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Creep recovery tests to measure the effects of wheat glutenins on doughs and the relationships to rheological and breadmaking properties

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ABSTRACT

The effects of high molecular weight-glutenin subunits on creep and recovery viscoelasticity of fully developed dough were investigated. The components of a Kelvin–Voigt model were used to evaluate viscoelasticity contributions and correlations. Elastic moduli of wheat dough G_1 and G_2 of specific glutenins correlated with quality indicators while shear modulus G_0 seem to contribute in a lower extent. $Glu-D1$ 5 + 10 showed higher elasticity in G_0 , G_1 and G_2 compared to $Glu-D1$ 2 + 12. $Glu-B1$ 17 + 18 presented higher elasticity for G_0 , G_1 and G_2 compared to 7 + 8 and 7 + 9. Viscosity η_0 showed higher correlation than viscosity (η_1 and η_2) indicating that the two factors were important in explaining swelling capacity of proteins η_2 and viscosity of non-gluten components η_0 in dough. Viscosities η_0 , η_1 and η_2 were higher in 5 + 10 and 17 + 18 compared to the other compositions. Viscosity η_1 seems to play minor role. Protein and wet gluten were significantly correlated indicating that dough viscoelasticity is related to glutenin composition.

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1. Introduction

Gluten proteins play a key role in determining the unique processing and baking quality of wheat by conferring water absorption capacity, cohesiveness, viscosity and elasticity of doughs [\(Wieser,](#page--1-0) [2007\)](#page--1-0). Glutenins are of great importance in explaining the variation that occurs in rheological and ''bread-making'' properties of wheat. At the genetic level gluten proteins are now relatively well known, but the specific three dimensional organization of gluten, and how the different individual proteins contribute to its properties, remains far from being completely understood [\(Lindsay and](#page--1-0) [Skerrit, 1999](#page--1-0)). The presence of specific high molecular weight glutenin subunits (HMW-GS) is significantly associated with several quality indicators ([Payne et al., 1987; Luo et al., 2001; Tohver,](#page--1-0) [2007\)](#page--1-0). Few publications have addressed the influence of glutenin proteins on viscoelastic properties of wheat doughs ([Lefebvre and](#page--1-0) [Mahmoudi, 2007](#page--1-0)), in spite of their importance and influence on the machinability of the dough, gas holding capacity dynamics and eventually quality of the baked bread [\(Bockstaele et al., 2011\)](#page--1-0).

The viscoelastic properties of cereals products have been investigated by different methods which vary in sample size and type of the applied deformation [\(Dobraszczyk and Morgenstern, 2003\)](#page--1-0).

⇑ Corresponding author. E-mail address: jfigueroa@qro.cinvestav.mx (J.D.C. Figueroa). Examples of such investigations include dough using dynamic oscillatory measurements [\(Lefebvre and Mahmoudi, 2007](#page--1-0)) and large deformation ([Mita and Bohlin, 1983\)](#page--1-0), in gluten ([Li et al.,](#page--1-0) [2003\)](#page--1-0), creep test in endosperm [\(Haddad et al., 2001](#page--1-0)), creep test in wheat kernels ([Hernández-Estrada et al., 2012](#page--1-0)), as well as stress relaxation tests in wheat kernels [\(Hernández et al., 2012\)](#page--1-0).

Dough is a macroscopically homogeneous mixture of starch, protein, lipid, and other minor components. At optimum development, dough is fully hydrated and has the highest elasticity ([Song](#page--1-0) [and Zheng, 2007](#page--1-0)). The rheological behavior of dough and how it is affected by extrinsic and intrinsic factors are far from understood ([Lefebvre and Mahmoudi, 2007\)](#page--1-0). Dough development is a function of several factors including composition, moisture content, energy input, temperature, and flour quality [\(Campos et al., 1997\)](#page--1-0). In particular, water as a plasticizer, protein content, and HMW-GS composition have strong influence on dough mixing properties and on its final consistency; but the available information about the rheological bases of these effects remains unclear and sometimes conflicting [\(Lefebvre and Mahmoudi, 2007\)](#page--1-0). It has been suggested that water above a given limiting value does not interfere with dough structure but acts as simple inert filler. Consequently, varying moisture content of the dough proportionally changes dynamic properties. On the other hand, it has been suggested that water molecules act as a plasticizer lowering the Tg of the doughs ([Masi et al., 1998](#page--1-0)). Another important factor affecting rheological

properties is the development of dough. Undeveloped doughs are less resistant to deformation than developed doughs, attributed to less alignment of the long polymer chains in the direction of the stress, less interactions and changes in protein conformation of the final protein matrix ([Campos et al., 1997; Masi et al.,](#page--1-0) [1998; Song and Zheng, 2007](#page--1-0)). In most studies different doughs are compared at a constant farinograph consistency (varying water absorption) resembling a consistency closer to the baker's doughs that allows evaluating differences in the performance of flours with different protein composition and quantity. In the majority of studies on dough rheology. Several aspects affect the evaluation of viscoelastic properties on doughs including: (1) dough preparation, (2) dough linear viscoelasticity which is extremely low with strain of about 0.1% in dynamic test (e.g. at low strains no differences in relaxation behavior were detected by dynamic analysis), [Safari-Ardi and Phan-Thien \(1998\)](#page--1-0) and (3) internal and external factors (e.g.: room temperature (25 °C), moisture, among others).

Rheological behavior of doughs may be obtained from quasistatic measurements such as creep and stress relaxation or from dynamic measurements ([Hibberd and Wallace, 1966\)](#page--1-0). Previous studies indicated that the dough weakening could not be clearly recognized in the linear viscoelastic properties of various doughs ([Peressini et al., 2008\)](#page--1-0). Upon increasing shear rate, a particular pattern was effective to detect differences in viscoelastic properties. Differences in dough extensional behavior have been attributed to entangled HMW glutenins [\(Edwards et al., 2001](#page--1-0)). [Safari-Ardi](#page--1-0) [and Phan-Thien \(1998\)](#page--1-0) showed that linear viscoelastic data, although important in the characterization of time scales in doughs, are largely irrelevant in differentiating between dough types and they proposed testing at a range of large strains (up to 29%) where creep and relaxation behavior was closely correlated with the baking behavior of doughs. Therefore, the objective of this study was to investigate the viscoelastic properties of fully developed doughs using creep and recovery tests at strains up 7%. The fitting of creep data to different rheological models was also attempted and discussed. The effects of HMW-GS on dough mechanical and rheological properties were examined.

2. Materials and methods

2.1. Plant materials

Nineteen hard red winter wheat lines grown in USA during the crop cycle 2010–2011 were studied. Among the lines, six had the HMW glutenin composition of 2^* , 7 + 8, and 5 + 10; four had 2^* , 7 + 9, and 2 + 12; three had 2^* , 7 + 9, 5 + 10; two had 1, 7 + 8, and $5 + 10$; two 1, $7 + 9$, and $5 + 10$; and two had 2^* , $17 + 18$, and 5 + 10 according to the nomenclature of [Payne et al. \(1987\).](#page--1-0) The HMW glutenins was separated according to [Naeem and](#page--1-0) [Sapirstein \(2007\),](#page--1-0) and subunits were determined by Reversed Phase High Performance Liquid Chromatography (RP-HPLC) by the procedure of [Naeem and Sapirstein \(2007\)](#page--1-0) using Agilent 2100 Bioanalyzer (Agilent Technologies Inc. Wilmington DE, USA).

2.2. Flour analysis

Grain test weight was evaluated according to AACC standard method (55–10.01), then the grain was milled in a Brabender Senior flour mill (C.W. Brabender Instruments Inc., South Hackensack, NJ). Refined flour was evaluated by AACC standard methods ([AACC Int., 2010](#page--1-0)) for sedimentation volume (56–60.01), protein content by combustion method (46–30.01), wet gluten (38– 12.02), ash-basic-method (08–01.01) and damaged starch (76– 30.02). Dough empirical rheology was assessed by mixograph, farinograph, and alveograph (54–40.02, 54–21.02, and 54–30.02, respectively) and extensigraph by modified preparation method of [\(Chen et al., 2009\)](#page--1-0) and method 54–10.01, and baking quality (10–11.01). Analyses were done at least in duplicated.

2.3. Rheology

2.3.1. Dough preparation and sample loading

The dough formula for rheological testing consisted of wheat flour $(10 g - 14%$ moisture basis) at optimum farinograph water absorption. Doughs were prepared in the farinograph by mixing until development of dough. Dough was immediately rolled into a ball-shape and relaxed in a press of 2.5 kg top plate and gap of 2.5 mm for 40 min of resting at room temperature. A 25 mm disc dough sample was obtained and loaded into AR1000 rheometer (TA Instruments, New Castle, DE) following the procedure described by [Zhao et al. \(2010\)](#page--1-0). The disc sample was re-trimmed to the 25 mm parallel-plate lowered to 2.5 mm gap. To prevent moisture loss during the test, mineral oil was applied to the edge of the sample. Probe used has geometry of hatched parallel plates of 25 mm diameter. The temperature of the dough was kept constant at 25 °C during creep-recovery test.

2.3.2. Selecting the viscoelastic conditions of the dough

A preliminary experiment was performed in order to check if the doughs were in the linear regime of viscoelasticity. The procedure indicates that the dough is linearly viscoelastic if the stress is proportional to strain at a given time, and the linear superposition principle holds ([Brinson and Brinson, 2008](#page--1-0)).

In which (γ) are the strain output and σ_0 stress input, respectively, and c is the proportional stress factor. Curves at 20 and 100 Pa were normalized at σ_0 of 40 Pa by multiplying the original strain by stress factor (c) . Creep curves of wheat dough under three stress levels (20, 40 and 100 Pa) are presented in [Fig. 1](#page--1-0)a. [Fig. 1b](#page--1-0) shows same dough samples but the curves were normalized at constant stress (σ_0) of 40 Pa, in order to find the linear viscoelastic regime. As shown in [Fig. 1b](#page--1-0) the normalized curves overlapped then all stresses evaluated met the viscoelastic linear range for the preparations of dough samples studied. Therefore creep recovery tests were performed at 100 Pa of stress in order to detect differences of viscoelastic properties as affected by HMW-GS composition.

2.3.3. Creep-recovery measurements

In a creep-recovery experiment, 100 Pa of shear stress was imposed on a dough sample and the sample's deformation or strain was recorded as a function of the creep time. Seven doughs were prepared and evaluated from each flour sample. The results were expressed as compliance (1/Pa), which corresponds to the strain divided by the imposed shear stress (Eq. (2)). In the recovery phase, the shear stress was removed and the sample is allowed to recover the elastic (instantaneous and retarded) part of the deformation. For a creep recovery experiment three variables have to be chosen, the length of the creep phase, the shear stress applied during creep and the length of the recovery phase. So, for this purpose the effect of creep time, shear stress and recovery time on creep-recovery parameters of flour–water dough was studied in more detail.

2.3.4. The generalized Kelvin–Voigt model

The viscoelastic behavior of the doughs were studied using mechanical analogues composed of springs and dashpots. The system comprises a combination of Hookean bodies (springs) and fluid bodies (dashpots filled with Newtonian liquid) to describe the experimental data. The general Kelvin–Voigt model of 6-elements under a creep test can be described as:

$$
\gamma(t) = \frac{\sigma_0}{G_0} + \frac{\sigma_0}{G_1} (1 - e^{-t/\lambda_1}) + \frac{\sigma_0}{G_2} (1 - e^{-t/\lambda_2}) + \frac{\sigma_0}{\eta_0} t \tag{1}
$$

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