20x10^{-5}$b	$1.90x10^{-7}$b	$2.50x10^{-7}$b
Residue[b]	86.11a	72.47a	788.17a	$4.71x10^4$a	$3.62x10^4$a	58.50a	$1.75x10^5$a	$1.44x10^6$a	$2.44x10^8$a	$8.38x10^{-4}$a	$1.50x10^{-5}$a	$1.90x10^{-5}$a
Water soluble pentosansc	2.39c	2.19d	33.04b	$1.60x10^3$b	$9.11x10^2$b	1.83e	$6.25x10^3$b	$3.93x10^4$b	$7.67x10^6$b	$2.16x10^{-5}$b	$4.50x10^{-7}$b	$7.90x10^{-7}$b
Σ		100	987.02	$6.56x10^4$	$4.94x10^4$	100	$2.31x10^5$	$1.90x10^6$	4.1710^8	$9.36x10^{-4}$	$1.63x10^{-5}$	$2.10x10^{-5}$
Starch	67.5		1160.95a	$1.19x10^5$a	$6.87x10^4$a		$6.37x10^8$a	$5.15x10^5$a	$3.00x10^6$a	$8.65x10^{-4}$c	$8.57x10^{-6}$b	$1.46x10^{-5}$c
Flour tablet	-		628.02b	$2.35x10^4$b	$2.09x10^4$b		$1.32x10^8$b	$1.04x10^5$b	$8.69x10^5$b	$1.57x10^{-3}$b	$4.35x10^{-5}$a	$4.81x10^{-5}$b
Bread tablet	-		454.22b	$2.25x10^4$b	$1.42x10^4$c		$6.35x10^7$c	$1.31x10^5$b	$5.43x10^5$b	$2.21x10^{-3}$a	$4.53x10^{-5}$a	$7.23x10^{-5}$a
Non-gluten		83.19				70.32						

a – Means (n= 6) followed by the same letter in the same column are not significant different at $p < 0.05$; b – residue is mainly composed by starch 68%, protein 4.4% and water insoluble pentosans about 3%, c – water soluble pentosans calculated by difference from recovery, d – E is expressed as a percent of weight on each fraction, where $E = E_0 + E_1 + E_2$; e – μ is expressed as of weight on each fraction, where $\mu = \mu_0 + \mu_1 + \mu_2$.

When compliances were analysed, the same trend was present, where the residue fraction showed the highest values among the other protein fractions, with magnitude of 1.0 x 10^{-2} MPa (Table 2). Haddad *et al.* (2001) demonstrated that endosperm of different wheat classes showed mechanical properties that could be explained by rheological models. They tested wheat endosperm in parallelepiped shaped form and described a viscoelastic behaviour with a threshold similar to that observed in other materials such as metals and polymers.

The latter proved that the all the wheat components interact among one another to give its unique viscoelastic characteristics. Since the residue was composed mainly of starch, in our study we isolated the rheological behaviour from the almost pure fraction, showing the important contribution of this and other non-gluten components to dough performance and to the quality characteristics of the products.

The estimated parameters of the Kelvin-Voigt model for creep test coefficients, from the Osborne solubility fractions and water soluble pentosans, were corrected by protein content in flour (Table 3). To our knowledge, there were no previous data to compare the viscoelastic parameters obtained in this study with. Nevertheless, it can be seen that the highest protein content is found in the gliadins fraction, followed by glutenins and albumins fraction, respectively. When the data were examined after the correction by protein in flour, it was observed that the highest elasticity value was provided by the gliadins fraction.

The instantaneous response (E_0) and the retarded elastic response elements (E_1 and E_2) of gliadins were the highest (57.4%), and were statistically different when compared with the other fractions (Table 3). The same trend was observed for the viscosity coefficients, since that fraction contributed with 72.2% of the total viscosity, almost 10 times the viscosity of the glutenins fraction. Li *et al.* (2006) studied the polymer conformation of structure of wheat proteins and noted that the presence of β-sheet structure and content in gliadins and glutenins are quite different, the glutenins being those of lower content. Those authors proposed that gliadin and soluble glutenin molecules mainly form β-turn structures, and insoluble glutenins in gel proteins form a β-sheet structure, which seem to be an important contributor to the formation of a network structure and, subsequently, affected the elastic and long-time relaxation properties of gluten proteins.

It was evident, when Table 1 and Table 3 were compared, that protein content in each fraction influences significantly the viscoelastic parameters, since the ratio of glutenins and gliadins is 1:4. These results were according to our previous study (Figueroa *et al*., 2016), where gliadins fraction showed the highest elastic and viscosity coefficients obtained by relaxation tests. Glutenins fraction represented only 8.7 and 6.5% of elasticity (E %) and viscosity (μ %), respectively.

The residue and albumin fractions provided similar values of elasticity (E %), however, the residue fraction presented higher instantaneous elastic response of the E_0 element. Statistical differences were shown among the fractions, which evidenced the specific rheological characteristics of each group of molecules (Table 3).

The sum of the elastic moduli for flour (E_0+E_1+E_2) was 4.50x10^4 MPa, however, the sums of the elastic moduli of the total proteins fraction (fractions) and starch in flour amounted to 5.49x10^4 and 1.88x10^5 MPa, respectively. Thus, starch showed a higher elastic modulus (E_0) than flour and bread, respectively. Our previous study noted that total proteins contributed with only 2.03 MPa and that the higher contribution to the elastic performance was provided by starch, which agrees with the results presented in this study. The contribution of water soluble pentosans was relatively low, as expected, since their protein content is minimum; those compounds showed values of E % = 0.93 and μ % = 0.49 from the total elasticity and viscosity, respectively (Table 3). However, even when those components show lower viscosity performance, their functionality and interaction with gluten proteins increase, as demonstrated by Santos *et al.* (2005). They noted that during gluten formation, the water soluble pentosans (WSP) caused a reinforcement of the network, forming structural rearrangements with other components of flour, which have effects on gluten functionality (probably by the differences on the polysaccharide size), increasing the elastic moduli and viscosity coefficients.

When compliances of Osborne extraction were analysed, the residue fraction showed the highest values for D_0, D_1 and D_2, followed by gliadins and glutenins fractions. However, flour and bread presented the highest compliances when compared with the fractions and WSP.

CONCLUSIONS

1. An important contribution of non-gluten components, such as starch, albumins and globulins to the viscoelastic performance of sintered tablets from Osborne fractions, flour and bread was found.

2. Reconstituted tablets (albumins, globulins, gliadins, glutenins, residue and water soluble pentosans) showed similar values to those obtained from the sum of the regression coefficients of the fractions.

3. Starch fraction showed remarkable viscoelastic behaviour, and as a major component of flour contributed greatly to its functionality.

4. Gliadins, albumins and glutenins showed the highest contribution to the elasticity, viscosity and compliance of sintered tablets.

5. When compliance data corrected by protein content in flour were analysed, the residue fraction showed the highest values, demonstrating that in this fraction strain increases for a constant applied stress, followed by gliadins and glutenins.

T a b l e 3. Viscoelastic properties of sintered tablets made from Osborne solubility fractions corrected by protein content in flour of the respective fractions of hard red spring and soft red winter wheat cultivars, flour and bread[a]

Sample	Modulus of elasticity and retardation time						Coefficient of viscosity				Compliance		
	Protein (%)		E (%)	E_0	E_1	E_2	μ (%)	μ_0	μ_1	μ_2	D_0	D_1	D_2
	fraction	flour		(MPa)				(MPa s^{-1})			(1/MPa)		
Albumins	42.7d	1.61c	16.71b	213.17c	$2.15x10^4$b	$1.52x10^4$b	12.11b	$7.23x10^4$b	$5.61x10^5$b	$1.53x10^8$b	$1.08x10^{-4}$c	$1.09x10^{-6}$cb	$1.54x10^{-6}$cb
Globulins	57.8c	0.46d	1.36d	44.96d	$1.67x10^3$c	$1.31x10^3$c	1.03d	1.13x104b	$7.75x10^4$b	$1.30x10^7$b	$3.02x10^{-5}$d	$8.11x10^{-7}$c	$1.03x10^{-6}$c
Gliadins	88.4a	4.62a	57.38a	620.53a	$7.19x10^4$a	$5.42x10^4$a	72.24a	$1.97x10^5$a	$1.79x10^6$a	$9.14x10^8$a	$2.48x10^{-4}$b	$2.28x10^{-6}$b	$2.94x10^{-6}$b
Glutenins	75.7b	1.27c	8.73c	144.50c	$1.15x10^4$cb	$7.64x10^3$cb	6.52c	$3.22x10^4$b	$2.94x10^5$b	$8.24x10^7$b	$7.58x10^{-5}$c	$1.20x10^{-6}$cb	$1.59x10^{-6}$cb
Residue[b]	4.6f	4.00b	14.85b	308.47b	1.84x104b	$1.41x10^4$b	7.58c	$6.81x10^4$b	$5.64x10^5$b	$9.56x10^7$b	$3.27x10^{-4}$a	$5.91x10^{-6}$a	$7.46x10^{-6}$a
Water soluble pentosansc	10.1e	0.27d	0.93d	26.86d	$1.30x10^3$c	7.41x102c	0.49d	$5.08x10^3$b	$3.20x10^4$	$6.24x10^6$b	$1.76x10^{-5}$d	$3.66x10^{-7}$b	$6.38x10^{-7}$c
Σ		12.22	100	1358.49	$3.12x10^4$	$2.24x10^4$	100	$3.85x10^5$	$6.63x10^6$	$1.26x10^9$	$2.80x10^{-4}$	$1.35x10^{-5}$	$6.39x10^{-6}$
Starch		68		1160.95	$1.19x10^5$	$6.87x10^4$		$6.37x10^8$	$5.15x10^5$	$3.00x10^6$	$8.65x10^{-4}$	$8.57x10^{-6}$	$1.46x10^{-5}$
Flour tablet				628.02	$2.35x10^4$	$2.09x10^4$		$1.32x10^8$	$1.04x10^5$	$8.69x10^5$	$1.57x10^{-3}$	$4.35x10^{-5}$	$4.81x10^{-5}$
Bread tablet				454.22	$2.25x10^4$	$1.42x10^4$		$6.35x10^7$	$1.31x10^5$	$5.43x10^5$	$2.21x10^{-3}$	$4.53x10^{-5}$	$7.23x10^{-5}$
Non-gluten			33.85				21.21						

a – Means (n= 6) followed by the same letter in the same column are not significant different at $p < 0.05$; b – residue is mainly by starch 68%, insoluble protein 4.4% and water insoluble pentosans about 3%, c – E is expressed as a percent of protein in flour for each fraction, where $E = E_0 + E_1 + E_2$, μ is expressed as a percent of protein in flour for each fraction, where $\mu = \mu_0 + \mu_1 + \mu_2$.

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