LWT - Food Science and Technology 154 (2022) 112819

Contents lists available at ScienceDirect

LWT

journal homepage: www.elsevier.com/locate/lwt

Effect of walnut flour addition on rheological, thermal and microstructural properties of a gluten free-batter

Juan J. Burbano [a], Darío M. Cabezas [b,c], M. Jimena Correa [a,*]

[a] Centro de Investigación y Desarrollo en Criotecnología de Alimentos (CIDCA), Facultad de Cs. Exactas-UNLP, CIC, CONICET, 47 y 116, 1900, La Plata, Argentina
[b] Laboratorio de Investigación en Funcionalidad y Tecnología de Alimentos (LIFTA), Departamento de Ciencia y Tecnología, Universidad Nacional de Quilmes, Roque Sáenz Peña 352, Bernal, Argentina
[c] Consejo Nacional de Investigaciones Científicas y Técnicas (CONICET), CABA, Argentina

ARTICLE INFO

Keywords:
Back-extrusion
Calorimetry
Microstructure
Weak gel model
^{1}H-NMR relaxation

ABSTRACT

For proper industrialization of gluten-free (GF) foods, it is important to know their properties and behavior under process. This is even more important when using novel ingredients. The by-product of walnut oil (*Juglans regia* L.) extraction is a press cake rich in polyunsaturated fatty acids and other bioactive compounds. From this cake, walnut flour (WF) is obtained by milling. This study was focused on evaluating the effects of adding WF on the rheological, textural, thermal, and microstructural properties of batters. Different levels of addition of WF (0, 100, 150, 200 g/kg flours blend) were assayed. The pasting properties of the flour blend (rice flour, cassava and maize starches) were altered by WF addition. Likewise, increased WF concentration on batters increased their firmness, cohesiveness and, consistency and viscosity indexes.

Regarding the molecular mobility of components of batters, a decrease in the relaxation times in NMR assays and in the amount of freezable water was found while the glass transition was not affected. Confocal scanning laser micrographs of batters showed a protein matrix with starch granules, oil droplets, and fiber fragments. Overall, these results showed that the addition of WF enhanced the quality of the GF batters.

1. Introduction

Celiac disease (CD) is one of the most prevalent lifelong illness worldwide, which is an immune enteropathy triggered by gliadin fraction of wheat and the prolamins of rye, barley and oats (Catassi & Fasano, 2008). Gluten consumption causes inflammation of the small intestine leading to the poor absorption of several important nutrients including iron, folic acid, calcium and fat soluble vitamins (Gallagher, Gormley, & Arendt, 2004). An increase in the disease incidence has been reported in many countries (Murray et al., 2003) and nowadays the only solution for celiac people is the avoidance of gluten.

When developing foods for celiac people, it is important to find an adequate balance between the nutritional and technological quality of the final products. Flours and starches from grains and tubers are suitable ingredients for the preparation of gluten-free (GF) products. Their advantages are colorless, having a mild flavor, and in the case of rice flour, hypoallergenicity. Since they have low amounts of protein, fiber, and unsaturated fats, it is important to find complementary ingredients for these flours, such as whole grains flours of pseudocereals and nutritive sub-products like oil press cakes. The walnut oil (*Juglans regia* L.) production yields a partially defatted cake, being its composition highly dependent on the extraction method (Labuckas, Maestri, & Lamarque, 2014). The walnut flour (WF) is obtained from the milling of this press cake and it is rich in polyunsaturated lipids. It is also a source of proteins, fibers, minerals, and other bioactive compounds (Burbano & Correa, 2021).

Characterization of GF batters is an essential task to ensure good technological quality. Characterization may involve textural, rheological, thermal, structural and microscopic aspects. These batter parameters are linked to the textural attributes of the final product, which, in turn, determine its sensory characteristics and consumer acceptability (Bozdogan, Kumcuoglu, & Tavman, 2019).

The aim of this study was to evaluate the effects of adding walnut flour on the rheological, textural, thermal and microstructural properties of gluten-free batters destinated to the production of cakes.

---

* Corresponding author. Centro de Investigación y Desarrollo en Criotecnología de Alimentos (CIDCA), Facultad de Cs. Exactas-UNLP, CIC, CONICET, 47 y 116, 1900, La Plata, Argentina.
E-mail address: mjcorrea@biol.unlp.edu.ar (M.J. Correa).

https://doi.org/10.1016/j.lwt.2021.112819
Received 20 July 2021; Received in revised form 7 November 2021; Accepted 13 November 2021

2. Materials and methods

2.1. Materials

The commercial ingredients employed in batters were rice flour (RF); cassava (CS) and maize (MS) starches (Santa María, La Plata, Argentina), sugar, sodium chloride, sunflower oil, vanilla essence and whole milk, all acquired from a local market. Besides, Walnut flour (557 g/kg lipids, 246 g/kg proteins, 94 g/kg dietary fiber content, 27 g/kg ashes in dry basis) (Aceites del Desierto, Córdoba, Argentina), dehydrated egg (Ovobrand, Brandsen, Argentina), Hydroxypropylmethylcellulose HPMC F4M (Dow Chemical, Midland, USA) and distilled water were used.

2.1.1. Formulations of premixes

The control premix was prepared by mixing RF (0.70 kg), MS (0.15 kg), and CS (0.15 kg). The walnut flour was added at 100, 150 and 200 g/kg respect to the control premix (PWF10, PWF15, and PWF20, respectively).

2.1.2. Formulations of batters

The following flour blend was employed: 700 g rice flour, 150 g cassava starch and 150 g maize starch. Additionally, the following ingredients were added: 664 g water, 500 g sugar, 400 g milk, 111 g dehydrated egg, 110 g sunflower oil, 22 g HPMC, 11 g salt, 2.5 g vanilla essence per each kg of flour blend. The WF was added on three levels 100, 150, and 200 g/kg flour blend (WF10, WF15, and WF20, respectively). As WF has high lipid content, the amount of sunflower oil used was progressively reduced to keep the fat content close to 110 g/kg while WF increased.

2.2. Methods

2.2.1. Pasting properties of premixes

The viscoamylographic behavior of raw materials and premixes was assessed with a RVA 4500 (Perten Instruments, Sydney, Australia) using the STD1 program which is in accordance with the 76-21.01 approved method (AACC, 2007).

The assay consisted in weighing 3.0 g of sample in an aluminum container and then adding 25.0 g distilled water (140 g/kg dry basis). The components were immediately mixed by vertically shaking the stirrer ten times. Then, the container was placed in the measuring position and the STD1 program started. The following parameters were determined using the specialized software Thermocline for Windows (TCW) v3.16.1.387 (Perten Instruments, Sydney, Australia) provided with the instrument: pasting temperature (T_p), pasting time (t_p), peak viscosity (η_p), minimum viscosity or through (η_{min}), breakdown or difference between peak and minimum viscosity ($\eta_p - \eta_{min}$), final viscosity (η_f), setback 1 or the difference between final and minimum viscosity ($\eta_f - \eta_{min}$) and setback 2 or the difference between final and peak viscosity ($\eta_f - \eta_p$). Each sample was tested in triplicate.

2.2.2. Batter preparation

In a planetary mixer Kenwood Major-1200W (Kenwood Ltd., Havant, UK) the reconstituted egg was whipped (speed 2 for 30 s). Milk, oil and water were added (speed 2 up to 2:30 min). Gradually the solid ingredients were added (speed 2 up to 6 min). Then, the speed was increased to 3 up to 7:30 min. In the case of batter with walnut flour, WF was moistened with half of milk and was added with the solids. Each batter formulation was prepared at least in duplicate.

2.2.3. Particle size distribution of oil in the batters

A Mastersizer 2000E (Malvern Instruments Ltd., Malvern, UK) equipped with the accessory Hydro 2000 MU (pump speed: 2000 rpm) was used to evaluate the particle size distribution present in batters. Samples were suspended in distilled water (100 mL/L H_2O) and an ultrasonic treatment (ultrasonic displacement 10 μm for 10 s) was applied to favor the dispersibility. The particle size was estimated in terms of the D(0.5) (50th percentile) which was obtained from the surface-weighted distribution using the Mastersizer 2000E software (v5.54). The assay was performed in duplicate.

2.2.4. Hydration properties of batters

2.2.4.1. Water availability and content. Water activity (Aw): Measurements were performed at 25 °C in an AquaLab (Decagon Devices Inc., Pullman, USA).

Water content was determined by dry at 105 °C laboratory oven (San Jor, San Martín, Argentina) until constant weight.

2.2.4.2. ^1H NMR relaxation measurements. Spin-spin ^1H relaxation times (T_2) were determined with a low-resolution MiniSpec pc120-spectrometer (Bruker, Billerica, MA, USA) using Carr–Purcell–Meiboom–Gill (CPMG) sequence with an interpulse spacing of 200 μs. For this purpose, batter was prepared in duplicate, and each duplicate was measured in different NMR tubes. Also, each tube was measured in duplicated, rotating it 90° between consecutive measurements (Arp, Correa, & Ferrero, 2018).

The transverse relaxation curves were fitted with the next exponential equation:

$$I_{(t)} = I_o + \sum_i F_{Pi} \exp\left(-\frac{t}{T_{2i}}\right) \quad \text{(eq. 1)}$$

where I_o is the intensity of the residual decay signal, T_{2i} and F_{Pi} represent the relaxation time and the fraction of protons relaxing with T_{2i} spin-spin time, respectively.

2.2.5. Differential scanning calorimetry: phase and state transitions of frozen batters

Thermal properties of batters were determined with a differential scanning calorimeter DSC Q100 (TA Instruments, New Castle, DE, USA). Approximately, ten mg of samples were weighed in aluminum hermetic pans. An empty pan was used as reference. Samples were cooled from 5 to −50 °C at 5 °C/min, then kept isothermal for 5 min, after which they were heated up to 10 °C at 5 °C/min. Thermograms were analyzed during the heating process using the Universal Analysis 2000 software (TA Instruments, New Castle, DE, USA). In the batters were evaluated the glass transition (T_g), the change on the heat capacity (ΔC_p) and, the ice melting (T_m) and freezing point temperatures. These ones were taken as the onset and peak temperatures, respectively (Ronda & Roos, 2008; Roos & Karel, 1991). Besides, the frozen water fraction (F_W) (g of ice/kg total water) was derived using equation (2).

$$F_W = \frac{\Delta H_m}{(\Delta H_{ice\ m} \Delta W_T)} \quad \text{(eq. 2)}$$

where ΔH_m is the enthalpy of ice melting in the maximally freeze-concentrated system (J/kg batter), $\Delta H_{ice\ m}$ the latent heat of ice melting (333.9 J/g ice) and W_T (kg total water/kg batter)

2.2.6. Textural measurements

Back extrusion essays were performed according to Cevoli, Balestra, Ragni, and Fabbri (2013) with some minor modifications. A texture analyzer TAXT-2i (Stable Micro Systems, Godalming, UK) with a load cell of 5 N was employed. Batter was poured into a cylindrical container (53.3 mm inner diameter) and was tested with a cylindrical probe (10 mm height, 45 mm width) at 24 ± 1 °C. The test was conducted at a pre and post-test speed of 2.5 mm/s, test speed of 1 mm/s, distance of 10 mm and trigger force of 0.5 N. The following parameters were determined from the curve: firmness [maximum positive force (N)], consistency index [positive area of the curve (N•s)], viscosity index [negative area of the curve (N•s)], and cohesiveness [maximum negative force (N)].

2.2.7. Viscoelastic behavior of batters

Dynamic oscillatory tests were carried out in an AR G2 rheometer (TA Instruments, New Castle, DE, USA) with parallel-plate geometry (gap 1 mm, diameter 40 mm). Stress and frequency sweeps were performed at 25 °C. The stress sweeps were performed in the range between 0.01 and 100 Pa at constant frequency of 1 Hz in order to determine the linear viscoelastic range (LVR) of each batter. In addition, from these curves the critical stress was determined as the value to produce a decreased of 5% of G_{max}', from this critical G' (G'_C) the critical strain (γ_C) was determined. Then, cohesive energy (E_C) was calculated as:

$$E_C = 1/2 G'_C \cdot \gamma_C \qquad \text{(eq. 3)}$$

The frequency sweeps (from 0.1 to 100 Hz) were performed at a 0.2 Pa within the LVR.

From the mechanical spectrum the storage (G′), loss (G″) and complex (G*) moduli were obtained as a function of frequency.

The dynamic complex modulus G* was fitted to the weak gel model proposed by Gabriele, de Cindio, and D'Antona (2001) (eq. (4)). In this approach, the food system is assumed to be a three-dimensional network composed by flow units that interact cooperatively.

$$G^*(\omega) = \sqrt{G'(\omega)^2 + G''(\omega)^2} = A_F \omega^{1/z} \qquad \text{(eq. 4)}$$

where z is the coordination degree and A_F represents the strength of the interactions among the flow units(Gabriele et al., 2001).

All the experiments were performed at least in duplicate.

2.2.8. Microstructure: confocal scanning laser microscopy (CSLM)

A non-covalent dyeing of samples was performed. The following four fluorophores were employed: rhodamine B (10 mg/L), calcofluor (0.1 g/L), fluorescein isothiocyanate (FITC) (0.1 g/L), and nile red (NR) (1 g/L). FITC and NR were not used together. Batter was spread onto a glass slide using a second slide and then a solution containing the fluorophores was added. Samples were rest for an hour in darkness. Finally, samples were washed with distilled water and covered with a glass cover slip. Then, all samples were observed with an FV1000 confocal microscope (Olympus, Shinjuku, Tokyo, Japan).

The excitation wavelengths were 488 nm, 568 nm and 347 nm, and emission wavelengths 518 nm, 625 nm and 450 nm for (FITC and NR), rhodamine B and calcofluor, respectively (Correa, Ferrer, Añón, & Ferrero, 2014). Micrographs were taken at 20x and batters without tinction did not show autofluorescence.

2.2.9. Statistical analysis

The non-parametric Kruskal-Wallis test was used for the determination of significant differences among samples (α: 0.05), with this purpose the InfoStat 2020e software was employed (Di Rienzo et al., 2011) (UNC, Córdoba, Argentina). The fitting of the rheological (eq. (4)) and ^{1}H NMR (eq. (1)) data to the models, as well as the comparison among datasets (F test) were performed with OriginPro8 v8.0724 (OriginLab Corporation, Northampton, MA, USA)

3. Results and discussion

3.1. Pasting properties of premixes

The viscoamylographic profiles of raw materials and premixes are depicted in Fig. 1. Walnut Flour (WF) did not swell and kept a constant viscosity around 0.012 Pa s during the entire test (not shown).

When starchy food is heated and then cooled, a series of physicochemical changes occur in excess water. The rice flour (RF), and cassava (CS), and maize (MS) starches showed a typical pasting profile (Fig. 1 A). Although, there were significant differences among their behavior. The stirring and the rise of temperature led to an increase in viscosity due to the swelling of granules. The peak viscosity of RF, CS, and MS was 4.31 ± 0.02, 4.39 ± 0.02 and 2.80 ± 0.06 Pa s, respectively. Besides, CS and MS developed their peak viscosity earlier than RF, as shown by the time to peak (4.13 ± 0.07, 4.53 ± 0.00 and 6.07 ± 0.09 min, respectively). After that, viscosity fell because with constant stirring and heating, more granules rupture and fragment (Bao & Bergman, 2018). On cooling, RF and MS showed an increase in final viscosity with respect to peak viscosity (Final viscosities: CS: 2.98 ± 0.08, MS: 3.27 ± 0.09 and RF: 6.76 ± 0.07 Pa s). This behavior led to higher setback values for RF (2.45 ± 0.05 Pa s) and MS (0.47 ± 0.04 Pa s) than CS (−1.4 ± 0.1 Pa s). The final viscosity value is associated with the formation of a gel by the retrograded amylose (Balet, Guelpa, Fox, & Manley, 2019). These results showed that amylose of rice flour has a higher tendency to retrograde (Kaur, Singh, McCarthy, & Singh, 2007).

The control premix was composed by RF (0.70 kg), CS (0.15 kg) and MS (0.15 kg) and its pasting behavior was a clear combination of its constituents (Fig. 1 B). When WF was added, the premixes had a later initiation of pasting, which was reflected in the increase of the peak time (Fig. 1 B). Besides, the pasting temperature increased along with WF content (Table 1).

In addition, peak and minimum viscosities and breakdown decreased significantly with the addition of WF. This effect could be related to a higher heating and shear stress resistance of these premixes in the presence of WF due to a restrained swelling of starch granules, combined with a dilution effect of total starch content (Burbano & Correa, 2021; Fu et al., 2020)

Otherwise, on cooling, the high final viscosity values of all samples indicate the formation of a gel network (Bao & Bergman, 2018). Likewise, the final viscosity increased when comparing control with PWF10, but a higher WF addition reduced the strength of the network. Finally, the values of setbacks 1 and 2 increased in samples with WF. In samples formed only by starches, this behavior evidences a higher tendency to retrograde. But in samples with lipids, this increment in the setbacks could also be an indicator of the formation of the amylose-lipid complex (Gelders, Goesaert, & Delcour, 2006; Okumus, Tacer-Caba, Kahraman, & Nilufer-Erdil, 2018) or it could be related to melting and re-solidification of fatty acid during RVA heating-cooling cycle (Ai, Hasjim, & Jane, 2013).

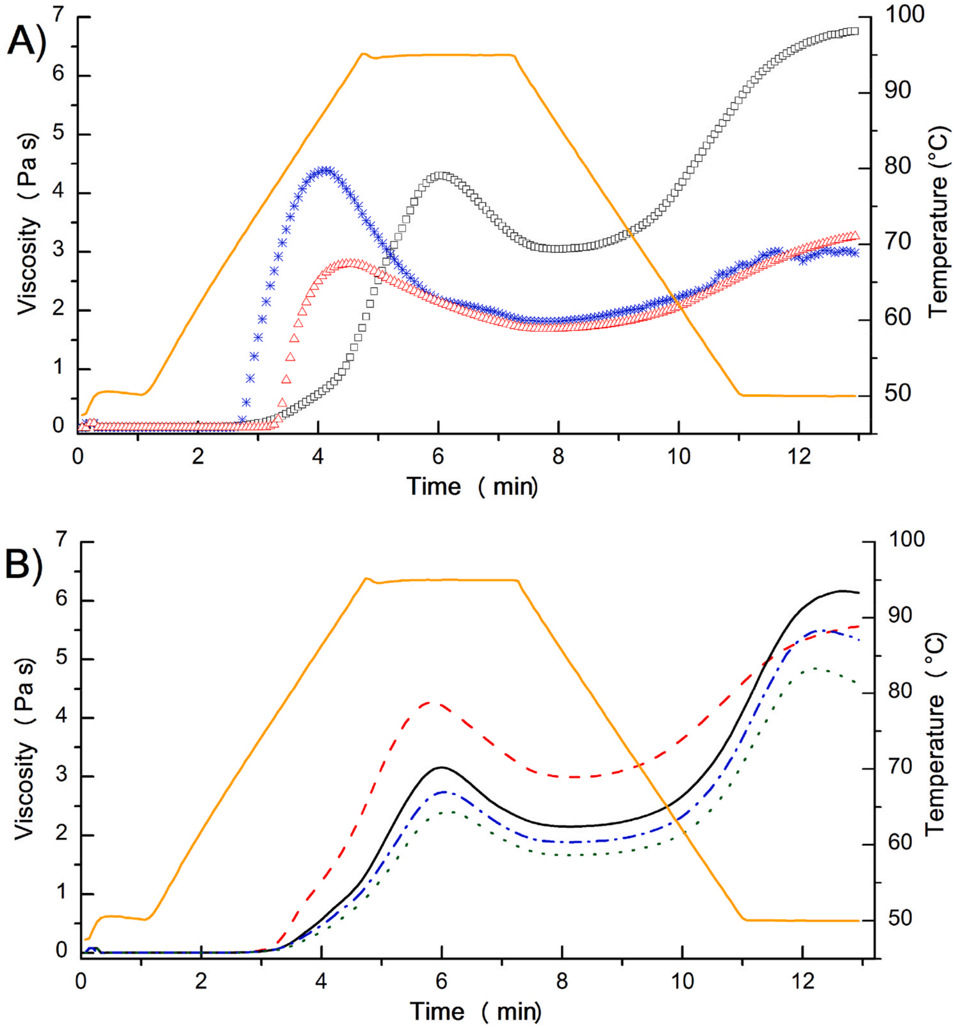

Fig.1. Viscoamylographic profiles of A) raw materials: rice flour (RF), cassava (CS) and maize (MS) starches, and B) control and walnut flour premixes (PWF). Control premix: rice flour (0.7 kg), cassava (0.15 kg) and maize (0.15 kg) starches. Addition levels of walnut flour premixes: 100, 150 and 200 g walnut flour/kg control premix named as PWF10, PWF15, and PWF20, respectively. ——— Temperature ramp ☐ RF ✳ CS △ MS - - - Control premix ——— PWF10 -·-·- PWF15 ······ PWF20

3.2. *Particle size distribution of oil in the batters*

Cake batters are complex systems since they consist of air bubbles dispersed in an aqueous phase (foam) and, simultaneously, oil droplets dispersed in this continuous aqueous phase (emulsion). The foams and emulsions are thermodynamically unstable systems and are mainly destabilized by the same process (Langevin, 2019). All the factors that contribute to stabilizing foam and emulsions are helping to the maintenance of batters matrix in time (Sumnu & Sahin, 2008, p. 304).

The particle size distribution of the oil droplets found in batters is shown in Fig. 2. Besides, the particle size distribution of WF was added to an easily evaluation of the role of WF in the lipid size distribution in batters. All batters presented two droplets populations; the ones presented in the control batter were larger, which is reflected in the D(0.5) value. The D(0.5) for the control sample was 6.869 μm, while in the case of batters with WF, a significant shift to lower D(0.5) values was observed (2.729 μm–3.481 μm). Thus, WF contributed to obtaining oil droplets of smaller size, which could help to a better dispersion of lipids and a higher homogeneity of the batter matrix. The different droplet pattern is observed in the CSLM micrographs included in Fig. 2. The presence of oil droplets of smaller size contributes to higher emulsion stability since oil droplets of larger size tend to coalesce. When droplets are larger, they are more prone to collide and the attractive forces involved in the collision are greater (McClements, 2015). Thus, WF

Table 1
Pasting parameters of control and walnut flour premixes (PWF).

	Pasting Temperature	Time to Peak	Viscosities (Pa·s)			Secondary parameters (Pa·s)		
			Peak	Minimum	Final	Breakdown	Setback 1	Setback 2
	(°C)	(min)	η_p	η_{min}	η_f	$\eta_p - \eta_{min}$	$\eta_f - \eta_{min}$	$\eta_f - \eta_p$
Control	75.6 ± 0.4^{a}	5.82 ± 0.04^{a}	4.26 ± 0.05^{c}	2.99 ± 0.06^{c}	5.57 ± 0.04^{bc}	1.27 ± 0.03^{c}	2.58 ± 0.04^{a}	1.30 ± 0.00^{a}
PWF10	78.27 ± 0.03^{ab}	5.98 ± 0.04^{ab}	3.16 ± 0.03^{bc}	2.15 ± 0.02^{bc}	6.14 ± 0.06^{c}	1.01 ± 0.03^{bc}	3.99 ± 0.04^{c}	2.98 ± 0.04^{c}
PWF15	78.23 ± 0.04^{ab}	6.04 ± 0.04^{b}	2.74 ± 0.03^{ab}	1.88 ± 0.03^{ab}	5.34 ± 0.08^{ab}	0.86 ± 0.01^{ab}	3.45 ± 0.05^{bc}	2.60 ± 0.05^{bc}
PWF20	79.08 ± 0.06^{b}	6.11 ± 0.04^{b}	2.40 ± 0.02^{a}	1.66 ± 0.02^{a}	4.59 ± 0.06^{a}	0.74 ± 0.01^{a}	2.93 ± 0.05^{ab}	2.19 ± 0.05^{ab}

Control premix: rice flour (0.70 kg), cassava (0.15 kg) and corn (0.15 kg) starches. Addition levels of walnut flour premixes: 100, 150 and 200 g walnut flour/kg control premix named as PWF10, PWF15, and PWF20, respectively. Mean ± standard deviation. Different letters in the same column indicate significant differences ($p < 0.05$, $n = 3$).

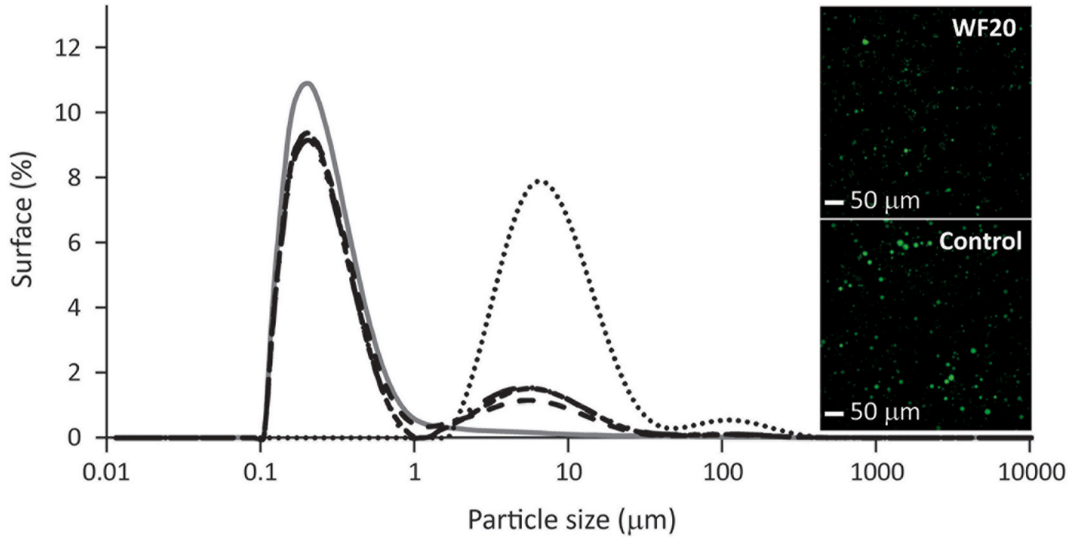

Fig.2. Particle size distribution of WF and oil droplets in batters. The confocal laser scanning micrographs correspond to control and WF20 batter dyed with Nile Red (20X). ——WF ••••••••Control ▬ ▬ ▬WF10 — — — WF15 —— • •WF20

would constitute a natural alternative to improve batter emulsion stability.

3.3. Hydration properties of batters: Moisture, water activity and mobility

Table 2 shows the water content, water activity, and the parameters obtained from the fitted of ^{1}H NMR relaxation curves. The moisture content of the control batter was 403.1 ± 0.8 g/kg. This parameter slightly decreased up to 392 ± 2 when 200 g/kg of WF was added. On the other hand, using NMR, the water mobility on dough and crumbs has been studied by several authors (Arp et al., 2018; Blanchard, Labouré, Verel, & Champion, 2012; Hager, Bosmans, & Delcour, 2014). The transverse relaxation curves obtained from a CPMG sequence were fitted with an exponential decay equation (eq. (1)). Equations from one to four exponential terms were tested but all formulations showed the best fit when single term equations were used (all datasets R^2 ranged from 0.9663 to 0.9913). The number of terms in the equation is relate to water populations with different mobility in the system (Assifaoui, Champion, Chiotelli, & Verel, 2006). Thus, in this case only one population was found. The addition of WF increased the fraction of protons (F_P) affected by the magnetic field during the test with values of 0.40 ± 0.01, 0.45 ± 0.01, 0.44 ± 0.01, and 0.47 ± 0.01 for control, WF10, WF15, and WF20, respectively. Otherwise, the relaxation times describe the mobility of water protons in the batter matrix, being lower values related to lower mobility. As the amount of more mobile water decreased, the spin-spin relaxation time also decreased. As expected, WF20 had the lowest relaxation time. Similarly, Doona and Baik (2007) found a reduction of T_2 on wheat dough when the moisture content decreased from 472 g moisture/kg dough ($T_2 = 10$ ms) to 331 g moisture/kg dough ($T_2 = 3$ ms). Likewise, the availability of water represented by water activity also decreased with the addition of WF. Proteins and fiber from WF could bound the water in batters, causing the reduction of T_2 and aw as the WF concentration increased.

Table 2
Hydration properties of batters: water activity (aw), moisture content and parameters of H NMR relaxation assays.

	Aw	Water content*	H-NMR parameters of batters		
			I_o	F_P	T_2 (ms)
Control	0.963 ± 0.002^c	403.1 ± 0.8^b	0.38 ± 0.02^c	0.40 ± 0.01^a	19 ± 1^c
WF10	0.962 ± 0.001^{bc}	399.9 ± 0.5^{ab}	0.36 ± 0.02^{bc}	0.45 ± 0.01^{bc}	16.7 ± 0.5^b
WF15	0.960 ± 0.001^{ab}	396 ± 2^{ab}	0.34 ± 0.01^a	0.44 ± 0.01^b	16.2 ± 0.9^b
WF20	0.959 ± 0.001^a	392 ± 2^a	0.35 ± 0.01^{ab}	0.47 ± 0.01^c	14.3 ± 0.9^a

Control, WF10, WF15 and WF20 contains 0, 100, 150 and 200 g walnut flour/kg flour blend. Flour blend: rice flour (0.70 kg), cassava (0.15 kg) and maize (0.15 kg) starches. **F_P:** fraction of protons relaxing. **Io:** intensity of the residual decay signal. **T_2:** spin-spin relaxation time. Mean ± standard deviation. *(*g water*/kg batter). Different letters in the same column indicate significant differences ($p < 0.05$, and n = 7, n = 3 and n = 9 for aw, water content and H NMR parameters, respectively).

3.4. Differential scanning calorimetry: Phase and state transitions of frozen batters

The DSC assays were performed with the aim to evaluate the molecular mobility in batters. The molecular mobility is affected by temperature, degree of hydration, and phase and state transitions (glass transition, melting, among others) (Roudaut, Simatos, Champion, Contreras-Lopez, & Le Meste, 2004; Sansano et al., 2018).

The glass transition was not affected by WF addition and the change in the heat capacity, ΔC_p, was near 0.47 ± 0.03 J/g solids °C for all samples. In the same manner, neither the melting temperatures nor the freezing point was affected by WF addition (Table 3). The freezing temperatures were in the range from −5.6 °C to-5.2 °C. However, the freezable water (g ice/kg total water) decreases with increasing levels of WF from 395 ± 18 for control to 212 ± 13 for WF20 (Table 3). These

Table 3
Glass transition temperature, ice melting temperature and freezable water of batters.

	Frozen state		
	T_g (°C)	T_m (°C)	F_W (g ice/kg total water)
Control	−30.1 ± 0.4	−10.4 ± 0.2	395 ± 18^b
WF10	−30.5 ± 0.4	−10.4 ± 0.3	366 ± 6^{ab}
WF15	−30.5 ± 0.5	−10.2 ± 0.1	325 ± 15^a
WF20	−30.5 ± 0.9	−10.3 ± 0.2	212 ± 13^a

Control, WF10, WF15 and WF20 contains 0, 100, 150 and 200 g walnut flour/kg flour blend. Flour blend: rice flour (0.70 kg), cassava (0.15 kg) and maize (0.15 kg) starches. **F_W:** freezable water, **T_g:** Glass transition temperature, **T_m:** Ice melting temperature. Mean ± standard deviation. Different letters in the same column indicate significant differences ($p < 0.05$, n = 3).

Table 4
Back-extrusion parameters of control and walnut flour (WF) gluten-free batters.

	Firmness (N)	Consistency index(N·s)	Viscosity index(N·s)	Cohesiveness (N)
Control	0.62 ± 0.05^a	5.5 ± 0.5^a	3.0 ± 0.2^a	0.54 ± 0.05^a
WF10	0.61 ± 0.05^a	5.4 ± 0.5^a	3.1 ± 0.2^a	0.55 ± 0.05^a
WF15	0.77 ± 0.03^b	6.8 ± 0.3^b	3.6 ± 0.1^b	0.74 ± 0.07^b
WF20	1.08 ± 0.09^b	6.9 ± 0.6^b	4.5 ± 0.3^b	1.08 ± 0.09^b

Control, WF10, WF15 and WF20 contains 0, 100, 150 and 200 g walnut flour/kg flour blend. Flour blend: rice flour (0.70 kg), cassava (0.15 kg) and maize (0.15 kg) starches. Mean ± standard deviation. Different letters in the same column indicate significant differences ($p < 0.05$, $n = 9$).

Table 5
Rheological behavior of batters under small deformations: critical strain, cohesive energy and parameters of the weak gel model.

	Stress sweeps*		**Frequency sweeps:The weak gel model****		
	$\gamma_C \cdot 10^3$	$E_C \cdot 10^4$ (J/m^3)	A_F (Pa*s$^{1/z}$)	z	F test
Control	1.5 ± 0.2^b	9 ± 3^b	677 ± 4	4.87 ± 0.06	b
WF10	1.08 ± 0.06^{ab}	1.7 ± 0.5^{ab}	288 ± 9	3.7 ± 0.2	d
WF15	0.94 ± 0.07^{ab}	2.2 ± 0.1^{ab}	419 ± 10	4.2 ± 0.2	c
WF20	0.7 ± 0.1^a	1.1 ± 0.3^a	888 ± 5	4.92 ± 0.06	a

Control, WF10, WF15 and WF20 contains 0, 100, 150 and 200 g walnut flour/kg flour blend. Flour blend: rice flour (0.70 kg), cassava (0.15 kg) and maize (0.15 kg) starches. **A_F:** gel strength, **Ec:** cohesive energy, **γ_C:** critical strain, **z:** coordination number.
*Mean ± standard deviation. Different letters in the same column indicate significant differences ($p < 0.05$, $n = 2$).
** Mean ± deviation error. Different letters in the F test indicate significant differences among datasets ($p < 0.05$, $n = 2$).

results are in accord with the reduction of T_2 and aw in batters. WF provided components that interact with water reducing its availability to freeze. The same behavior was observed by Sciarini, Pérez, and León (2011) when soybean flour is added to rice and maize flour batters. The maintenance of T_g, having a lower amount of freezable water, seems to be contradictory. However, it is noteworthy to remark that the batters presented a different composition: decreasing starch content and increasing protein and fiber contents as the WF level increases. Thus, the different compositional characteristics of batters would explain that all the formulations presented the same T_g value. The glass transition presents relevance from a technological point of view since it is related to the stability of a product, being temperatures below T_g adequate to storage products (Wang & Zhou, 2017). In the case of gluten-free dough, frozen storage has not been widely studied. However, it represents a manner to add value and offers consumers fresh products at any time. Thus, this common practice in wheat-based products may increase over time in gluten-free as well (Mezaize, Chevallier, Le Bail & de Lamballeri, 2010; Ozkoc & Seyhun, 2015).

3.5. Textural measurements

Understanding the textural properties of batters is important because it shows the behavior of batters during its manipulation (e.g., mold filling). Using back-extrusion essay some textural properties were obtained (Table 4).

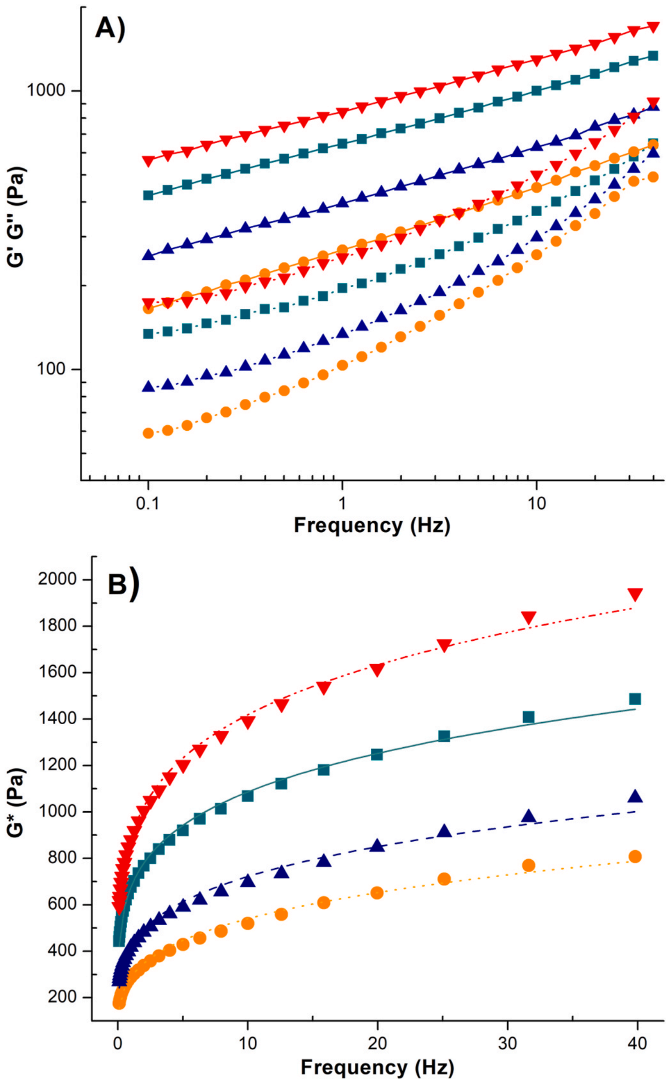

Fig. 3. Rheological behavior of batters in small oscillatory assays. Effect of frequency on: A) Mechanical spectra of batters and B) complex modulus and power law fitted curves (PLFC) Control: ■ G’ ■ G” ■ G* — PLFC, WF10: ● G’ ● G” ● G* ····· PLFC, WF15: ▲ G’ ▲ G” ▲ G* – – PLFC, WF20: ▼ G’ ▼ G” ▼ G* -·-·- PLFC

The increase of walnut flour concentration on batters, increased their firmness, consistency index, viscosity index, and cohesiveness. This trend was also observed when carrot pomace powder was included in gluten-free batters (Majzoobi, Poor, Jamalian, & Farahnaky, 2016). The 100 g/kg addition of WF was not enough to change significantly the textural parameters. However, firmness and cohesiveness almost doubled with the addition of 200 g/kg WF. Likewise, the higher consistency and viscosity indexes of WF20 batter were probably the reason that pouring that batter into beaker and molds were easier.

Batters that present low consistency are able to rise quickly, losing air bubbles which lead to cakes of low volume (Lee, Kim, & Inglett, 2005). On the other hand, batters that present extra-high batter consistency restrains the expansion of cakes, leading to lower specific volumes (Gularte, Gómez, & Rosell, 2012). This states that there might be an optimum consistency to achieve cakes with high volume. The consistency and firmness values obtained in this work by batters with WF are comparable with the ones found by other authors for products with high specific volume (Majzoobi et al., 2016).

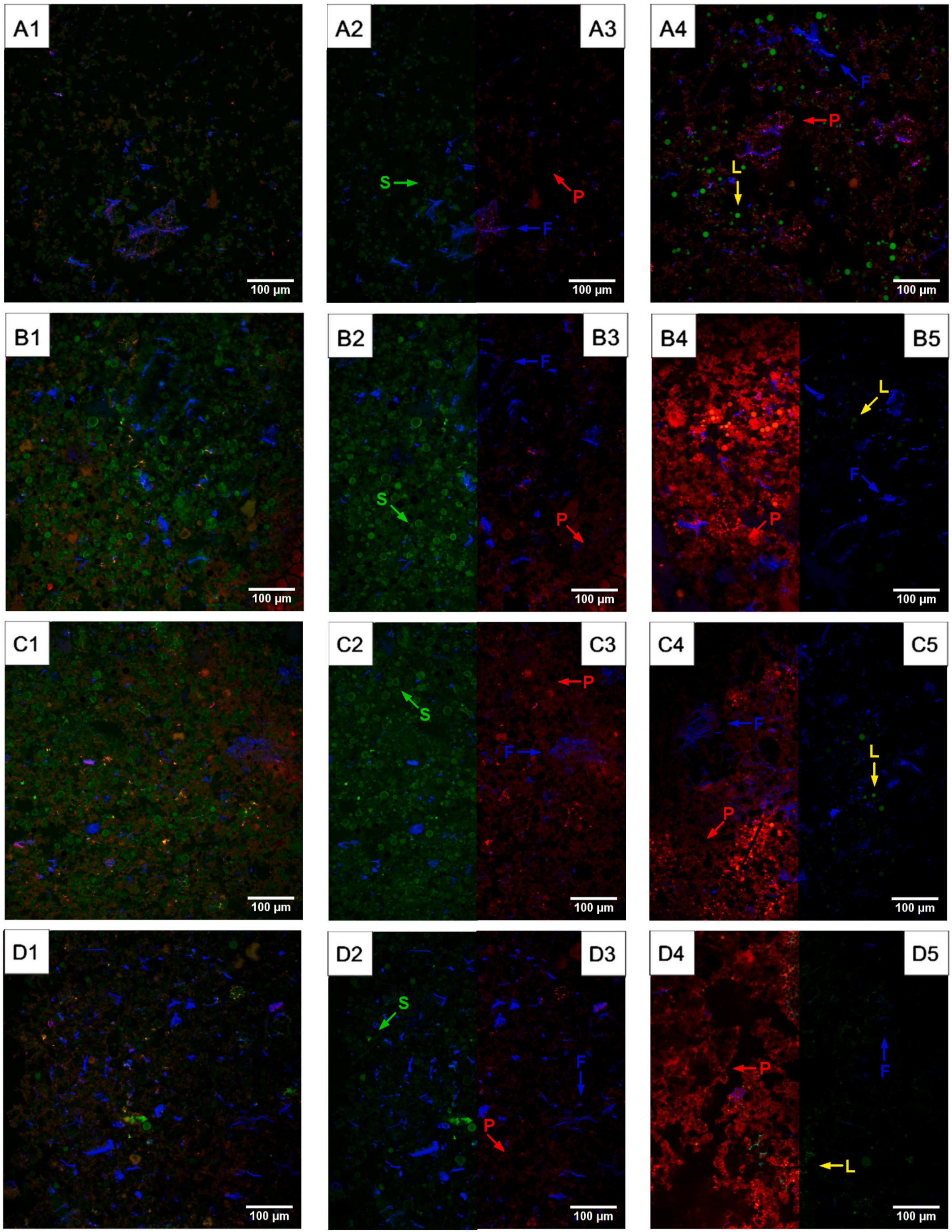

Fig. 4.

Fig. 4. Confocal scanning laser microscopy micrographs of gluten free batters at 20x. Images A, B, C and D represent Control, WF10, WF15 and WF20, respectively. The micrographs numbered as 1, 2 and 3 were dyed with Rhodamine B, FITC, and Calcofluor, while micrographs numbered as 4 and 5 were dyed with Rhodamine B, Nile Red, and Calcofluor. The micrographs numbered as 1 and 4 showed all their corresponding channels on, while 2 and 5 showed Rhodamine B channel off and micrograph numbered as 3 showed FITC channel off. F: fiber, L: lipids, P: protein and S: starch. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

3.6. *Viscoelastic behavior of batters*

Table 5 shows the rheological parameters of batters. As the WF level increased, the LVR was reduced as it was reflected by the shift of γ_C to lower values. If values beyond γ_C are applied, a significant destruction of the sample structure occurs. The cohesive energy of batters with WF tended to lower values than control but only WF20 presented significant differences respect to it. The Ec reflects the strength of a gel like structure (Tadros, 2010). In gluten-free batters, proteins dispersed in the aqueous phase contribute to the conformation of the matrix. The presence of walnut flour could interrupt this matrix leading to one more susceptible to increasing stress.

Fig. 3A shows the frequency dependence of the storage (G′) and loss (G″) moduli of batters. All formulations showed values of G′ higher than G″, a characteristic behavior of soft gels. Comparing the formulations with WF among each other, it was observed that WF produced a reinforcement effect in both moduli in the whole range of the assay as the concentration of WF increased. The control sample showed an intermediate behavior between WF15 and WF20.

The complex modulus (in the range 0.1–39 Hz) was modeled following the weak gel approach (Fig. 3B). There were found significant differences in the behavior exhibited among all samples. The parameters that characterize the weak gel model are shown in Table 5. The addition of a low level of WF led to a reduction of A_F value (WF10). However, the use of increasing levels of WF led to steadily strengthened batter's matrixes when a small deformation inside de LVR was applied. Thus, the formulation WF20 presented the highest AF value. This parameter is relevant because it is related to the gel strength. The behavior exhibited by samples in this assay was in accord with the results observed in back-extrusion assays. But in oscillatory assays control batter exhibited an intermediated behavior between WF20 and WF15 as it was evidenced by the higher A_F values of control than WF10 and WF15 samples. This different effect could be related to the fact that large deformations are applied in back-extrusion assays while small ones are applied in oscillatory assays.

The coordination degree (z) of flow units in these weak gels increased with the WF level. When WF was added at 100 g/kg a disruption of the network occurred compared to control batter. But increasing levels led to the formation of a network constituted by different flow units which was reflected in the increasing values of z and the modification of A_F. In this type of systems, the gel is formed by weak strands that connect topological points, being this network stabilized by weak interactions instead of covalent bounds (Gabriele et al., 2001). The values of z found here are comparable to the ones informed in other gluten free batters (Wolter, Hager, Zannini, Czerny, & Arendt, 2014).

The rheological behavior and the parameters obtained from the model for the batters with WF were similar to the ones informed by other authors in gluten-free batters (Mäkinen, Zannini, & Arendt, 2013; Wolter et al., 2014). Thus, WF conferred the technological features to obtain batters suitable to obtain gluten free cakes.

3.7. Batter microstructure

Batter images were acquired by confocal scanning laser microscopy at 20x (Fig. 4). The letters A, B, C, and D represent control, WF10, WF15, and WF20, respectively. The images numbered 1, 2 and 3 were dyed with a mix formed by Rhodamine B, FITC, and Calcofluor, while micrographs 4 and 5 were dyed with Rhodamine B, Nile Red, and Calcofluor.

Fluorophore FITC binds to proteins and starch granules emitting a yellowish-green color, Rhodamine B is a red dye that binds preferentially to proteins (Correa et al., 2014), Calcofluor binds strongly to structures containing cellulose (Öhgren, Lopez-Sanchez, & Lorén, 2019) and chitin, giving a blue color, which allows its association to the fiber, and finally, Nile Red binds to lipids dyeing them of colour green (Nicholson & Marangoni, 2019). FITC and Nile Red were not used together as they absorb and emit at the same wavelength, so their signal could not be distinguished.

The images numbered as 1 and 4 show all fluorophores channels turned on. These micrographs showed that all batters had a protein matrix where starch granules, particles of fiber, and lipid droplets are immersed, giving to the batter structure a heterogeneous aspect. Similar observation was reported in other gluten-free batters (O'Shea, Doran, Auty, Arendt, & Gallagher, 2013). Besides, the black zones could be due to air bubbles or, in images numbered as 1, sunflower oil droplets and walnut lipids since lipids are not dyed by the fluorophores used. In a complex combination, all these constituents represented a conjunction of several types of material systems: dispersion, solution, emulsion, and foam. The blue particles in all batters indicated the presence of fiber. In control (Fig. 4A), the fiber came mostly from rice flour and HPMC. Likewise, the addition of walnut flour increased the number of fiber (blue particles) in the micrographs (Fig. 4B, C, D), being WF20 the one with the highest level (Fig. 4D1, D2, D3).

The fluorophores channels were selectively turned on and off to differentiate the components more easily inside the batter matrix. The micrographs numbered as 2 and 5 showed Rhodamine B channel off, removing the proteins, while the images numbered as 3 showed FITC channel off, removing the starch from micrographs.

Control batter stained with Nile Red (Fig. 4A4) showed sunflower oil droplets dispersed in the matrix (green). As the WF level increased, the amount of sunflower oil was reduced with the purpose to maintain constant the content of lipids in the batters (Fig. 4B5, C5, D5). Thus, micrographs showed that the sunflower oil droplets were reduced and replaced by walnut lipids that still were trapped on some walnut remaining structures since a semi-continuous green matrix was visible. These structures surely contributed to increase consistency as it was observed in the back-extrusion assays when the WF level increased.

4. Conclusions

The addition of walnut flour (WF) greatly affected batter microstructural characteristics leading to differential molecular mobility and rheological behavior. WF presented a high-water affinity reflected in the lower relaxation times, and the reduction of water activity and freezable water of batters as the WF level increased. In the same way, the addition of WF improved the consistency of the batters, making them much easier to handle. This study may conclude that WF (200 g/kg flour blend) can successfully be incorporated into a gluten-free cake formulation without creating any negative effect. These results contribute to the knowledge and potential applications of a subproduct of walnut oil production. Based on these findings, the next step will be focus on the evaluation of the technological quality of gluten-free cakes with WF.

Declaration of interest

The authors declare not conflict of interest.

Declaration of competing interest

The authors have no affiliation with any organization with a direct or indirect financial interest in the subject matter discussed in the manuscript.

Acknowledgements

To the UNLP (X661 and X771) and MINCYT (PICT 2014–3421, PICT 2015-0007 and PICT 2016–3047) for the financial support. Authors also want to thank the kind cooperation of C. Javier Lecot in differential scanning calorimetry measurements.

References

AACC. (2007). *Approved methods of the American association of cereal chemists*. AACC.

Ai, Y., Hasjim, J., & Jane, J. (2013). Effects of lipids on enzymatic hydrolysis and physical properties of starch. *Carbohydrate Polymers, 92*(1), 120–127. https://doi.org/10.1016/j.carbpol.2012.08.092

Arp, C. G., Correa, M. J., & Ferrero, C. (2018). Rheological and microstructural characterization of wheat dough formulated with high levels of resistant starch. *Food and Bioprocess Technology, 11*(6), 1149–1163. https://doi.org/10.1007/s11947-018-2083-8

Assifaoui, A., Champion, D., Chiotelli, E., & Verel, A. (2006). Characterization of water mobility in biscuit dough using a low-field 1H NMR technique. *Carbohydrate Polymers, 64*(2), 197–204. https://doi.org/10.1016/j.carbpol.2005.11.020

Balet, S., Guelpa, A., Fox, G., & Manley, M. (2019). Rapid visco analyser (RVA) as a tool for measuring starch-related physiochemical properties in cereals: A review. *Food Analytical Methods, 12*(10), 2344–2360. https://doi.org/10.1007/s12161-019-01581-w

Bao, J., & Bergman, C. J. (2018). Rice flour and starch functionality. In M. Sjöö, & L. Nilsson (Eds.), *Starch in food* (pp. 373–419). Duxford: Woodhead Publishing - Elsevier. https://doi.org/10.1016/B978-0-08-100868-3.00010-X.

Blanchard, C., Labouré, H., Verel, A., & Champion, D. (2012). Study of the impact of wheat flour type, flour particle size and protein content in a cake-like dough: Proton mobility and rheological properties assessment. *Journal of Cereal Science, 56*(3), 691–698. https://doi.org/10.1016/j.jcs.2012.08.005

Bozdogan, N., Kumcuoglu, S., & Tavman, S. (2019). Investigation of the effects of using quinoa flour on gluten-free cake batters and cake properties. *Journal of Food Science & Technology, 56*(2), 683–694. https://doi.org/10.1007/s13197-018-3523-1

Burbano, J. J., & Correa, M. J. (2021). Composition and physicochemical characterization of walnut flour, a by-product of oil extraction. *Plant Foods for Human Nutrition, 76*(2), 233–239. https://doi.org/10.1007/s11130-021-00898-4

Catassi, C., & Fasano, A. (2008). Celiac disease. In E. K. Arendt, & F. Dal Bello (Eds.), *Gluten-free cereal products and beverages* (pp. 1–27). Burlington: Academic Press. https://doi.org/10.1016/B978-012373739-7.50003-4.

Cevoli, C., Balestra, F., Ragni, L., & Fabbri, A. (2013). Rheological characterisation of selected food hydrocolloids by traditional and simplified techniques. *Food Hydrocolloids, 33*, 142–150. https://doi.org/10.1016/j.foodhyd.2013.02.022

Correa, M. J., Ferrer, E., Añón, M. C., & Ferrero, C. (2014). Interaction of modified celluloses and pectins with gluten proteins. *Food Hydrocolloids, 35*, 91–99. https://doi.org/10.1016/j.foodhyd.2013.04.020, 0.

Di Rienzo, J., Casanoves, F., Balzarini, M., Gonzalez, L., Tablada, M., & Robledo, C. (2011). *InfoStat*. Universidad Nacional de Córdoba. https://www.infostat.com.ar.

Doona, C. J., & Baik, M.-Y. (2007). Molecular mobility in model dough systems studied by time-domain nuclear magnetic resonance spectroscopy. *Journal of Cereal Science, 45*(3), 257–262. https://doi.org/10.1016/j.jcs.2006.07.015

Fu, M., Sun, X., Wu, D., Meng, L., Feng, X., Cheng, W., et al. (2020). Effect of partial substitution of buckwheat on cooking characteristics, nutritional composition, and in vitro starch digestibility of extruded gluten-free rice noodles. *LWT-Food Science and Technology, 126*, 109332. https://doi.org/10.1016/j.lwt.2020.109332

Gabriele, D., de Cindio, B., & D'Antona, P. (2001). A weak gel model for foods. *Rheologica Acta, 40*(2), 120–127. https://doi.org/10.1007/s003970000139

Gallagher, E., Gormley, T. R., & Arendt, E. K. (2004). Recent advances in the formulation of gluten-free cereal-based products. *Trends in Food Science & Technology, 15*(3–4), 143–152. https://doi.org/10.1016/j.tifs.2003.09.012

Gelders, G. G., Goesaert, H., & Delcour, J. A. (2006). Amylose−Lipid complexes as controlled lipid release agents during starch gelatinization and pasting. *Journal of Agricultural and Food Chemistry, 54*(4), 1493–1499. https://doi.org/10.1021/jf051743c

Gularte, M. A., Gómez, M., & Rosell, C. M. (2012). Impact of legume flours on quality and in vitro digestibility of starch and protein from gluten-free cakes. *Food and Bioprocess Technology, 5*(8), 3142–3150. https://doi.org/10.1007/s11947-011-0642-3

Hager, A.-S., Bosmans, G. M., & Delcour, J. A. (2014). Physical and molecular changes during the storage of gluten-free rice and oat bread. *Journal of Agricultural and Food Chemistry, 62*(24), 5682–5689. https://doi.org/10.1021/jf502036x

Kaur, L., Singh, J., McCarthy, O. J., & Singh, H. (2007). Physico-chemical, rheological and structural properties of fractionated potato starches. *Journal of Food Engineering, 82*(3), 383–394. https://doi.org/10.1016/j.jfoodeng.2007.02.059

Labuckas, D., Maestri, D., & Lamarque, A. (2014). Effect of different oil extraction methods on proximate composition and protein characteristics of walnut (Juglans regia L.) flour. *Lebensmittel-Wissenschaft und -Technologie- Food Science and Technology, 59*, 794–799. https://doi.org/10.1016/j.lwt.2014.06.038

Langevin, D. (2019). Coalescence in foams and emulsions: Similarities and differences. *Current Opinion in Colloid & Interface Science, 44*, 23–31. https://doi.org/10.1016/j.cocis.2019.09.001

Lee, S., Kim, S., & Inglett, G. E. (2005). Effect of shortening replacement with oatrim on the physical and rheological properties of cakes. *Cereal Chemistry Journal, 82*(2), 120–124. https://doi.org/10.1094/CC-82-0120

Majzoobi, M., Poor, Z. V., Jamalian, J., & Farahnaky, A. (2016). Improvement of the quality of gluten-free sponge cake using different levels and particle sizes of carrot pomace powder. *International Journal of Food Science and Technology, 51*(6), 1369–1377. https://doi.org/10.1111/ijfs.13104

Mäkinen, O. E., Zannini, E., & Arendt, E. K. (2013). Germination of oat and quinoa and evaluation of the malts as gluten free baking ingredients. *Plant Foods for Human Nutrition, 68*(1), 90–95. https://doi.org/10.1007/s11130-013-0335-3

McClements, D. J. (2015). Emulsion stability. In *Food emulsions* (3rd ed., pp. 289–373). Boca Raton: CRC Press.

Mezaize, S., Chevallier, S., Le Bail, A., & de Lamballerie, M. (2010). Gluten-free frozen dough: Influence of freezing on dough rheological properties and bread quality. *Food Research International, 43*(8), 2186–2192. https://doi.org/10.1016/j.foodres.2010.07.030

Murray, J. A., Dyke, C. V., Plevak, M. F., Dierkhising, R. A., Zinsmeister, A. R., & Melton, L. J. (2003). Trends in the identification and clinical features of celiac disease in a North American community, 1950–2001. *Clinical Gastroenterology and Hepatology, 1*(1), 19–27. https://doi.org/10.1053/jcgh.2003.50004

Nicholson, R. A., & Marangoni, A. G. (2019). Chapter 5. Food structure development in oil and fat systems. In F. Spyropoulos, A. Lazidis, & I. Norton (Eds.), *Food chemistry, function and Analysis* (pp. 115–133). Cambridge: Royal Society of Chemistry. https://doi.org/10.1039/9781788016155-00115.

Öhgren, C., Lopez-Sanchez, P., & Lorén, N. (2019). Chapter 12. Food structure Analysis using light and confocal microscopy. In F. Spyropoulos, A. Lazidis, & I. Norton (Eds.), *Food chemistry, function and Analysis* (pp. 285–308). Cambridge: Royal Society of Chemistry. https://doi.org/10.1039/9781788016155-00285.

Okumus, B. N., Tacer-Caba, Z., Kahraman, K., & Nilufer-Erdil, D. (2018). Resistant starch type V formation in brown lentil (Lens culinaris Medikus) starch with different lipids/fatty acids. *Food Chemistry, 240*, 550–558. https://doi.org/10.1016/j.foodchem.2017.07.157

O'Shea, N., Doran, L., Auty, M., Arendt, E., & Gallagher, E. (2013). The rheology, microstructure and sensory characteristics of a gluten-free bread formulation enhanced with orange pomace. *Food & Function, 4*(12), 1856–1863. https://doi.org/10.1039/C3FO60294J

Ozkoc, S. O., & Seyhun, N. (2015). Effect of gum type and flaxseed concentration on quality of gluten-free breads made from frozen dough baked in infrared-microwave combination oven. *Food and Bioprocess Technology, 8*(12), 2500–2506. https://doi.org/10.1007/s11947-015-1615-8

Ronda, F., & Roos, Y. H. (2008). Gelatinization and freeze-concentration effects on recrystallization in corn and potato starch gels. *Carbohydrate Research, 343*(5), 903–911. https://doi.org/10.1016/j.carres.2008.01.026

Roos, Y., & Karel, M. (1991). Water and molecular weight effects on glass transitions in amorphous carbohydrates and carbohydrate solutions. *Journal of Food Science, 56*(6), 1676–1681. https://doi.org/10.1111/j.1365-2621.1991.tb08669.x

Roudaut, G., Simatos, D., Champion, D., Contreras-Lopez, E., & Le Meste, M. (2004). Molecular mobility around the glass transition temperature: A mini review. *Innovative Food Science & Emerging Technologies, 5*(2), 127–134. https://doi.org/10.1016/j.ifset.2003.12.003

Sansano, M., Heredia, A., Glicerina, V., Balestra, F., Romani, S., & Andrés, A. (2018). Influence of chitosan on thermal, microstructural and rheological properties of rice and wheat flours-based batters. *LWT-Food Science and Technology, 87*, 529–536. https://doi.org/10.1016/j.lwt.2017.09.036

Sciarini, L. S., Pérez, G. T., & León, A. E. (2011). *Estudio del efecto de diferentes aditivos sobre la calidad y la conservación de panes libres de gluten. [Study of the effect of different additives on the quality and shelf life of gluten-free breads*. https://doi.org/10.35537/10915/2637 [Spanish] Universidad Nacional de La Plata.

Sumnu, S. G., & Sahin, S. (2008). *Food Engineering aspects of baking sweet goods*. Boca Raton: CRC Press.

Tadros, T. F. (2010). *Rheology of dispersions: Principles and applications* (pp. 78–79). Wiley-VCH Verlag GmbH & Co. KGaA. https://doi.org/10.1002/9783527631568

Wang, Y., & Zhou, W. (2017). Chapter 3—non-equilibrium states and glass transitions in bakery products. In B. Bhandari, & Y. H. Roos (Eds.), *Non-equilibrium states and glass transitions in foods* (pp. 63–87). Cambridge: Woodhead Publishing. https://doi.org/10.1016/B978-0-08-100309-1.00005-5.

Wolter, A., Hager, A.-S., Zannini, E., Czerny, M., & Arendt, E. K. (2014). Influence of dextran-producing Weissella cibaria on baking properties and sensory profile of gluten-free and wheat breads. *International Journal of Food Microbiology, 172*, 83–91. https://doi.org/10.1016/j.ijfoodmicro.2013.11.015