

1           **Effect of sodium chloride, sucrose and chestnut starch on**  
2                           **rheological properties of chestnut flour doughs**

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6           **Abstract**

7           The influence of the addition of NaCl (0.6, 1.2, 1.8%, w/w), sucrose (0.6, 1.8, 3.4,  
8           5.0%, w/w), chestnut starch (5.0, 10.0, 15.0%, w/w) and NaCl-sucrose mixtures  
9           (0.6-0.6, 1.8-1.8%, w/w) on the rheological properties of chestnut flour (CF)  
10           doughs were studied using a controlled stress rheometer. Mixing and complete  
11           tests were achieved by the Mixolab<sup>®</sup> apparatus. Shear (0.01 to 10 s<sup>-1</sup>), oscillation  
12           (1 to 100 rad s<sup>-1</sup>), temperature sweep (30 to 100°C) and creep-recovery (loading of  
13           50 Pa) measurements were performed. Steady-flow curves exhibited a Newtonian  
14           plateau at <0.1 s<sup>-1</sup> that was shifted to lower shear rates with the additives.  
15           Apparent viscosities were satisfactorily fitted using Cross model. Moduli values  
16           of storage and loss decreased, at constant angular frequency, with increasing  
17           additives. Gelatinization temperatures were slightly modified. Creep-recovery  
18           data, that were fitted using Burgers model, showed that elasticity was low (23.0%)  
19           and doughs with chestnut starch presented the highest recoverable proportion  
20           (45.6%).

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22  
23           *Keywords:* Steady shear curves, viscoelasticity, gelatinization, creep, mixing,  
24           chestnut starch.

## 25 **1. Introduction**

26 The development of new gluten-free bakery products of high quality that are suitable for  
27 people with celiac disease is necessary, since there are more intolerant people to wheat  
28 proteins (Ronda, Gómez, Caballero, Oliete, & Blanco, 2009). Chestnut flour (CF), that  
29 is gluten-free, could be employed in these special diets. CF is also a good source of  
30 essential fatty acids (Borges, Carvalho, Correia, & Silva, 2007). The main disadvantage  
31 of the CF doughs is the low protein content and the absence of proteins with viscoelastic  
32 properties like gluten. However, additives and processing procedures can modify  
33 significantly the rheological characteristics of doughs (Yu & Ngadi, 2006).

34

35 The main techniques used for measuring doughs properties can be classified as  
36 empirical or fundamental. Empirical tests are traditionally carried out by means of  
37 farinograph, rapid visco analyzer (RVA), among others, and multifunctional new  
38 apparatus like Mixolab<sup>®</sup> (Jansen, van Vliet, & Vereijken, 1996). In recent years, several  
39 fundamental rheological measurements to monitoring fundamental viscoelastic  
40 properties of dough with or without additives have been used employing rheometers. In  
41 particular, shear measurements are used to evaluate the effect of mixing and type of  
42 flour on dough properties, oscillation measurements are a powerful tool for examining  
43 the fundamental viscoelastic properties of dough and creep-recovery tests are useful to  
44 establish relations with measurements carried out by empirical techniques (Campos,  
45 Steffe, & Perry, 1997; Keentok, Newberry, Gras, Bekes, & Tanner, 2002; Salvador,  
46 Sanz, & Fiszman, 2006). Empirical and fundamental properties of wheat flour doughs  
47 with additives were studied by several authors (Maache-Rezzoug, Boouvier, Allaf, &  
48 Patras, 1998; Lynch, Dall Bello, Sheehan, Cashman, & Arendt, 2009). It is necessary to  
49 understand the functions and interactions of the ingredients on doughs, such as salt

50 (sodium chloride) and sugar (sucrose), to improve the rheological properties and storage  
51 stability of the derived products (Sumnu, Ndife, & Baymdirh, 1999).

52

53 The presence of sodium chloride (NaCl), which is generally used at concentrations  
54 below 2% (flour basis, f.b.), plays an important role on dough properties. Salt acts as a  
55 flavour enhancer, toughens the protein and improves the dough tolerance to mixing  
56 (Butow, Gras, Haraszi, & Bekes, 2002). Some authors indicated that salt increases  
57 dough development time and its stability (Angioloni & Dalla Rosa, 2005; Miller &  
58 Hosney, 2008). However, fundamental rheology has been recently used to describe the  
59 salt effect on dough with opposite results, founding for wheat flour a slight reduction in  
60 the viscoelastic moduli (Larsson, 2002; Wu, Beta, & Clarke, 2006; Salvador et al.,  
61 2006). This behaviour was explained by decreasing of the inter-chain hydrophobic  
62 interactions of proteins (Melander & Horváth, 1977). These differences could be caused  
63 by the different characteristics of materials, (i.e. the moisture content and flour type),  
64 mixer types and process conditions. Salts, depending on their nature and concentration,  
65 can cause either an elevation or a depression of the gelatinization temperature, and  
66 similarly, increase or decrease the retrogradation. Salt effect on gelatinization is  
67 certainly different and more complex than the one of non ionic solutes, as sucrose  
68 (Chiotelli, Pulosio, & Le Meste, 2002).

69

70 The sucrose effect is other important factor on dough behaviour (Maache-Rezzoug et  
71 al., 1998). Sugar is mainly employed in pastrymaking and optionally in breadmaking to  
72 improve nutritional, sensory and keeping quality of products (Singh, Bajaj, Singh, &  
73 Gujral, 2002). This additive affects flavour, dimensions, colour, hardness and surface  
74 finish. In excess, sugar causes a dough softening, due partially to the competition

75 between the added sugar and the available water in the system (Gallagher, O'Brien,  
76 Scannell, & Arendt, 2003). Sugar makes the product fragile, since it tends to disperse  
77 the protein and starch molecules, thereby preventing the formation of a continuous mass  
78 (Maache-Rezzoug et al., 1998). Mizukoshi (1985) reported the existence of a threshold  
79 shear modulus value associated with the variation of sugar content (30-40%) on the  
80 dough. Sugars affect significantly the retrogradation depending on the sugar and starch  
81 types (Chang, Lim, & Yoo, 2004). Sugars delay starch gelatinization by increasing the  
82 gelatinization temperature (Mezreb, Goullieux, Ralainiria, & Queneudec, 2006). This  
83 behaviour was explained as result from the reduction of the water activity of the system  
84 due to the action of sugars as well as related to interactions among the sugars and the  
85 starch chains (Sumnu et al., 1999).

86

87 Nowadays, low sugar content in the diet is a recommendation (Singh & Mohamed,  
88 2007). CF products are high in sucrose, so the decrease in sugar contents would  
89 facilitate the opening of new markets. Sugar content can be reduced by the addition of  
90 other components like starch. The addition of potato starch (0-7.5%) to noodles doughs  
91 showed a positive influence on its rheological properties (Yu & Ngadi, 2006). The  
92 partial substitution of rice flour by pregelatinized corn starch also improved the  
93 quality of gluten-free cakes (Ronda et al., 2009).

94

95 The main aims of this work were to prepare CF doughs with different concentrations of  
96 sodium chloride, sucrose and chestnut starch using Mixolab<sup>®</sup> and to study their  
97 rheological properties by steady-shear, oscillatory, temperature sweep and creep-  
98 recovery tests. The influence of adding sodium chloride and sucrose mixtures on the  
99 rheological behaviour of CF doughs is also discussed. The study of the rheological

100 properties of these systems, for which data are not available in the literature, is of  
101 industrial and scientific interest.

102

## 103 **2. Materials and Methods**

### 104 *2.1. Raw materials*

105 Commercial chestnut flour with a moisture content of  $9.1 \pm 0.3\%$  (dry basis, d.b.) (ICC-  
106 Standard Methods No.110/1, 2008) was considered as control sample. Chemical  
107 composition (% d.b.) of the main components of the CF was obtained. The starch ( $67.4$   
108  $\pm 8.8$ ), protein ( $6.3 \pm 0.1$ ), fibre ( $3.8 \pm 1.6$ ) and fat ( $1.8 \pm 0.1$ ) contents were determined  
109 in triplicates following the AOAC approved methods No. 996.11, 979.09, 992.16 and  
110 948.22, respectively (AOAC, 1995). Sugars ( $20.7 \pm 3.4$ ) content was also determined by  
111 triplicate using high-performance liquid chromatography (HPLC) following the method  
112 applied for chestnuts by Míguez-Bernárdez, De la Montaña-Miguélez, & García-  
113 Queijeiro (2004).

114

115 Doughs of CF with several concentrations (% f.b.) of NaCl (0.6, 1.2, 1.8), sucrose (0.6,  
116 1.8, 3.4, 5.0) and chestnut starch (5.0, 10.0, 15.0) were prepared separately to evaluate  
117 the concentration effect of each ingredient on the rheological properties. Samples with  
118 NaCl-sucrose mixtures (0.6-0.6, 1.8-1.8) were also prepared. Table sodium chloride and  
119 sucrose were employed. However, chestnut starch was extracted from fresh chestnuts  
120 using a quantity of 300g of CF that was mixed with 600 mL of distilled water (ratio 1:2)  
121 following the method employed and described in detail by Moreira, Chenlo, Torres, &  
122 Prieto (2010a).

123

### 124 *2.2. Empirical techniques: Mixolab<sup>®</sup> characterization*

125 The preparation and characterization of the doughs was according to the standard  
126 method (ICC-Standard Method No. 173, 2008). Two different assays were performed  
127 by Mixolab<sup>®</sup> (Chopin Technologies, France). The first one, mixing test, consist of  
128 dough mixing at constant mixing rate during 30 min at 30 °C and gives similar  
129 information on dough behaviour to that experiments carried out with farinograph. The  
130 second one, complete test, after a shorter (8 min) mixing step, involves a heating-  
131 cooling cycle (37 min) and gives similar information to that the experiments carried out  
132 by RVA. In both tests, CF with the corresponding additive was placed into the  
133 Mixolab<sup>®</sup> bowl, mixed at 80 rpm for sample homogenization and heated up to 30 °C. At  
134 this moment, the apparatus adds distilled water to achieve pre-fixed hydration (f.b.,  
135 corrected to 14% moisture basis). The total mass of flour and distilled water placed into  
136 bowl was 75 g. Several preliminary mixing tests were necessary to determine the  
137 optimum hydration level to reach the maximum consistency of dough (C1: 1.1 Nm)  
138 (Rosell, Collar, & Haros, 2007). Complete test has been used after determination of the  
139 optimum hydration level to characterize the dough properties as function of mixing and  
140 temperature. All assays were carried out by triplicate and the corresponding standard  
141 deviations, s, were calculated. The main parameters obtained from Mixolab<sup>®</sup> tests were  
142 previously reported (Rosell et al., 2007; Moreira, Chenlo, Torres, & Prieto, 2010b).

143

### 144 *2.3. Fundamental techniques: Rheological characterization*

145 Rheological measurements were determined by a controlled stress rheometer (MCR  
146 301, Anton Paar Physica, Austria). The measurement system consisted of parallel plate  
147 geometry (50 mm diameter, 2 mm gap). Temperature was  $30.00 \pm 0.01^{\circ}\text{C}$  (Peltier  
148 system). The CF dough, previously prepared using Mixolab<sup>®</sup> up to reach C1, was  
149 placed between the plates. The rim of the sample was coated with paraffin (Panreac

150 Química S.A.) to prevent evaporation during the measurements. A rest time of 15 min  
151 was applied to all samples before measuring. Three replicates of each measurement  
152 were made and the corresponding standard deviations,  $s$ , were calculated.

153

### 154 2.3.1. Steady-shear flow tests

155 Shear tests were performed in controlled-stress mode over the range of shear rates from  
156 0.01 to 10  $s^{-1}$ . The shear rate range was limited to allow the shear measurements ( $\geq 0.01$   
157  $s^{-1}$ ) and to avoid problems of sample loss ( $\leq 10 s^{-1}$ ). Steady-shear flow data were fitted  
158 by Cross model (Cross, 1965):

$$159 \quad \eta = \eta_{\infty} + \frac{\eta_0 - \eta_{\infty}}{1 + \left(k \dot{\gamma}\right)^{(1-n)}} \quad (1)$$

160 where  $\eta$  (Pa s) is the apparent viscosity;  $\dot{\gamma}$  ( $s^{-1}$ ) is the shear rate;  $\eta_0$  and  $\eta_{\infty}$  (Pa s) are the  
161 zero-shear and infinite-shear rate viscosities, respectively;  $k$  (s) is the time constant and  
162  $n$  is the flow index.

163

### 164 2.3.2. Oscillatory shear tests

165 Strain sweep at standard frequency of 6.28  $rad s^{-1}$  (1 Hz) were previously carried out to  
166 determine the linear viscoelastic zone below 1% strain,  $\gamma$ . Frequency sweep tests were  
167 performed from 1 to 100  $rad s^{-1}$  at 0.1% strain value to determine the storage,  $G'$ , and  
168 loss,  $G''$ , moduli (Pa). Experimental  $G'$  and  $G''$  were fitted by the following equations:

$$169 \quad \log G' = \log a' + b' \log \omega \quad (2)$$

$$170 \quad \log G'' = \log a'' + b'' \log \omega \quad (3)$$

171 where  $\omega$  ( $rad s^{-1}$ ) is the angular frequency and  $a'$ ,  $a''$ ,  $b'$  and  $b''$  are the corresponding  
172 fitting parameters.

173

### 174 2.3.3. Creep-recovery tests

175 Creep-recovery tests were carried out applying on the dough a constant stress,  $\sigma$  (50 Pa)  
176 during 60 s out of linear viscoelastic region and allowing strain recovery in 180 s after  
177 removal of load,  $\sigma = 0$  Pa. Creep data was described with creep compliance rheological  
178 parameters,  $J(t)$  (1/Pa) =  $\gamma/\sigma$  where  $\sigma$  is the constant stress during creep test (Steffe,  
179 1996). Compliance curve data of CF dough systems were fitted to the Burgers model  
180 (Burgers, 1935) by the following equations for creep and recovery phase, respectively:

$$181 \quad J(t) = J_0 + J_m (1 - \exp(-t/\lambda)) + t/\eta_0 \quad (4)$$

$$182 \quad J(t) = J_{\max} - J_0 - J_m (1 - \exp(-t/\lambda)) \quad (5)$$

183 where  $J_0$  (1/Pa) is the instantaneous compliance,  $J_m$  (1/Pa) is the viscoelastic  
184 compliance,  $t$  (s) is the phase time,  $\lambda$  (s) is mean retardation time,  $\eta_0$  (Pa s) is the zero  
185 shear viscosity and  $J_{\max}$  (1/Pa) is the maximum creep compliance. The recovery  
186 compliance,  $J_r$  (1/Pa) is calculated by the sum of  $J_0$  and  $J_m$  corresponding to recovery  
187 phase, Eq. (5). The  $J_r/J_{\max}$  ratio gives information on relative elastic part of the  
188 maximum creep compliance.

189

### 190 2.3.4. Temperature sweep tests

191 Doughs were heated from 30 to 90 °C at constant rate of 4 °C/min in the rheometer cell.  
192 The assays were performed at strain of 0.1% and frequency of 6.28 rad s<sup>-1</sup> (1 Hz). These  
193 tests allowed to determine the gelatinization temperatures range ( $T_0$  -  $T_1$ ) and to  
194 establish a comparison with data obtained using Mixolab<sup>®</sup>. The rate of weakening of  
195 proteins ( $-\alpha$ ) and gelatinization ( $\beta$ ) were calculated as the slope before  $T_0$  and between  
196  $T_0$  and  $T_1$ , respectively.

197

198 *2.4 Statistical analysis*

199 The influence of additive concentrations on the rheological properties of CF doughs was  
200 compared. Differences among means were identified by one-factor analysis of variance  
201 (ANOVA), followed by the Duncan test ( $p \leq 0.05$ ), (SPSS 18.0 statistical package).

202

203 **3. Results and discussion**

204 *3.1. Mixolab<sup>®</sup> mixing tests*

205 The influence of NaCl, sucrose, chestnut starch or mixtures of NaCl-sucrose addition on  
206 CF dough mixing properties at maximum torque value (C1:  $1.10 \pm 0.07$  Nm) was  
207 determined using mixing tests at 30°C, following the aforementioned protocol. The  
208 main mixing parameters, water absorption (WA), development time and stability, are  
209 given in Table 1. Torque in all studied doughs initially increased with mixing time until  
210 to reach C1, whose value depended on the WA. The WA of CF dough was significantly  
211 affected by addition of NaCl above 1.2%, sucrose above 3.4% and chestnut starch in all  
212 assayed contents range. The WA increased with increasing NaCl and chestnut starch  
213 concentration. This effect can be related to the high hygroscopic character of these  
214 additives. Nevertheless, it has been reported previously that the addition of NaCl on  
215 wheat flour doughs decreased WA (Hlynka, 1962), but also the presence of other salts  
216 (NaBr, NaI) gave higher WA values (Preston, 1989). Sucrose had the opposite effect,  
217 WA of CF doughs slightly decreased with increasing sugar concentration. This  
218 behaviour is restricted in this type of flour because sugar content of CF is originally  
219 high and it can inhibit the granular starch hydration and involve sucrose-starch  
220 interactions (Sacchetti, Pinnavaia, Guidolin, & Dalla Rosa, 2004).

221

222 High concentrations of NaCl (above 1.2%) increased significantly the development time  
223 and stability of CF, which is in agreement with previous studies for wheat flour (Hlynka  
224 1962; Preston, 1989; Singh et al., 2002). Doughs with salt addition showed the highest  
225 development time and stability at the consistency of 1.1 Nm. High stability values are  
226 usually related to the strength of flours (Marco & Rosell, 2008). Development time of  
227 CF without additives were significantly reduced (from 4.7 to 2.0 min) by adding of  
228 chestnut starch, but unfortunately shorter stability times (from 12.2 to 1.6 min) were  
229 also found, indicating that this dough was less tolerant to mixing than the other assayed  
230 systems (Galal, Varriano-Marston, & Johnson, 1978). However, the development time  
231 and stability of doughs fortified with chestnut starch at different levels (5.0% to 15.0%)  
232 did not presented significantly differences. In the case of sucrose, development times  
233 and stability were only significantly different at 5.0% regarding to the obtained values  
234 for CF without additives. Both parameters decreased with increasing sugar content,  
235 indicating a softening of dough regarding to CF without additives. The WA,  
236 development time and stability of CF showed no significant differences when NaCl-  
237 sucrose mixtures were added. The original dough mixing profile (CF without additives)  
238 could be achieved by adding NaCl-sucrose mixtures with the same proportion (0.6-0.6  
239 or 1.8-1.8). In all studied systems once the period of dough stability finished, the torque  
240 began to decrease because dough became less elastic, stickier, and lost gas holding  
241 properties (Levine & Boehmer, 1997).

242

### 243 *3.2. Rheometer measurements*

#### 244 *3.2.1 Steady-shear tests*

245 All CF doughs at the maximum consistency (C1) obtained previously by Mixolab<sup>®</sup> were  
246 rheologically characterized at 30 °C at different shear rates (0.01 to 10 s<sup>-1</sup>). Fig. 1 shows

247 the steady shear flow curves for CF doughs without additives and with the tested  
248 maximum amounts of NaCl (1.8%), sucrose (5.0%) and NaCl-sucrose mixtures (1.8-  
249 1.8%). CF doughs exhibited shear-thinning behaviour. Apparent viscosity of all assayed  
250 CF doughs decreased with increasing shear rate above certain values of shear rate as  
251 function of the type and concentration of additives. The apparent viscosity of CF  
252 doughs with sucrose showed the highest decreasing in apparent viscosity regarding to  
253 CF without additives in the whole range. NaCl and NaCl-sucrose mixtures also  
254 decreased the apparent viscosity at each shear rate regarding to CF without additives. In  
255 CF dough with maximum additives concentrations the Newtonian plateau is only  
256 observed at low shear rates (close to  $0.01 \text{ s}^{-1}$ ). It may be hypothesised that the addition  
257 of the maximum amounts of all studied additives can result in higher flow ability during  
258 dough preparation and handling. The Newtonian plateau  $\eta_0$  (at low shear rate  $< 0.1 \text{ s}^{-1}$ )  
259 was shifted to lower shear rates by increasing of NaCl, sucrose, NaCl-sucrose mixtures  
260 (data not shown). The presence of Newtonian plateau at low shear rates ( $10^{-5} \text{ s}^{-1}$ ) was  
261 reported for wheat flour doughs (Rouillé, Della Vallea, Lefebvrea, & Sliwinski, 2005)  
262 and also was previously identified for other gluten-free doughs like chickpea flour (Ravi  
263 & Bhattacharya, 2004).

264

265 Fig. 2 shows the flow curves for CF doughs without additives and with tested chestnut  
266 starch concentrations (5.0, 10.0, 15.0%). CF doughs with chestnut starch addition also  
267 exhibited shear-thinning behaviour. The influence of chestnut starch addition can be  
268 noticed above values of shear rate higher than  $0.1 \text{ s}^{-1}$  and particularly showed the  
269 highest differences regarding to CF without additives at shear rates from 1 to  $10 \text{ s}^{-1}$ .  
270 Apparent viscosity remains practically constant with chestnut starch addition below 0.1

271  $s^{-1}$  shear rate and it decreased with increasing additives concentration at constant shear  
272 rate above  $0.1 s^{-1}$ .

273

274 The flow curves for all assayed doughs were satisfactorily fitted ( $R^2 > 0.987$  and  $s <$   
275  $0.012 Pa s$ ) using the Cross model, Eq. (1). Table 2 shows the Cross model parameters  
276 and statistical parameters of fitting for all assayed doughs. The values of time constant,  
277  $k$ , of all studied systems increased significantly with increasing additives  
278 concentrations. An increase in  $k$  values with the presence of additives indicated a higher  
279 rate of breakdown of agglomerated structure (Ravi & Bhattacharya, 2004). The values  
280 of the flow index,  $n$ , increased regarding to CF with addition of NaCl, sucrose and  
281 chestnut starch. Doughs with the highest additives concentration showed higher  $n$   
282 values, however  $n$  values did not varied significantly at high concentration of additives,  
283 meaning that the CF doughs did not varied significantly the pseudoplastic behaviour  
284 when salt (1.2 or 1.8%), sucrose (1.8, 3.4 or 5.0%) or chestnut starch (10.0 or 15.0%)  
285 was added. The values of the flow index,  $n$ , can be considered low (from 0.10 to 0.29)  
286 for all doughs indicating the strong shear-thinning character of the samples. Also, wheat  
287 flour doughs were previously fitted using Cross model and reported  $n$  values are in a  
288 similar range (from 0.03 to 0.20) (Rouillé et al., 2005). With the additives used in the  
289 present study  $\eta_0$  value varied in a restricted range ( $204.6-253.8 \cdot 10^3 Pa s$ ). Salt and  
290 chestnut starch increased slightly the  $\eta_0$  value regarding to CF dough without additives,  
291 while sucrose imparted a reverse trend above 1.8% of concentration. The  $\eta_\infty$  values  
292 decreased significantly with increasing additives amounts. A marked decrease in  $\eta_\infty$  can  
293 indicate that high shearing disrupts the agglomerated structure (Ravi & Bhattacharya,  
294 2004). Regarding to  $k$ ,  $n$ ,  $\eta_0$  and  $\eta_\infty$  values for the studied CF doughs with NaCl-sucrose  
295 mixtures showed intermediate values among flours with NaCl and sucrose added at the

296 same level. In fact, all these parameters can be successfully evaluated with the Cross  
297 model parameters of doughs with alone NaCl or sucrose according to:

$$298 \quad (k, n, \eta_0, \eta_\infty)_{\text{NaCl-sucrose mixtures}} = \sum_{i=1}^2 x_i \varepsilon_i \quad (6)$$

299 where  $x_i$  is the weight ratio between each component concentration (NaCl or sucrose)  
300 and total additive concentration (NaCl plus sucrose) on doughs and  $\varepsilon_i$  represents the  $k$ ,  
301  $n$ ,  $\eta_0$  and  $\eta_\infty$  parameters values for CF doughs with alone NaCl and sucrose. This  
302 relationship allows the estimation by Cross model of apparent viscosity of CF doughs  
303 with NaCl-sucrose mixtures between 0.6 and 1.8% from apparent viscosity values of CF  
304 doughs fortified with sucrose or sodium chloride.

305

### 306 *3.2.2. Oscillatory tests*

307 The effects of NaCl, sucrose and chestnut starch addition regarding to CF without  
308 additives at 30°C on the evolution of storage or elastic ( $G'$ ) and loss or viscous ( $G''$ )  
309 moduli values with frequency is shown in Fig. 3, 4 and 5, respectively. All tested  
310 doughs systems, as expected, showed  $G' > G''$  throughout the frequency range indicating  
311 a gel structure.  $G'$  and  $G''$  values slightly increased with increasing angular frequency  
312 from 1 to 100  $\text{rad s}^{-1}$ . The slightly increase can be attributed to the absence of binding  
313 agents like gluten in the CF dough (Sivaramakrishnan, Senge, & Chattopadhyay, 2004).  
314 Doughs with chestnut starch addition showed the highest slopes of  $G'$  with angular  
315 frequency and this behaviour could be attributed to the fact that starch is a hydrocolloid  
316 that exhibits similar characteristics to gums (Yu & Ngadi, 2006). The increasing of  $G'$   
317 and  $G''$  slope values with addition of hydrocolloid was previously reported for rice flour  
318 doughs (Sivaramakrishnan et al., 2004).

319

320 Both moduli for all assayed doughs decreased with increasing additives amount at  
321 constant angular frequency. The reduction of  $G'$  and  $G''$  with increasing NaCl and  
322 sucrose addition are in accordance with those reported by Salvador et al. (2006) and  
323 Angioloni & Dalla-Rosa (2004) for wheat flour doughs, while Yu & Ngadi (2006)  
324 reported that  $G'$  increases with increasing starch addition. However, slight changes were  
325 observed in the slope of both moduli with increasing frequency or the relative  
326 contribution of the both elastic and viscous components ( $\tan \delta$ ) and consequently the  
327 viscoelastic behaviour of the systems with NaCl, sucrose and NaCl-sucrose remain  
328 practically constant. In all assayed range of frequency for these additives the  $\tan \delta$   
329 values increased from  $0.18 \pm 0.01$  for  $1 \text{ rad s}^{-1}$  to  $0.21 \pm 0.01$  for  $70 \text{ rad s}^{-1}$ . These constant  
330  $\tan \delta$  values mean that doughs exhibit the same degree of cross-linking and that no  
331 major structural changes take place with the addition of NaCl and sucrose. This effect  
332 was previously reported for wheat noodle dough system with salt (Wu et al., 2006). In  
333 opposite way, the addition of chestnut starch varied significantly the dependence with  
334 increasing frequency and the relative contribution of both moduli ( $\tan \delta$  from  $0.25 \pm 0.01$   
335 for  $1 \text{ rad s}^{-1}$  to  $0.18 \pm 0.01$  for  $70 \text{ rad s}^{-1}$ ), which showed the highest reduction in  $G'$   
336 values at low angular frequencies.

337

338 The values of  $G'$  and  $G''$  showed a pronounced increase above  $70 \text{ rad s}^{-1}$  for some  
339 samples (doughs of CF without additives and with the lowest additives amounts). This  
340 behaviour can be related to the sample slippage on the rheometer plate at the highest  
341 frequencies. Therefore oscillation tests were modelled by Eqs. (2) and (3) in the range  
342 of angular frequency from 1 to  $70 \text{ rad s}^{-1}$ . The values of  $a'$ ,  $a''$ ,  $b'$ , and  $b''$  parameters of  
343 calculated from Eqs. (2) and (3) and the coefficients of fitting ( $R^2 > 0.99$  for  $G'$  and  $R^2$   
344  $> 0.98$  for  $G''$ ) for all tested preparations are given in Table 2. The values of  $a'$  and  $a''$

345 (corresponding to  $G'$  and  $G''$  values at  $1 \text{ rad s}^{-1}$  of angular frequency) decreased  
346 significantly with increasing amounts of each additive. Linear relationships for  
347 decreasing of  $a'$  (elastic) and  $a''$ (viscous) with sucrose and NaCl-sucrose content and  $a''$   
348 with chestnut starch addition were found. Tested doughs showed practically  $b'$  constant  
349 value ( $0.11 \pm 0.02$ ), except for doughs with chestnut starch ( $b'$ :  $0.21 \pm 0.01$ ), while  $b''$   
350 ( $0.15 \pm 0.03$ ) exhibited constant values for all assayed doughs. The increase of  $b'$  with  
351 chestnut starch addition can be attributed to interactions between starch granules in high  
352 concentration systems (Lii, Shao, & Tseng, 1995).

353

### 354 3.2.3. Creep-recovery tests

355 Fig. 6 shows the influence of addition of NaCl (1.8%), sucrose (5.0%), NaCl-sucrose  
356 mixtures (1.8-1.8%) and chestnut starch (15.0%) on the creep-recovery curves of CF  
357 doughs, where recovery phase was shortened to the first 120 s in order to improve the  
358 visualization. All assayed curves exhibited viscoelastic behaviour with similar shapes  
359 and  $J(t)$  values (from 0.0005 to 0.007  $1/\text{Pa}$ ) to those reported for gluten free  
360 formulations curves (from 0.0002 to 0.005  $1/\text{Pa}$ ) (Lazaridou, Duta, Papageorgiou, Belc,  
361 & Biliaderis, 2007) and lower than those found for rice flour doughs curves (from 0.004  
362 to 0.028  $1/\text{Pa}$ ) (Sivaramakrishnan et al., 2004). The maximum creep strains achieved by  
363 creep tests were ranged from 17 to 33% depending on the type and concentration of  
364 additive. The most pronounced variations were obtained for doughs with chestnut starch  
365 addition. CF doughs with NaCl and sucrose showed significant variations, but in a  
366 narrow range. This result is in agreement with the obtained results by oscillatory and  
367 steady-shear flow tests. In the same way, in the literature no significant differences were  
368 observed in creep and recovery tests for wheat flour doughs with NaCl (1.2 and 2.4%)  
369 and sucrose (10 and 20%) addition (Salvador et al., 2006).

370

371 The compliance curve data of all tested doughs were successfully fitted ( $R^2 > 0.984$  and  
372  $s < 0.025$  Pa s) to the Burgers model, Eqs. (4) and (5), and the corresponding parameters  
373 are collected in Table 2. The analysis of the parameters for creep phase, Eq. (4),  
374 indicated that instantaneous and maximum compliance,  $J_0$  and  $J_{max}$  (directly related to  
375 maximum strain), decreased linearly with increasing NaCl, sucrose and NaCl-sucrose  
376 mixtures concentration.  $J_0$  also decreased significantly with chestnut starch addition, but  
377 it remained constant with increasing concentration, while  $J_{max}$  increased significantly  
378 with increasing chestnut starch amount. On the other hand,  $J_m$  decreased slightly with  
379 increasing additives concentrations, except for chestnut starch that increased and the  
380 retardation time also increased with addition of additives, especially for chestnut starch  
381 addition (from 3.8 to 8.5 s). Finally, the flow resistance (viscosity parameter,  $\eta_0$ ) also  
382 varied in a restricted range of values ( $18.5$ - $23.8 \cdot 10^3$  Pa s) with additives addition. This  
383 parameter increased significantly regarding to CF without additives with NaCl above  
384 1.8%. It also increased significantly with increasing sucrose above 1.8% and NaCl-  
385 sucrose also at 1.8%. In the case of chestnut starch addition,  $\eta_0$  increased significantly at  
386 5.0% regarding to CF without additives, but no significant differences were observed  
387 above 10% concentration.

388

389 The creep-recovery curves showed qualitatively similar shape, except for chestnut  
390 starch addition, and the unrecoverable viscous proportion was larger than the  
391 recoverable elastic proportion independently on additive employed. This result is  
392 characteristic of a weak cross-linked polymer system (Moreira et al., 2010b). Recovery  
393 phase data, presented in Table 2, clearly showed with low  $J_r/J_{max}$  values the lack of  
394 elasticity of CF dough, which improves significantly with chestnut starch addition.  $J_m$

395 and  $\lambda$  increased significantly with additives regarding to CF, especially for chestnut  
396 starch addition. The  $J_m$  parameter decreased in a restricted range ( $111.1 \cdot 10^{-5}$  to  $78.8 \cdot 10^{-5}$   
397 1/Pa) with increasing NaCl, sucrose and its mixtures. The opposite behaviour was found  
398 for CF doughs with chestnut starch addition. The  $J_v/J_{max}$  ratio, which is a measure of  
399 dough elasticity, also varied in a restricted range (20.3 to 23.7%) with NaCl, sucrose  
400 and NaCl-sucrose mixtures addition and it must be considered low compared to these  
401 ratios for wheat flours that has been found around 65 % (Sivaramakrishnan et al, 2004).  
402 The  $J_v/J_{max}$  ratio ( $45.4 \pm 0.2$  %) improves significantly with chestnut starch addition  
403 independently of concentration.

404

#### 405 3.2.4. *Temperature sweep tests*

406 The influence of NaCl (1.8%), sucrose (5.0%), chestnut starch (15.0%) and NaCl-  
407 sucrose mixtures (1.8-1.8%) on the evolution of the storage modulus ( $G'$ ) with  
408 temperature of CF doughs is presented in Fig. 7. All assayed CF dough with additives  
409 showed lower  $G'$  values than CF without additives at each studied temperature. Below  
410 65 °C,  $G'$ , for all tested CF doughs, gradually decreased as temperature increased,  
411 indicating softening of the dough. These changes are caused by heat-induced processes  
412 and protein weakening (Rosell et al., 2007). Thereafter, the storage modulus began to  
413 increase rapidly between 66.4 and 72.6°C, reaching a peak between 80.8 and 86.2 °C,  
414 depending on type and amount of additive and then slowly decreased. The increase of  
415 storage modulus during heating has been reported to be proportional to the starch  
416 content of the dough; indicating the physicochemical changes in heated dough are  
417 essentially due to changes in the starch fraction (Angioloni & Dalla Rosa, 2005).

418

419 The specific ranges corresponding to the gelatinization temperatures ( $T_0$  and  $T_1$ ) of all  
420 assayed CF doughs are shown in Table 2. The presence of NaCl at 1.8% concentration  
421 caused a significant rise in the inflection point temperature ( $T_0$ ), however no significant  
422 differences were noted below 1.2%. The temperatures ( $T_1$ ) at which maximum values of  
423  $G'$  appeared were also higher in the presence of NaCl at 1.8%. The effect of NaCl in  
424 delaying the starch gelatinization has been reported (Galal et al., 1978; Preston, 1989;  
425 Chiotelli et al., 2002) and different explanations for this phenomenon proposed. When  
426 salt is added to dough, it lowers water activity and increases the energy necessary for  
427 chemical and physical reactions involving water (Angioloni & Dalla Rosa, 2005). The  
428 presence of sucrose (above 1.8%) caused a delay in the appearance of  $T_0$  and  $T_1$ , which  
429 would be coherent with restricted access to the available water (Salvador et al., 2006).  
430 The highest gelatinization temperatures were achieved at 3.4 and 5.0% sucrose  
431 concentrations. Understanding the delay in starch gelatinization is crucial for improving  
432 the texture and other qualities of starch based products containing higher amounts of  
433 sucrose (Sharma, Oberoi, Oberoi, Sogi, & Gill, 2009). The effect of NaCl-sucrose  
434 mixtures in gelatinization temperatures did not varied significantly regarding to each  
435 alone additive. However, a reverse trend in  $T_0$  and  $T_1$  was found with increasing  
436 chestnut starch concentrations. As expected, both temperatures showed the lowest  
437 values of all studied systems due to the reduction of sugar content in the doughs. This  
438 behaviour was previously reported for gluten-free doughs containing different amounts  
439 of corn starch (Mariotti, Lucisano, Pagani, & Perry, 2009).

440

441 In Table 2 additional parameters like  $-\alpha$  and  $\beta$  calculated from temperature sweep tests  
442 (Fig. 7) are also provided. The  $-\alpha$  slope no varied significantly with additives, except  
443 for chestnut starch addition, where the slope values decreased significantly regarding to

444 CF without additives, but no significant variations with increasing chestnut starch  
445 concentration were found. The  $\beta$  slope decreased regarding to CF slightly with NaCl  
446 addition and significantly with increasing sucrose concentration above 3.4%. The  
447 reduction in rate of gelatinization by addition of salt and sugar on doughs was  
448 previously reported in the literature (Salvador et al., 2006; Lynch et al., 2009). The  $\beta$   
449 value for CF showed no significant differences with the rest of assayed additives.

450

### 451 3.3. *Mixolab*<sup>®</sup> complete tests

452 Fig. 8 shows the curves from *Mixolab*<sup>®</sup> complete test for CF dough fortified with NaCl  
453 (1.8%), sucrose (5.0%), chestnut starch (15.0%) and NaCl-sucrose mixtures (1.8-1.8%).  
454 Representative characteristic parameters necessary to analyze in detail the information  
455 reported in this type of figures are exposed in Fig. 8. The parameters values for all  
456 assayed systems with their standard deviations are summarized in Table 3. The C2  
457 parameter showed no significant differences with tested additives. The values of C3, C4  
458 and C5 decreased with increasing NaCl and sucrose amounts. The C3 and C4 reduction  
459 was more pronounced with the concentration of NaCl, since NaCl at 1.8% presented the  
460 same value of these parameters as sucrose at 5.0%. In opposite way, the C5 reduction  
461 was more pronounced with sucrose addition. In fact, sucrose at 5.0% showed the lowest  
462 C5 values of all studied systems. In contrast, the values of C3, C4 and C5 for the added  
463 amounts of chestnut starch and NaCl-sucrose mixtures exhibited no significant  
464 differences regarding to CF without additives, except for NaCl-sucrose mixtures at  
465 1.8% where all parameters decreased significantly regarding to CF without additives.  
466 Recently, it was reported that different wheat flours with high values of C2, C3, C4 and  
467 C5 (0.79, 2.31, 2.33 and 3.49 Nm, respectively) gave low cakes volume (Kahraman,  
468 Sakiyan, Ozturk, Koksel, Sumnu, & Dubat, 2008). Taking into account these

469 considerations, it can be established that systems with sucrose (5.0%) are more suitable  
470 to obtain cakes because presented the lowest values of these parameters. In the same  
471 way, the NaCl or sucrose addition reduced the effects of staling and crumbs firmness on  
472 breads made with this flour. Ozturk, Kahraman, Tiftik, & Koksel (2008) reported that  
473 good cookie flours should have high values of C3, C4 and C5 (2.45, 2.46, 3.38 Nm,  
474 respectively). In this case, the addition of chestnut starch or low amounts of salt or sugar  
475 to CF doughs could be adequate. Other authors previously have reported the use of  
476 corn, potato, banana and seewtsop starches as additive to improve the quality of cakes,  
477 noodles, spaghetti or frozen foods, respectively (Ronda et al., 2009; Hernández-Nava,  
478 Berrios, Pan, Osorio-Díaz, & Bello-Pérez, 2009; Yu & Ngadi, 2006; Nwokocha &  
479 Williams, 2009).

480

481 In Table 3 the parameters of  $-\alpha$ ,  $\beta$ ,  $\gamma$  and  $T_0$ - $T_1$  range obtained using Mixolab<sup>®</sup> are also  
482 given. The  $-\alpha$  and  $\gamma$  slopes are not varied significantly with additives addition, except  
483 for chestnut starch addition. In this case, rate of protein weakening decreased  
484 significantly regarding to CF without additives, but did not vary significantly with  
485 increasing chestnut starch concentration, while rate of enzymatic (amylase) degradation  
486 decreased slightly. Particularly, the addition of chestnut starch at 15% modified the  $\gamma$   
487 slope sign (-0.019 Nm/min), obtained similar values to data previously reported for rice  
488 flour dough (Rosell & Marco, 2008). The  $\beta$  slope decreased slightly with increasing  
489 NaCl and sucrose concentration and the lowest  $\beta$  value (0.25 Nm/min) of all studied  
490 systems was for sucrose at 5.0%. The  $\beta$  value for CF showed no significant differences  
491 with the rest of assayed additives. The results for  $\alpha$  and  $\beta$  slopes trends obtained in  
492 rheometer were corroborated with Mixolab<sup>®</sup> tests.

493

494 Initial and final gelatinization temperatures ( $T_0$ - $T_1$ ) of chestnut starch can be also  
495 obtained using Mixolab<sup>®</sup> by the evaluation of temperature dough when C2 and C3 are  
496 achieved (Fig. 8). Nevertheless, Mixolab<sup>®</sup> obtained systematically higher  $T_0$  ( $2.1 \pm 0.2$   
497 °C) and lower  $T_1$  ( $0.7 \pm 0.4$  °C) values comparing to those derived from temperature  
498 sweep resulting in a lower ( $T_0$ - $T_1$ ) range. The aforementioned comments about the  
499 influence of assayed additives on the trend of gelatinization temperatures obtained by  
500 rheometer were corroborated here. On the whole, it can be observed that the obtained  
501 results for CF doughs with additives in rheometer were in agreement with the obtained  
502 data by Mixolab<sup>®</sup>, except for doughs fortified with salt. In this case, contradictory  
503 results were found, since Mixolab<sup>®</sup> showed a trend to strengthen doughs with salt  
504 addition regarding to CF by increasing development time and stability values; while  
505 rheometer tests showed a reverse trend with the reduction of apparent viscosity, storage  
506 and loss moduli as well as creep-recovery curves regarding to CF. Some explanations  
507 for the occasional differences among macro (Mixolab<sup>®</sup> mixing tests) and micro  
508 (rheometer oscillatory tests) scale deformation measurements are found in the literature  
509 (Lynch et al., 2009). One explanation could be the different deformation rates and  
510 stresses used in the measurements. Empirical measurements impart high different  
511 simultaneous stresses, whereas fundamental (rheometry) only evaluates the stress in  
512 controlled measurement conditions. Nevertheless, it is generally agreed that the effect of  
513 salt depend on the quality and quantity of protein (Wu et al., 2006) and low protein  
514 dough (as chestnut flour, about 6.0%) shows decreasing elastic behaviour with  
515 increasing salt content.

516

#### 517 **4. Conclusions**

518 The addition of chestnut starch and conventional additives like salt and sugar modified  
519 the rheological behaviour of CF dough during mixing and heating-cooling operations as  
520 obtained by the Mixolab<sup>®</sup> measurements. The influence of additives on rheological  
521 properties was also studied with steady-shear, oscillatory, temperature sweep and creep-  
522 recovery tests carried out in the rheometer. In this line, apparent viscosity, storage and  
523 loss moduli decreased with increasing additives concentration. During creep-recovery  
524 tests, only doughs with chestnut starch addition showed higher elastic properties.  
525 Temperature sweep results from rheometer and the data from Mixolab<sup>®</sup> complete tests  
526 were in accordance. Acceptable agreement between empirical and fundamental assays  
527 was found, except for CF doughs with salt addition. The analysis of the results indicated  
528 that assayed additives allow improving the weak interactions between components of  
529 the chestnut flour dough, typical gluten-free flours, and this flour is more suitable for  
530 pastrymaking. Further studies are required to investigate the influence of other additives  
531 that allow improving the elastic characteristics of CF doughs.

532

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537

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670

671 **Captions for Figures**

672

673 **Fig. 1.** Experimental steady shear flow curves for CF dough without and with additives:  
674 NaCl (1.8%), sucrose (5.0%) and NaCl-sucrose mixtures (1.8-1.8%). Lines correspond  
675 to Cross model, Eq. (1).

676 **Fig. 2.** Experimental steady shear flow curves for CF dough without additives and with  
677 several chestnut starch concentrations (5.0, 10, 15.0%). Lines correspond to Cross  
678 model, Eq. (1).

679 **Fig. 3.** Experimental  $G'$  and  $G''$  data for CF dough without additives and with several  
680 NaCl concentrations (0.6, 1.2, 1.8%). Lines correspond to Eq. (2 and 3).

681 **Fig. 4.** Experimental  $G'$  and  $G''$  data for CF dough without additives and with several  
682 sucrose concentrations (0.6, 1.8, 3.4, 5.0%). Lines correspond to Eq. (2 and 3).

683 **Fig. 5.** Experimental  $G'$  and  $G''$  data for CF dough without additives and with several  
684 chestnut starch concentrations (5.0, 10, 15%). Lines correspond to Eq. (2 and 3).

685 **Fig. 6.** Creep and recovery curves for CF dough without and with additives: NaCl  
686 (1.8%), sucrose (5.0%), NaCl-sucrose mixtures (1.8-1.8%) and chestnut starch (15.0%).  
687 Lines correspond to Eqs. (4 and 5).

688 **Fig. 7.** Evolution of  $G'$  with temperature sweep for CF dough without and with  
689 additives: NaCl (1.8%), sucrose (5.0%), NaCl-sucrose mixtures (1.8-1.8%) and chestnut  
690 starch (15.0%).

691 **Fig. 8.** Mixolab<sup>®</sup> complete test curves for CF dough without and with additives: NaCl  
692 (1.8%), sucrose (5.0%) and chestnut starch (15.0%).

693