J Food Sci Technol (December 2015) 52(12):7954–7963
DOI 10.1007/s13197-015-1953-6

ORIGINAL ARTICLE

Physicochemical characterization of white, yellow and purple maize flours and rheological characterization of their doughs

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Revised: 14 June 2015 / Accepted: 10 July 2015 / Published online: 19 July 2015

Abstract White, yellow and purple maize flours were obtained after dried kernels milling with two different sieves (200 and 500 μm). Hygroscopic characteristics, particle size distribution, colour and total starch and damaged starch (DS) of flours were determined. Maize flour doughs were obtained by mixing of flour and water in a laboratory kneader (Mixolab®) at constant dough consistency (1.10 ± 0.07 Nm). Dough properties like water absorption (WA), development and stability times were determined. Rheological characterization was carried out at 30 °C by means of oscillatory frequency sweep (1–100 rad s^{-1}) at 0.1 % strain and creep (50 Pa, 60 s) - recovery (0 Pa, 180 s) tests using a controlled stress rheometer. No significant differences were observed among water desorption isotherms of maize varieties and Halsey model was satisfactorily employed. Under the same milling conditions, white maize flours showed higher average particles size than purple and yellow maize flours. A model to predict flours colour involving colour parameters of the particle size fractions is proposed. Flours obtained with smaller particle size showed higher DS content and WA. For tested doughs, the mechanical spectra showed that elastic component was dominant over the viscous one. Damping factor varied slightly with angular frequency. Moduli values depended on average particle size and WA of dough. Creep-recovery data were satisfactorily fit with Burgers model. Instantaneous creep compliance varied with the same trend than elastic modulus. Viscoelastic creep compliance increased linearly with WA of the tested doughs and, at constant average flour particle size, increased with increasing DS.

Keywords Particle size · Colour · Damaged starch · Mixolab® · Dough rheology

Highlights
- Flour colour can be estimated from colour of its particle size fractions.
- Damaged starch decreases with particle size and affects to water absorption.
- Viscoelastic characteristics of flour doughs depend on water absorption.
- Rheology of dough depends on maize variety and average particle size of flour.

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Introduction

Maize (*Zea mays* L.) is the most produced cereal in 2013 in the world (FAO 2015). Corn is used for many food (human and animal) and non-food (pharmaceutical, cosmetics, chemical, among others) applications. Specifically, in the northwest of Spain, maize is cultivated for self-consumption in small farms. Maize bread is traditionally made with whole grain of maize, following similar procedures as for manufacturing the common wheat bread (Revilla et al. 2008). Although most consumers prefer white maize (Rebordanes variety), other types are also used for making bread, like yellow (Sarreaus variety) and purple (Meiro variety) maize.

Chemical composition of different maize kernels can be considered homogeneous but it depends on cultivation conditions, temperature, variety and maize type (white, yellow, purple, black, etc). The main components in maize are (%, *w/w* wet basis, w.b.): carbohydrates (≈77), water (≈11), protein (≈7), total fibre (≈7), total lipids (≈4) and ash (≈1.5) (Gwirtz and García-Casal 2014). The main compound of the maize is starch and is responsible of the high nutritional value (either for human and/or animal consumption).

The maize kernel is composed of four primary structures: endosperm, germ, pericarp, and tip cap, representing 83, 11, 5, and 1 % of the maize kernel respectively (Gwirtz and García-Casal 2014). There are differences in composition between these structures. For example, the pericarp has high fibre content (86 %, *w/w* dry basis, d.b), the endosperm presents the highest starch content (87 %, d.b.) and ca. 8 % d.b. of proteins. The germ presents high percentage (d.b.) in lipids (33 %), proteins (20 %) and minerals (11 %) (Watson 1987).

Although starch and proteins are the major compounds of maize grains, several other substances produced by secondary metabolism, such as carotenoids and anthocyanins, have been found, mainly in cereals genotypes (Escribano-Bailón et al. 2004). The carotenoids are tetraterpenes responsible for the yellow, orange and red colours of several vegetables (Kuhnen et al. 2011). Otherwise, the anthocyanins are water-soluble pigments responsible for the purple, blue and red colours in vegetal tissues, belonging to the class of flavonoids (Escribano-Bailón et al. 2004). Anthocyanin-rich foods and anthocyanin pigments have been suggested as potential agents to reduce the risk of colon cancer (Jing et al. 2008).

Celiac disease is characterized by inflammation of the small-intestinal mucosa that results from a genetically based immunologic intolerance to ingested gluten. This fact promotes the development of new products with no presence of gluten suitable for celiac patients. In recent years the interest in gluten-free products is growing due to the increase in celiac disease (Lazaridou et al. 2007). In the case of bakery products, the formulations can include gluten-free flours (mainly, rice and maize) with addition of other components like protein, fibres, fats, hydrocolloids and others, in order to simulate the viscoelastic properties of gluten and mimic the structure, texture and degree of acceptance of the finished products. From the technological point of view, the development of such products presents considerable difficulties. The absence of gluten has an impact on the characteristics of the crust, crumb, volume, porosity and other parameters of baked product quality (Sivaramakrishnan et al. 2004). For this reason, many gluten-free products that are available on the market offer a lower quality (both technological and nutritional) compared with the same products made with wheat. In fact, the gluten-free products composition ranges for the main components from 35 to 45 % carbohydrates, 2.5–6 % protein, 2–10 % lipids and significantly differs from conventional products (41–56 % carbohydrate, 8–13 % protein and 2–4 % lipid) (Collar et al. 2007). The design and development of gluten-free products that include maize flour is postulated as a potential alternative to meet the organoleptic and nutritional deficiencies thereof. The characterization of dough from one kind of flour is necessary to predict the behaviour of the respective dough made from flours blend.

Gluten-free flours from cereals (maize and rice) and beans (chickpea) are commonly manufactured (Moreira et al. 2013a). Some studies on these flour doughs are available (Lazaridou et al. 2007) but no comparative studies on different types of maize have been reported. The gluten-free doughs rheological behaviour can be affected by water absorption, particle size, temperature and shear rate (Marco and Rosell 2008). Rheological characterization can be related to the final product quality and is also fundamental for different technological purposes like process design, selection of units and operation conditions, etc.

The main aim of this work is to characterize physically and chemically different flours made from three types of maize, and rheologically the corresponding doughs manufactured at the same consistency using a laboratory kneader. Furthermore, results of rheological behaviour of doughs will be discussed in relation to determined physical and chemical properties of flours and compared to some commercial gluten and gluten-free flour doughs.

Materials and methods

Raw materials Three different types of Spanish maize kernels ((40 ± 5) % d.b.), white (WF, Rebordanes variety), yellow (YF, Sarreaus variety) and purple (PF, Meiro variety) acquired in a local market were employed as raw material.

Flour production The process for production of maize flours consisted on air-drying the maize kernels using a pilot-scale tray dryer (Angelantoni Challenge 250, Italy) with a constant temperature of 45 °C, 2 m/s of air velocity, 30 % of relative humidity and 5 kg/m^2 of loading density until average maize moisture content of 11 % d.b. was achieved. Dried maize kernels were triturated using a blender (Waring Blender HGBTWT, USA) and then milled using an ultra-centrifugal mill (ZM200 Retsch GmbH, Germany) with internal sieves of 200 and 500 μm. Six different maize flours (WM200, WM500, YM200, YM500, PM200, PM500) were obtained depending on the kind of maize and the internal sieve of ultra-centrifugal mill used during milling.

The flours obtained were placed in a closed vessel (desiccator) with constant temperature (25 °C) and relative humidity of air (54 %) employing saturated solution of $Mg(NO_3)_2$, until equilibrium between samples and surrounding air was reached in order to reach constant moisture content in flours (9–10 %, d.b.). Flours were then sealed in polyethylene plastic bags with a vacuum-packer (Sammic V201, Spain) and stored at 4 °C until its utilization.

Physicochemical characterization of flours Physical characterization of flours consisted of colour and particle size measurement. The particle size distribution of the obtained flours was determined by sieving employing standard sieves of 40, 63, 80, 125, 200, 250 and 500 μm (Cisa Cedacería Industrial,

Spain). Average particle diameter by mass (D_w) was calculated by Eq. (1):

$$D_w = \sum x_i D_{pi} \quad (1)$$

where D_{pi} (μm) is the average diameter of the fraction and x_i (%) the mass fraction.

Colour measure of the flour particle size fractions obtained after sieving was carried out by means of CIElab coordinates (L*, a*, and b*) using a colorimeter (CR-400, Konica Minolta, Japan). The L* value indicates the lightness (0–100 representing dark to light). The a* value gives the red–green colour degree (+a* = more red, −a* = more green). The b* value indicates the yellow–blue colour degree, (+b* = more yellow, −b* = more blue). The instrument was calibrated against a standard white reference. Total colour difference (ΔE*) between the samples and commercial maize flours taken as reference was calculated by Eq. (2):

$$\Delta E^* = \sqrt{\left(L_r^* - L_i^*\right)^2 + \left(a_r^* - a_i^*\right)^2 \left(b_r^* - b_i^*\right)^2} \quad (2)$$

where L_r*, a_r*, b_r* are colour parameters of commercial flour maize taken as reference and L_i*, a_i*, b_i* the colour parameters of each flour fraction. Colour differences might be analytically classified as: trace level (ΔE* = 0–0.5), slight (ΔE* = 0.5–1.5), noticeable (ΔE* = 1.5–3.0), appreciable (ΔE* = 3.0–6.0), large (ΔE* = 6.0–12.0), and obvious (ΔE* > 12.0) differences, Li (1998). For YM and WM flours, commercial white and yellow flours were taken as reference, Collar et al. (2014), Table 2. No comparison was performed for PM due to the absence of bibliographic data. Colour was measured ten times in each flour sample.

A model to estimate the colour characteristics of tested maize flours from the colour parameters of the particle size fractions was tested. The model is based on the relative number of particles of each particle size fraction in the maize flours and average colour parameters are calculated as the average volume based particle size. Under these assumptions, the proposed model is given by Eq. (3):

$$\left(L^*, a^*, b^*\right)_c = \sum_{i=<40\ \mu m}^{i=<500\ \mu m} \left(\frac{x_i}{\left(L^*, a^*, b^*\right)_i^3} \right)^{-1/3} \quad (3)$$

where L_c*, a_c*, b_c* are the estimated colour parameters of whole flours.

Water desorption isotherms of maize kernels were determined at 25 °C using a static gravimetric method (Wolf et al. 1985) in order to determine the equilibrium moisture content of maize flours at environmental conditions. This method consisted in placing small samples (0.5–0.8 g) in sealed jars provided with different saturated salt solutions that generate different relative humidity (from 0.11 to 0.85) of the surrounding air until equilibrium was achieved (ca. 12 weeks). At this moment, air and sample have reached the same water activity, a_w. Triplicate samples were placed into each jar. Halsey model, Eq. (4), was employed to fit the hygroscopic equilibrium data (Halsey 1948):

$$X = \left(\frac{-A}{\ln\left(a_w\right)} \right)^{1/B} \quad (4)$$

where X (d.b.) is the equilibrium moisture content, and A (d.b.) and B (−) are the Halsey model parameters.

Chemical characterization of flours was realised by means of moisture content and total and damaged starch. Moisture content of maize flour was determined gravimetrically. Dry solid was obtained after drying using a vacuum oven (Heareus Vacutherm 5250 VT6025, Langenselbold, Germany) at 70 °C and 0.15 atm until constant weight (AOAC 1995). Starch characterization was carried out by means of both recommended enzymatic methods for total starch (TS, % g starch/g d.s.) and damaged starch (DS, % g damaged starch/g d.s.). Specifically, TS was measured as total starch in flour without previous gelatinization using the total starch assay kit (Megazyme Int., Ireland) following AACC Method 76.13 (AACC 2000) and DS was determined as the starch fraction that is thermal or mechanically damaged, using the starch damage kit (Megazyme Int., Ireland) following ICC Method no. 164 (ICC 2008).

Dough preparation and mixing behaviour measurement Maize flour doughs preparation and mixing behaviour studies were carried out in a laboratory kneader (Mixolab®, Chopin, France) following a standard protocol (ICC 2008). Flour and water were mixed at constant temperature (30 °C) and mixing rate (80 rpm) until the torque value (target consistency) achieved to 1.10 ± 0.07 Nm (consistency required to industrial wheat flours doughs). At target consistency, the main dough mixing properties (water absorption (WA), development (DT) and stability time (ST)) were determined. WA (% d.b.) is defined as the amount of water necessary to obtain determined dough consistency. DT and ST are defined as the time to reach the maximum torque and the period at which the dough torque is kept at 1.10 ± 0.07 Nm, respectively (Rosell et al. 2007).

Rheological measurements The rheological characterization was carried out using a controlled stress rheometer (MCR 301, Anton Paar, Austria) equipped with a chamber (CTD 450, Anton Paar, Austria) with parallel plates (50 mm diameter, 2 mm gap) at 30 °C (±0.1 °C). Doughs obtained with Mixolab® at the target consistency were placed between the plates. Excess volume of dough sample, in relation to the volume given by gap between plates, was trimmed and the edge was coated with paraffin (Panreac, Barcelona, Spain) to prevent water evaporation during the measurement. A rest

time of 5 min was applied to all samples before measuring. All measurement was carried out at least in triplicate.

The linear viscoelastic region (LVER) was determined by means of a strain sweep (γ, 0.01–10 %) at frequency of 1 Hz. The mechanical spectra of doughs were obtained by frequency sweep tests from 1 to 100 rad/s of angular frequency (ω) at 0.1 % strain (inside the LVER of the samples) to determine the storage, G′ (Pa), and loss, G′′ (Pa), moduli and the damping factor (tanδ = G′′/G′). Experimental data of G′ and G′′ vs. ω were fitted by Eqs. (5) and (6):

$$logG' = loga' + b' \, log\omega \quad (5)$$

$$logG'' = loga'' + b'' log\omega \quad (6)$$

where a′, a′′, b′ and b′′ are the fitting parameters.

Creep and recovery tests were performed by application of a constant stress (σ) of 50 Pa during 60 s outside the LVER (creep phase) and allowing strain recovery during 180 s after stress removal, σ = 0 (recovery phase). Experimental data of creep and recovery were analysed by creep compliance rheological parameters J(t) (Pa^{-1}) = γ/σ (Steffe 1996) and modelling by Burgers model (Burgers 1935) using the Eqs. (7) and (8) for creep and recovery phases, respectively:

$$J(t) = J_0 + J_m\left(1 - exp\left(\frac{-t}{\lambda_c}\right)\right) + \frac{t}{\eta_0} \quad (7)$$

$$J(t) = J_{max} + J_0 + J_m\left(1 - exp\left(\frac{-(t-60)}{\lambda_r}\right)\right) \quad (8)$$

where J_0, J_m and J_{max} (Pa^{-1}) are the instantaneous, viscoelastic and maximum creep compliance, respectively, t (s) is the phase time, λ_c (s) and λ_r (s) are the mean retardation time of creep and recovery steps, respectively, and η_o (Pa s) is the zero-shear viscosity. The recovery compliance, J_r (Pa^{-1}), is calculated by the sum of J_0 and J_m corresponding to recovery phase, Eq. (8). The J_r/J_{max} ratio gives information on relative elastic component of the maximum creep compliance.

Statistical analysis Experimental data were statistically analysed. Differences among means were identified by one-factor analysis of variance (ANOVA), followed by the Scheffe test and considering significant *P*-values ≤0.05 (IBM SPSS Statistics 22).

Results and discussion

Physicochemical characterization of flours Equilibrium moisture content of tested maize kernels at different water activities at 25 °C are shown in Fig. 1. It is observed that moisture content increases (from 0.05 up to 0.19 d.b.) with increasing water activity (from 0.11 up to 0.85). No significant differences were observed among tested maize varieties. This result indicated that maize kernels can be dried up to the same moisture content and dried samples can be stored at the same environmental conditions. For modelling purposes, all experimental data were considered together and Halsey model was satisfactorily ($R^2 > 0.98$, ERMS = $5\ 10^{-4}$) employed. Parameters values (A = 0.005 d.b., B = 2.109) were obtained after non-linear fitting. Water desorption isotherm shape and values of the parameters are in accordance with reported data by other authors (Doymaz and Pala 2003). Figure 1 shows the good agreement between experimental data and estimated water sorption isotherm given by the Halsey model. Maize samples with moisture content between 0.09 and 0.10 d.b. are in equilibrium with air of relative humidity from 0.45 to 0.55 at 25 °C.

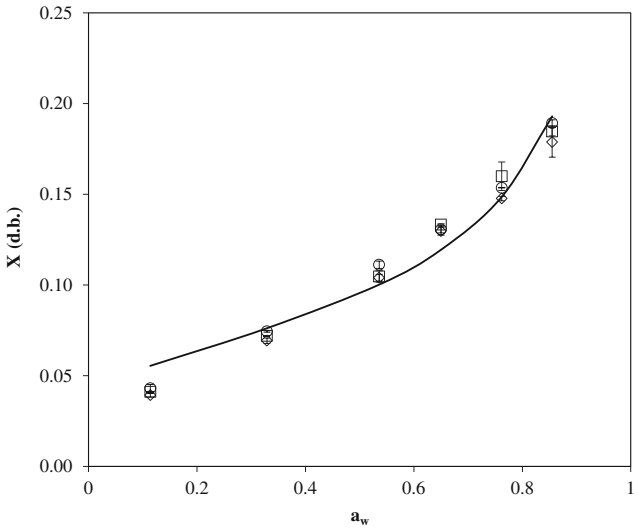

Fig. 1 Water desorption isotherms of yellow (○), white (◊) and purple (□) maize kernels at 25 °C. Line corresponds to Halsey model, Eq. (4)

Flours obtained after milling dried maize kernel with an internal sieve of 500 μm showed a maximum mass fraction (21.7–41.1 %) for particles with size from 250 to 500 μm, Table 1. The second more important mass fraction corresponded to particles with size between 125 and 200 μm (19.2–22.8 %). The lower mass fraction corresponded to particles with size below 40 μm for all flours (considering residual the fraction with particle size >500 μm). The average particle diameter by mass, D_w (Eq. (1)), for maize flours showed no differences between YM500 (176 μm) and PM500 (184 μm), but it was higher for WM500 (250 μm). For flours obtained using an internal sieve of 200 μm the maximum mass fraction (28.0–50.3 %) corresponded to particles with sizes between 80 and 125 μm. PM200 showed two more mass fraction with high weigh percent (40–63 μm, 27.7 % and 125–200 μm, 23.4 %). YM200 and WM200 showed only one more important mass fraction (63–80 μm, 29.8 % and 125–200 μm, 27 %, respectively). In spite of the different particle size distributions of these flours D_w varied in a narrow interval (90–113 μm). However, as in flours obtained with an internal sieve of 500 μm no differences in D_w was observed between YM200 (90 μm) and PM200 (93 μm) and the highest average diameter

Table 1 Particle size distribution and average particle size in mass, D_w Eq. (1), of tested maize flours

Fraction (μm)	D_p (μm)	Mass fraction (%)					
		YM200	WM200	PM200	YM500	WM500	PM500
>500	500.0	–	–	–	0.6	1.0	0.4
250–500	375.0	–	–	–	21.7	41.1	23.2
200–250	225.0	2.2	2.1	2.3	10.8	16.1	13.6
125–200	162.5	12.3	27.0	23.4	19.2	22.3	22.8
80–125	102.5	34.9	50.3	28.0	19.9	9.3	13.3
63–80	71.5	29.8	11.7	6.5	12.2	5.0	9.1
40–63	51.5	13.5	8.0	27.7	11.8	4.6	10.2
<40	20.0	7.4	0.9	12.0	3.8	0.6	7.4
D_w (μm)		90	113	93	176	250	184

Standard deviations of mass fraction were ±0.10

yellow (YM), white (WM) and purple (PM) maize flours obtained milling dried kernels with sieves of 200 (200) and 500 μm (500). Dp is the is the arithmetic mean diameter of the fraction

corresponded to WM200 (113 μm). Higher values for WM flours were probably due to the higher hardness of WM kernels in comparison to YM and PM kernels.

Colour characteristics of maize flours showed differences between them. The measured parameters (L*, a* and b*) values of whole flours (WF_m) showed the same behaviour independently on the average particle size, Table 2. PM flours showed the lowest value of L* (53.80 and 54.49 for PM500 and PM200, respectively) and WM the highest (65.25 and 66.41 for WM500 and WM200, respectively). PM flours showed the highest value of a* (2.22 and 2.94 for PM500 and PM200, respectively) indicating the domination of red over the green colour in this flour. This could be related to the high anthocyanin content of PM (Yang et al. 2008). For WM flours a* values (0.19–0.12) were near to zero indicating no domination of red over green colour. The lowest values of a*, corresponded to YM (−0.79 and −0.87 for YM200 and YM500, respectively), were negative which indicates the presence of green over red colour in these flours. Values of b* parameter were much higher than a* for all studied flours except PM flour. YM flour showed the highest b* values (20.92 and 17.24 for YM500 and YM200, respectively) that is indicative of its yellow colour due to the presence of high amount of carotenoids (Sandhu et al. 2007). The lowest b* value corresponded to PM (2.54 and 0.55 for PM500 and PM200, respectively). ΔE* parameter, Eq. (2), showed that the difference between every WF_m obtained could be considered as large difference (ΔE* = 6.0–12.0) compared to its respective commercial flour taken as reference, Table 2.

L* in PM was lower than YM and WM flours for every particle size studied, Table 2. L* decreased with particle size increased in YM and PM flours. In the case of YM and WM flours a* increased with particle size and for PM flours remained constant. Parameter b* showed the same trend for all studied flours and their values increased with particle size. The same trend has been reported for other maize flours (Bolade et al. 2009). Particle size fractions of PM flours showed the lowest b* values. These values, near to zero, indicate the tendency to blue colour of these flours. All fractions from WM and YM flours showed much higher b* values that indicates the clearly predomination of yellow colour respect to blue colour. It also remarkable the fact that, for particle size fractions of PM flours less than 125 μm, b* values were lower than a* values indicating that red-green colour dominates yellow-blue colour. All particle size fractions had a large difference (ΔE* = 6.0–12.0) in comparison to the commercial flour taken as reference with exception of some YM500 fractions (80–63 and 63–40 μm) that showed noticeable differences (ΔE* = 1.5–3.0). This result indicates that these fractions are the most similar to commercial flours according to their colour. These colour differences could be justified by the use in this work of whole maize flours and flours used as reference could be refined during processing.

Estimated colour parameters of whole flours (WF_c) were obtained by using Eq. (3). It can be observed that the employed model predicts satisfactorily the colour parameter values in comparison to experimentally measured values of whole flours (WF_m), Table 2. Particularly, for L* and a* high accuracy was observed among WF_c values and WF_m data. Total colour difference, ΔE* (Eq. (2)), between WF_m and WF_c (taking in this case as reference parameters (L_r*, a_r*, b_r*) those corresponding to WF_m) was evaluated. In tested flours, ΔE* values were low inside the range of 1.5 and 3.0 and can be classified like noticeable (Li 1998).

Total starch (TS, *%w/w*, d.b.) content of tested maize flours, YM, WM and PM, ranged from 60.1 ± 7.6 (WM500) up to 75.2 ± 6.3 (PM200) and no clear differences between varieties were found, Table 3. Specifically, WM flours showed total starch content (60.1 to 73.8) slightly higher than those reported by other authors for white maize (57.88 %), (Flores-Silva et al. 2014). In

Table 2 Colour parameters of maize flours and particle size fractions

Fraction (μm)	YM200	WM200	PM200	YM500	WM500	PM500
L*						
WF_m	63.13 ± 0.16^b	66.41 ± 0.33^c	54.49 ± 0.06^a	62.35 ± 0.02^b	65.25 ± 0.22^c	53.80 ± 0.03^a
500–250	–	–	–	52.09 ± 0.01^a	58.72 ± 0.02^c	53.16 ± 0.04^b
250–200	–	–	–	53.17 ± 0.02^b	62.27 ± 0.02^c	51.59 ± 0.03^a
200–125	55.45 ± 0.01^c	65.02 ± 0.02^e	49.49 ± 0.01^a	58.22 ± 0.01^d	66.74 ± 0.03^f	52.61 ± 0.03^b
125–80	60.67 ± 0.06^c	65.35 ± 0.02^e	50.75 ± 0.02^a	66.36 ± 0.01^f	64.02 ± 0.02^d	51.82 ± 0.03^b
80–63	63.26 ± 0.03^d	62.56 ± 0.02^c	51.13 ± 0.02^a	66.46 ± 0.05^f	64.31 ± 0.03^e	52.40 ± 0.02^b
63–40	64.46 ± 0.02^c	63.13 ± 0.03^b	54.11 ± 0.02^a	69.49 ± 0.02^e	65.73 ± 0.02^d	54.23 ± 0.05^a
< 40	62.78 ± 0.03^b	–	52.67 ± 0.28^a	66.96 ± 0.04^c	–	53.01 ± 0.02^a
WF_c, Eq. (3)	61.68 ± 0.03^d	65.36 ± 0.02^f	51.95 ± 0.08^a	59.28 ± 0.03^c	62.23 ± 0.02^e	52.72 ± 0.03^b
a*						
WF_m	−0.79 ± 0.01^a	0.12 ± 0.02^b	2.94 ± 0.02^d	−0.87 ± 0.03^a	0.19 ± 0.03^b	2.22 ± 0.02^c
500–250	–	–	–	1.48 ± 0.04^b	0.29 ± 0.01^a	2.78 ± 0.01^c
250–200	–	–	–	−0.69 ± 0.03^a	0.05 ± 0.01^b	2.04 ± 0.04^c
200–125	0.90 ± 0.05^d	0.18 ± 0.03^c	2.79 ± 0.03^f	−0.68 ± 0.03^a	−0.05 ± 0.01^b	2.14 ± 0.02^e
125–80	−0.57 ± 0.03^b	0.04 ± 0.03^d	2.96 ± 0.03^f	−1.54 ± 0.02^a	−0.26 ± 0.02^c	2.21 ± 0.03^e
80–63	−1.02 ± 0.03^b	−0.03 ± 0.02^d	3.04 ± 0.01^f	−1.65 ± 0.03^a	−0.37 ± 0.04^c	2.31 ± 0.02^e
63–40	−1.18 ± 0.03^b	−0.10 ± 0.01^d	3.12 ± 0.03^f	−1.76 ± 0.03^a	−0.41 ± 0.02^c	2.33 ± 0.04^e
< 40	−1.36 ± 0.02^b	–	2.94 ± 0.02^d	−1.60 ± 0.02^a	–	2.26 ± 0.03^c
WF_c, Eq. (3)	−0.78 ± 0.03^b	0.07 ± 0.02^d	2.98 ± 0.02^f	−1.01 ± 0.03^a	−0.13 ± 0.02^c	2.28 ± 0.03^e
b*						
WF_m	17.24 ± 0.03^e	7.47 ± 0.15^c	0.55 ± 0.03^a	20.92 ± 0.01^f	8.59 ± 0.18^d	2.54 ± 0.02^b
500–250	–	–	–	34.12 ± 0.04^c	10.37 ± 0.01^b	6.81 ± 0.01^a
250–200	–	–	–	29.31 ± 0.03^c	9.22 ± 0.02^b	3.11 ± 0.02^a
200–125	30.99 ± 0.03^f	8.80 ± 0.02^c	2.22 ± 0.02^a	26.40 ± 0.01^e	9.61 ± 0.01^d	2.73 ± 0.02^b
125–80	20.65 ± 0.03^e	7.92 ± 0.01^c	0.97 ± 0.01^a	22.04 ± 0.01^f	9.93 ± 0.01^d	1.77 ± 0.02^b
80–63	16.73 ± 0.02^e	7.61 ± 0.01^c	0.30 ± 0.01^a	17.58 ± 0.02^f	9.12 ± 0.02^d	0.79 ± 0.01^b
63–40	16.07 ± 0.01^e	7.28 ± 0.01^c	−0.30 ± 0.02^a	16.17 ± 0.02^f	8.42 ± 0.01^d	0.30 ± 0.01^b
< 40	13.80 ± 0.01^d	–	−0.56 ± 0.01^a	13.04 ± 0.02^c	–	−0.07 ± 0.02^b
WF_c, Eq. (3)	18.12 ± 0.02^e	8.10 ± 0.01^c	−0.49 ± 0.01^a	21.12 ± 0.02^f	9.80 ± 0.01^d	−0.16 ± 0.02^b
ΔE*, Eq. (2)*						
WF_m	7.86	7.11	–	9.72	9.18	–
500–250	–	–	–	22.34	11.76	–
250–200	–	–	–	18.19	8.03	–
200–125	17.71	5.41	–	12.43	4.50	–
125–80	7.60	4.74	–	3.69	6.75	–
80–63	5.11	7.31	–	1.84	6.09	–
63–40	4.38	6.69	–	2.99	4.51	–
< 40	7.11	–	–	5.74	–	–

Data are presented as means ± standard deviation. Data value of each parameter with different superscript letters in rows are significantly different, $P \leq 0.05$. Colour parameters of size fractions of yellow (YM), white (WM) and purple (PM) maize flours obtained milling dried maize kernels with sieves of 200 (200) or 500 μm (500) and also the experimental data of whole flour (WF_m) and calculated values by Eq, (3) (WF_c)

* ΔE* was evaluated taken commercial flours as references: yellow maize flour ($L_r^* = 67.9$, $a_r^* = -1.87$, $b_r^* = 18.7$) and white maize flour ($L_r^* = 69.7$, $a_r^* = -0.99$, $b_r^* = 6.35$) (Collar et al. 2014)

the remaining flours, TS content varied in a narrower range (68.1–75.2 d.b.) and in accordance with bibliographic data for maize flours (77.5 %), (Malumba et al. 2015). Damaged starch (DS, % *w/w*, d.b.) content showed significantly differences for studied flours, Table 3. Regardless of the average particle size of the flour (given by the used sieve during milling, 200 or 500 μm), WM variety always showed the highest (25.0 and 12.6 % for WM200 and WM500,

Table 3 Total and damaged starch of tested flours and mixing curves parameters of the corresponding doughs obtained in Mixolab® apparatus (target torque, C1: 1.10 ± 0.07 Nm)

Parameters	YM200	WM200	PM200	YM500	WM500	PM500
TS (% *w/w*, d.b.)	68.1 ± 2.2^{a,b}	73.8 ± 2.2^{b}	75.2 ± 6.3^{b}	71.6 ± 6.0^{b}	60.1 ± 7.6^{a}	71.3 ± .28^{a,b}
DS (% *w/w*, d.b.)	8.6 ± 0.2^{b}	25.0 ± 2.1^{e}	18.2 ± 0.5^{d}	2.6 ± 0.7^{a}	12.6 ± 0.6^{c}	8.3 ± 0.6^{b}
WA (%, d.b.)	63.0 ± 1.0^{b}	90.0 ± 2.0^{d}	81.1 ± 1.4^{c}	-	48.7 ± 2.0^{a}	-
C1 (Nm)	1.09 ± 0.02	1.11 ± 0.03	1.05 ± 0.04	0.50 ± 0.02	1.13 ± 0.04	0.25 ± 0.10
DT (min)	0.9 ± 0.1^{a}	0.8 ± 0.1^{a}	0.7 ± 0.1^{a}	-	7.6 ± 1.6^{b}	-
ST (min)	2.0 ± 0.1^{a,b}	1.5 ± 0.1^{a,b}	1.1 ± 0.1^{a}	-	2.9 ± 1.2^{b}	-

Data are presented as means ± standard deviation

Data value with different superscript letters in rows are significantly different, $P \leq 0.05$

TS total starch, *DS* damaged starch, *WA* water absorption, *DT* development time, *ST* stability time

yellow (YM), white (WM) and purple (PM) maize flours obtained milling dried maize kernels with sieves of 200 (200) or 500 μm (500)

respectively) and YM the lowest (8.6 and 2.6 % for YM200 and YM500, respectively) DS content. This fact can be related to the different hardness and friability of maize variety. It was also found that DS content of maize flours increased with increasing average particle size (employing the same sieve during milling). The milling operation affects directly the starch structure (Li et al. 2014). It can be observed that the flours obtained with a sieve of 200 μm showed higher DS content than 500 μm flours due to higher mechanical and thermal damage caused during milling. Bolade et al. (2009) observed the same trends, lower particle size fraction of flours showed higher DS content (from 12.2 to 17.4 % d.b. for 450 to <75 μm fractions, respectively).

Mixing behaviour Table 3 shows mixing curves parameters obtained in Mixolab® for all assayed flour doughs. From YM500 and PM500 flours, doughs with the target consistency could not be obtained (the consistency was lower) indicating that flour particle size was critical for the dough formation capacity. The obtained flour doughs showed WA values significantly different. The lowest WA corresponded to WM500 (48.7 %). This value is similar to WA of other gluten-free flour doughs (chestnut) with the same flour particle size (Moreira et al. 2010). The results showed like WA decreases with increasing flour particle size by comparison of WM samples (WM200, 90.0 %). WA for YM200 was 63 % and is in the range to those observed for wheat, rice and yellow maize flours doughs (Rosell et al. 2007; Hadnadev et al. 2011; Moreira et al. 2012). WM200 and PM200 doughs showed higher WA (>80 %). The presence of DS increases WA (Greer and Stewart 1959) and these high values can be related to the high DS content of its respective flours (>18 %). A linear relationship ($R^2 > 0.98$) was found among WA and DS for flour doughs with similar average particle size (kernels milled with 200 μm sieve). DT values (<1 min) were significantly lower for maize flour doughs with small average particle size (obtained with a 200 μm sieve) and no significant differences among maize varieties were observed. These DT values are comparable to those found for some gluten-free commercial flour doughs like rice (1.13 min), (Moreira et al. 2012). WM500 showed a DT value (7.6 min) comparable to those observed for whole wheat flour doughs (Moreira et al. 2012). High stability values are usually related to the strength of flours (Marco and Rosell 2008). ST values obtained are in the same range (from 1.1 up to 2.9 min) than those observed for soft wheat, rice and amaranth flour doughs (Moreira et al. 2012; Hadnadev et al. 2011). ST values increased with increasing average particle size (from 1.5 min for WM200 to 2.9 min for WM500).

Rheological properties Strain sweep tests established that linear viscoelasticity range was at strain <0.4 % for all flour doughs. To ensure that all tests were carried out within the linear viscoelastic range, a strain of 0.1 % was employed.

Figure 2 shows the mechanical spectra (data of storage modulus (G′), loss modulus (G′′) and damping factor (tanδ) at 30 °C for all maize flour doughs obtained at target consistency (1.10 ± 0.07 Nm). In all cases, in the studied range, G′ and G′′ values increased with increasing ω. This behaviour can be attributed to the absence of binding agents in the dough (absence of gluten) and repulsive forces between starch granules are predominant (Sivaramakrishnan et al. 2004). For all maize flour doughs G′ values were higher than G′′ indicating that elastic proportion was dominant over the viscous one, (tanδ < 0.5). This behaviour is according to the solid behaviour for other gluten-free flours like rice (Pruska-Kedzior et al. 2008) and chestnut (Moreira et al. 2013b) flour doughs. In tested doughs, tanδ varied in a restricted range indicating that elastic/viscous proportion is slightly modified with the angular frequency.

Oscillatory data were successfully fitted ($R^2 > 0.96$) by means of Eqs. (5) and (6), Table 4. The analysis of the fitting parameters values allows the better discussion on the differences of the flour

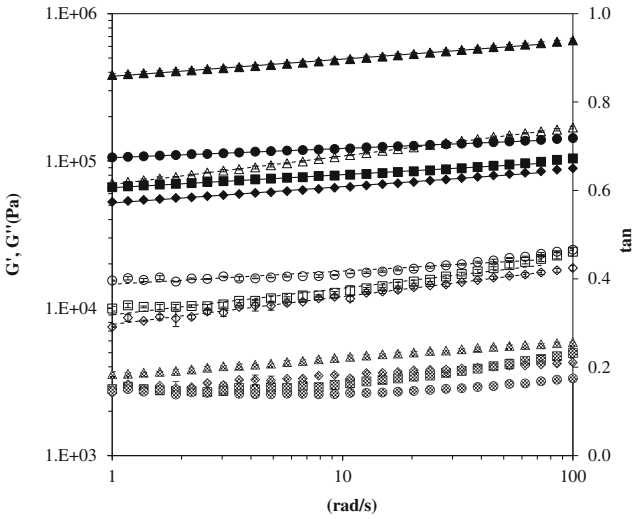

Fig. 2 Experimental data of G′ (filled markers), G′′ (empty markers) and tanδ (dot filled markers) of YM200 (●), WM200 (♦), PM200 (■) and WM500 (▲) maize flour doughs. Lines correspond to Eqs. (5) and (6)

doughs viscoelastic behaviour. WM500 flour dough showed high values for a′ and a′′ parameters demonstrating that average particle size of flour modifies strongly the viscoelastic character of dough. This increase might be attributed to different interactions between starch granules with different particle size distribution and the integrity of flour particles produces stiffer doughs (Moreira et al. 2010). Flour doughs with smaller average particle size (milled with a 200 μm sieve) were also significantly different between them. YM200 flour dough showed the highest a′ and a′′ values, and WM200 and PM200 flour doughs the lowest ones. This could be related to the high WA required for WM200 and PM200 flour doughs, Table 3. G′ and G′′ values of studied doughs are comparable to those observed for chestnut flour doughs (Moreira et al. 2013b), but they are higher than those observed for commercial wheat (Hadnadev et al. 2013) and maize (Pruska-Kedzior et al. 2008) flour doughs with similar WA. On the other hand, the slopes (b′ and b′′) indicated that flour doughs of white maize variety, independently on particle size (WM200 and WM500), showed greater dependence of both moduli (higher values of b′ and b′′) on angular frequency than yellow and purple maize flour doughs.

Experimental data of creep and recovery tests for all assayed doughs are shown in Fig. 3. During creep step (first 60 s) creep compliance increased and after stress removal (recovery step, last 180 s) creep compliance decreased until almost to achieve stationary state. The curves shape is similar to other gluten free systems previously studied (Lazaridou et al. 2007; Moreira et al. 2013b).

The parameters of Burgers model for creep and recovery curves parameters, Eqs. (7) and (8) respectively, for the tested flour doughs are shown in Table 5. Creep step was successfully modelled ($R^2 > 0.95$). WM200 and PM200 flour doughs showed the highest ($\approx 14 \cdot 10^{-6}$ Pa^{-1}) instantaneous creep compliance (J_0). This value is comparable to those obtained for gluten-free doughs, with similar WA, made from chestnut flour (Moreira et al. 2013b). The lowest value of J_0 ($3.6 \cdot 10^{-6}$ Pa^{-1}) corresponded to dough with highest particle size (WM500). These results are in good agreement with the elastic modulus values obtained in the sweep frequency test discussed above. Stiffer flour doughs showed lower J_0. The same trend was observed before in other gluten free flour doughs (Moreira et al. 2010). Viscoelastic creep compliance (J_m) was significantly different for all assayed doughs. The highest ($11.2 \cdot 10^{-6}$ Pa^{-1}) and lowest ($1.9 \cdot 10^{-6}$ Pa^{-1}) value of J_m corresponded to WM200 and WM500, respectively. J_m increased linearly ($R^2 > 0.94$) with WA in the tested flour doughs. Moreover, for maize flour doughs with smaller average particle size (obtained with 200 μm sieve), J_m increased with increasing DS, indicating that DS modified the viscoelastic behaviour of doughs. The mean retardation time of creep step (λ_c) varied from 1.5 up to 5.0 s. These values are comparable to those showed in literature for gluten-free doughs (Lazaridou et al. 2007). The λ_c parameter was significantly different between flour doughs obtained made from small particle size (200 μm sieve). In fact, for these flour doughs λ_c decreased with increasing DS. Flour dough with higher average particle size, WM500, showed an intermediate value of λ_c

Table 4 Rheological characterization of maize flours: parameters of Eqs. (5) and (6) for oscillatory shear modelling

Parameter	YM200	WM200	PM200	WM500
G′				
$a' \cdot 10^{-3}$ (Pa $s^{-b'}$)	105.2 ± 2.0^{b}	51.9 ± 1.1^{a}	65.9 ± 0.4^{a}	375.6 ± 10.2^{c}
b′	0.06 ± 0.01^{a}	0.11 ± 0.01^{c}	0.09 ± 0.01^{b}	0.12 ± 0.01^{c}
R^2	0.99	0.99	0.99	0.99
G′′				
$a'' \cdot 10^{-3}$ (Pa $s^{-b''}$)	14.6 ± 0.2^{b}	7.8 ± 0.3^{a}	9.0 ± 0.3^{a}	69.2 ± 0.9^{c}
b′′	0.09 ± 0.01^{a}	$0.19 \pm 0.01^{b,c}$	$0.18 \pm 0.01^{b,c}$	0.19 ± 0.01^{c}
R^2	0.95	0.99	0.96	0.99

Data are presented as means ± standard deviation

Data value with different superscript letters in rows are significantly different, $P \leq 0.05$

yellow (YM), white (WM) and purple (PM) maize flour doughs

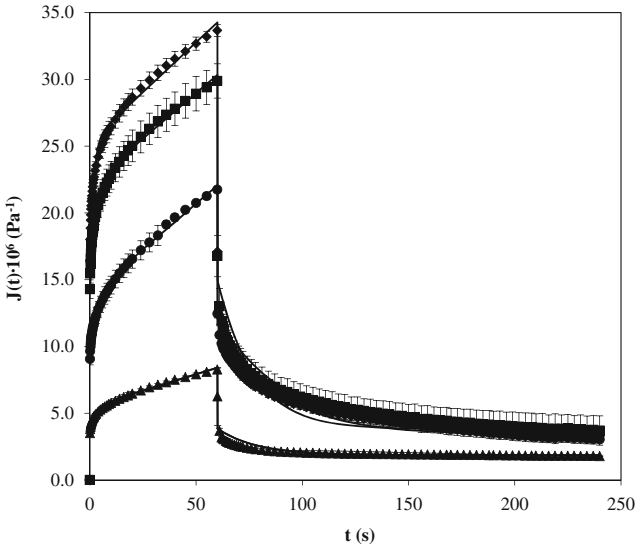

Fig. 3 Experimental creep and recovery data at 30 °C of YM200 (●), WM200 (♦), PM200 (■) and WM500 (▲) maize flour doughs. Lines correspond to Eqs. (7) and (8)

(3.2 s). Zero shear viscosity parameter (η_0) was significantly higher for WM500 ($20.5 \cdot 10^6$ Pa s) dough and no significant differences ($6.7–7.4 \cdot 10^6$ Pa s) among remaining flour doughs were observed. This fact confirms that flow resistance of doughs depends strongly on average particle size of flour from which they are obtained (Moreira et al. 2010).

Recovery phases of tested flours were acceptably fitted ($R^2 > 0.86$) by means of Eq. (8) and the corresponding parameters are shown in Table 5. The maximum creep compliance ($J_{máx}$) was significantly lower ($8.1 \cdot 10^{-6}$ Pa^{-1}) for WM500 indicating that doughs obtained from flour with large particle size show less deformation capacity. $J_{máx}$ increased linearly ($R^2 > 0.94$) with increasing WA in the tested flour doughs. This could be related to the formation of weak material structures promoted by high WA (Lazaridou et al. 2007). Instantaneous recovery compliance (J_0, $4.3–18.3 \cdot 10^{-6}$ Pa^{-1}) and viscoelastic recovery compliance (J_m, $2.0–11.2 \cdot 10^{-6}$ Pa^{-1}) values were in the same range as those observed in creep step. Values of mean retardation time of recovery step, λ_r, were higher than those observed for λ_c. These differences can be explained because the creep phase was performed outside the LVER and irreversible changes in the structure of the flours can be produced. The highest value of λ_r, as in the creep phase, corresponded to YM200 dough (34.0 s). Similar values were obtained for gluten-free doughs, with WA in the same range, from rice flour (38.1 s), (Lazaridou et al. 2007). WM500 showed the lowest value of λ_r (18.5 s) meaning that this dough variety needs less time to achieve the stationary state. On the other hand, J_r/J_{max} ratio values increased with WA in the tested flour doughs. All of them showed higher values (75.8–87.6 %) than wheat doughs (65 %) indicating a higher elastic character of doughs.

Conclusions

The results reveal that physical and chemical characteristics of maize flours and rheological properties of the corresponding doughs depend on maize variety and milling conditions. Using the same milling procedure, the average particle sizes of white maize flours were higher than those obtained for yellow and purple maize flours. Colour parameters of tested maize flours were significantly different. A weighted model, involving the colour characteristics of mass fractions, was satisfactorily tested to reproduce the colour parameters of the

Table 5 Rheological characterization of maize flours: parameters of creep, Eq. (7), and recovery, Eq. (8), models

Phase	Parameters	YM200	WM200	PM200	WM500
Creep	$J_0\ 10^6$ (Pa^{-1})	9.1 ± 0.1^b	14.1 ± 0.2^c	14.2 ± 0.9^c	3.6 ± 0.2^a
	$J_m\ 10^6$ (Pa^{-1})	4.8 ± 0.1^b	11.2 ± 0.5^d	8.0 ± 0.3^c	1.9 ± 0.1^a
	λ_c (s)	5.0 ± 0.1^d	1.5 ± 0.1^a	2.9 ± 0.2^b	3.2 ± 0.3^b
	$\eta_0\ 10^{-6}$ (Pa·s)	7.3 ± 0.9^a	6.7 ± 0.2^a	7.4 ± 0.2^a	20.5 ± 1.1^b
	R^2	0.98	0.95	0.96	0.97
Recovery	$J_{max}\ 10^6$ (Pa^{-1})·	21.7 ± 0.3^b	32.0 ± 1.4^c	30.5 ± 0.7^c	8.1 ± 0.3^a
	$J_0\ 10^6$ (Pa^{-1})·	9.6 ± 0.2^b	17.0 ± 1.4^c	18.3 ± 0.4^c	4.3 ± 0.1^a
	$J_m\ 10^6$ (Pa^{-1})·	8.8 ± 0.1^b	11.2 ± 0.1^c	8.9 ± 0.2^b	2.0 ± 0.1^a
	λ_r (s)	34.0 ± 1.4^d	17.0 ± 0.1^a	27.5 ± 0.7^b	18.5 ± 0.7^a
	$J_r/J_{máx}$ (%)	83.1 ± 0.1^b	87.6 ± 1.3^d	86.5 ± 0.9^c	75.8 ± 1.1^a
	R^2	0.95	0.85	0.91	0.86

Data are presented as means ± standard deviation

Data value with different superscript letters in rows are significantly different, $P \leq 0.05$

yellow (YM), white (WM) and purple (PM) maize flour doughs. J_0, J_m, Jr and J_{max} are the instantaneous, viscoelastic, recovery and maximum creep compliance, respectively, λ_c and λ_r are the mean retardation time of creep and recovery steps, η_0 is the zero-shear viscosity

tested maize flours. Milling is critical for the flour dough properties due to smaller average particle size increases the damaged starch content. In fact, water absorption decreases with increasing average flour particle size and also increases linearly with increasing damaged starch. Small amplitude oscillatory sweep tests applied for rheological characterization of doughs reveal that elastic behaviour was predominant in relation to viscous component. Viscoelastic moduli values increase with increasing average particle size and decrease with increasing water absorption. The corresponding, instantaneous and viscoelastic, compliances values determined by means of Burgers model showed the opposite behaviour. These results reveal that dough characteristics depend strongly on processing and intrinsic characteristics of raw materials.

Acknowledgments The authors acknowledge the financial support to Ministerio de Economía y Competitividad of Spain and FEDER (CTQ 2013-43616/P).

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