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22 **A novel laboratory scale method for studying heat**
23 **treatment of cake flour**

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28

29 **Abstract**

30 A lab-scale method for replicating the time-temperature history experienced by cake flours
31 undergoing heat treatment was developed based on a packed bed configuration. The
32 performance of heat-treated flours was compared with untreated and commercially heat-
33 treated flour by test baking a high ratio cake formulation. Both cake volume and AACC shape
34 measures were optimal after 15 minutes treatment at 130°C, though their values varied
35 between harvests. Separate oscillatory rheometry tests of cake batter at 80-100°C exhibited
36 similar behaviour to the baking tests. The gel strength parameter in the weak gel model,
37 measured at 100°C, was shown to correlate with flour quality and was identified as a
38 possible alternative to test baking as a means of assessing flour quality after heat treatment.

39

40 Keywords: baking, cake, flour, heat treatment, rheology

41

42 **Introduction**

43 The UK cake market is worth more than £1bn in sales annually (www.talkingretail.com,
44 2009). Cake is a luxury food item, enjoyed for its sweet taste and tender eating quality. The

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45 latter is achieved by cake being a solid foam, and the development and solidification of this
46 microstructure through the batter preparation and baking stages are critical to cake quality.
47 Historically, cake contained sugar and liquid in equal quantity to flour (McGee, 2004; Indrani
48 and Rao, 2008), but demand for sweet, moist cakes – particularly in the UK and USA – has
49 led to increased proportions of sugar and liquid in commercial cake recipes. The vast
50 majority of commercial recipes have a larger weight of sugar and/or liquid than flour (Premier
51 Foods, personal communication). Such recipes are termed ‘high ratio’ and are defined as
52 those containing a ratio of sugar-to-flour, or liquid-to-flour, in excess of 1 (McGee, 2004).

53

54 High ratio recipes tend to be sweeter, moister, more tender, and with a longer shelf life than
55 other recipes. The disadvantage, however, is that the large proportions of sugar and liquid
56 put stress on the structure-building components, namely flour and egg. Cakes produced with
57 base flour (*i.e.* not heat treated) tend to decrease in volume towards the end of baking and
58 subsequent cooling. In some instances the cake collapses, resulting in a dense or dipped
59 product. Loss of volume and collapse are serious problems for cake manufacturers. Heat-
60 treatment of the flour prior to baking helps prevents this collapse, giving improved final
61 product volume and stability, whilst maintaining a sweet taste (Sahin, 2008).

62

63 Although there have been some previous studies on the influence of heat treatment on the
64 physical and chemical characteristics of wheat flours (Guerrieri and Cerletti, 1996; Guerrieri
65 *et al.*, 1996; Ozawa and Seguchi, 2006; Ozawa *et al.*, 2009), the effect of heat-treatment on
66 batters, baking and cake quality is poorly understood, largely because the chemical and
67 physical changes are hard to detect (Nicholas *et al.*, 1974) and difficult to relate to individual
68 factors such as starch nature and protein content. Neill *et al.* (2012) summarized the studies
69 in this area and reported that heat treatment affects gluten extensibility and water absorption,
70 starch gelatinization and cake structure. While the precise mechanism(s) are the subject of
71 debate, the need for heat-treatment is clear, as without it less sugar and fat can be added to

72 the recipe, compromising eating quality and shelf life (Premier Foods, personal
73 communication).

74

75 In the UK the majority of cake flours are subjected to some form of heat-treatment prior to the
76 cake baking process. Heat-treatment was first reported by Mangels in 1934 as a method of
77 beneficially altering the properties of flour, and patents detailing industrial processes
78 appeared in the 1960s (Doe and Russo, 1968). Heat-treatment was widespread in industry
79 long before the phase out of the prior chlorination process in the early 1990s.

80

81 A typical industrial heat treatment process involves the following steps (Premier Foods,
82 personal communication):

- 83 1. Pre-drying the flour to below 4 wt% moisture while raising its temperature to 125-
84 140°C.
- 85 2. Holding the flour for around 20 min in a series of heated screws at 125-140°C.
- 86 3. Cooling the flour to halt the heat treatment.

87

88 Re-humidification after heat-treatment to 7 wt% moisture is necessary to minimise the
89 evolution of heat (via hydration) during subsequent batter creation, and to produce a reliable
90 product. An unavoidable consequence of hydration, however, is the formation of
91 agglomerates, and so a final milling step is necessary to achieve the desired particle size
92 distribution.

93

94 The optimal time and temperature for heat treatment in stage 2 can vary with harvest year as
95 a result of annual variation in both wheat supply and properties. Hence the optimal conditions
96 and grist have to be established each year, requiring a campaign of testing. Currently the
97 only method of assessing the quality of heat-treated flour is to test bake, using a set
98 laboratory recipe incorporating high levels of sugar and liquid, designed to test the
99 robustness of the flour. Such tests are time consuming, require specialist operators, and are

100 subject to inherent variability. Furthermore, assessment of the 'quality' of a cake is non-trivial.
101 Parameters such as volume and height are recorded quantitatively, but aspects such as
102 shape, evenness and texture are assessed qualitatively by a trained operator. Neill *et al.*
103 (2012) studied heat treatment of a flour using a fluidized bed to deliver between 5 and 60
104 minutes of heat treatment at 120°C and 130°C. They assessed the effect of heat treatment
105 by Brabender viscosity measurements, gluten extensibility, starch gelatinisation and test
106 baking of Madeira cake. Quantifiable improvement in cake quality was observed and they
107 reported an optimal heat treatment as 30 min at 130°C. They did not report results for
108 different harvests. Thomasson *et al.* (1997) heat treated flour by placing a layer of flour on a
109 tray in an oven and reported an optimal treatment as 30 min at 125°C. Different harvests
110 were again not considered.

111

112 A more rapid and reproducible method of assessing the quality of flour heat-treatment is
113 desirable. There is considerable interest in developing methods to replace test baking
114 completely, or at least to give indicators of test baking performance in order to reduce the
115 number of tests to be conducted. In particular, it is important that any methods are robust to
116 changes in wheat properties over time, i.e. not just for a single harvest, and this has largely
117 been ignored by previous work in the literature.

118

119 In this paper we describe a new protocol for replicating heat treatment of flour at the lab
120 scale, aimed at controlling the time and temperature of treatment accurately, to produce flour
121 of a similar quality to that produced commercially. In addition to its small scale, lab-scale
122 heat-treatment eliminates the additional post-processing required in the industrial process,
123 notably milling. Thus it allows the effect of heat treatment to be separated from the other
124 processing effects inherent in the industrial process.

125

126 We then address two important issues in heat treatment:

127 1. The optimal process conditions for heat-treatment. The current time and temperature
128 variables used in the industrial process generally produce good quality flour, but
129 knowledge of the optimal conditions is desired for adjusting the process between
130 harvests. Flour quality was assessed by test baking.

131

132 2. Development of a novel method of assessing flour quality. Test-baking is time-
133 consuming, requires skilled operators and has inherent variability. A method is
134 required that correlates well with baking performance but is quicker, simpler or more
135 reproducible. Ideally such a method could be implemented at an industrial mill for
136 quality control purposes. The method described here is based on estimates of batter
137 strength estimated using the weak gel model interpretation of oscillatory shear testing
138 (Gabriele *et al.*, 2001). Meza *et al.* (2011) studied batter rheology at temperatures
139 from 70-90°C and reported that commercially heat-treated flours formed stronger gels
140 in cake batter above the gelatinisation temperature than untreated flours, allowing
141 them to support larger mechanical stresses.

142

143 The paper does not contain detailed analyses of flour chemistry and functionality, as the aim
144 of the paper is to introduce the heat treatment method. Elucidation of the mechanisms
145 responsible for the improvement in flour performance caused by heat treatment will require
146 this information, in due course.

147

148 **Materials and Methods**

149 *Flours*

150 Untreated flour, labelled 'base', and commercially heat-treated wheat flours were obtained
151 from the Premier Foods mill at Selby, UK. Flours were obtained from three recent harvests.
152 Their compositions are reported in Table 1. The flour sources were not disclosed for reasons
153 of commercial confidentiality.

154

155 The particle size distributions of the base and heat-treated flours were determined by light
156 scattering using a Coulter LS230 laser diffraction particle size analyser (Beckmann Coulter,
157 Buckinghamshire, UK) fitted with a small volume module. Samples (~50 mg) were dispersed
158 in isopropyl alcohol (20 mL) and sonicated using an Ultrawave U500 ultrasound bath
159 (Ultrawave Ld., Cardiff, UK) for 1 min at room temperature to separate loosely connected
160 particles. Laser diffraction measurements were interpreted using Mie theory, with a refractive
161 index (RI) of 1.533 (Sevenou *et al.*, 2002) and an opacity value (Im) of 0.01 (Coulter, 1994).
162 The refractive index of the solvent (isopropyl alcohol) was 1.374. Almost all the particle sizes
163 lay in the range 1-200 μm . All the flours exhibited a trimodal size distribution, with a smaller
164 peak with respect to volume centred at 4 μm associated with fines, and modal peaks at
165 25 μm and 65 μm . The heat-treated flour exhibited a smaller number of particles in the third
166 mode, which is attributed to the extra milling stage employed during its processing.

167

168 *Ingredients*

169 A model high-ratio cake recipe was used for test baking. The relative quantities of flour and
170 water were adjusted for flour moisture content, and Table 2 presents the formulation used for
171 the 2006-07 harvest data as an example. Skimmed milk powder (Marvel, Premier Foods,
172 UK), margarine (Marvello, BakeMark, UK), baking powder (BEX*, ThermPhos International
173 BV, UK) and emulsifier (propylene glycol monostearate and monoglyceride, Advitagel Food

174 Ltd., UK) were supplied by Premier Foods (High Wycombe, UK). Caster sugar, whole liquid
175 eggs and salt were purchased in local shops.

176

177 *Baking method*

178 A typical batch of ingredients had a combined weight of 1.09 kg, with an unaerated volume of
179 0.91 litres. The ingredients were combined in a Hobart N50-110 planetary mixer, mixed to
180 give a slurry and then aerated in the same device. The stages were

181 (i) The dry ingredients (flour, caster sugar, skimmed milk powder, baking powder and
182 salt) were combined in the mixer, fitted with its standard whisk, at its lowest speed
183 (105 rpm). This typically took 1-2 min;

184 (ii) Whole liquid egg, emulsifier and water were added and combined separately;

185 (iii) The wet ingredients were added to the dry ingredients slowly, over a period of 1 min,
186 whilst mixing at the lowest speed.

187 (iv) The slurry was aerated by whisking at the fastest speed (550 rpm) for 6 min. The
188 effect of aeration time on bubble size distributions was reported by Chesterton *et al.*
189 (2013).

190 (v) Since fat is foam-inhibiting, the margarine was added separately as a final stage. The
191 fat was melted and added slowly, over a period of 30 s whilst mixing at a slow speed.

192 The test baking protocol required batches of four cakes. 170 g of batter was poured into each
193 pre-greased circular steel baking tin (diameter 150 mm, wall height 30 mm) and the tins
194 placed on the middle tray in a fan oven preheated to 170 °C. The cakes were removed after
195 15 min, placed on a grill and allowed to cool to room temperature.

196

197 *Cake properties*

198 *Cross-sectional images*

199 Cakes were bisected and scanned using a HP Scanjet 3570c device. Cakes were placed
200 face-down on the scanner and covered with black cloth to increase the contrast between the

201 image and background. Cake images were then removed from the background using
202 Photoshop CS5 software for presentation.

203

204 *Volume measurement*

205 Cake volume was measured using a bespoke system similar to that described by Gomez *et*
206 *al.* (2008). A computer-controlled *x-y* stage moved the cake beneath a pulsed red laser
207 diode (Type OADM, Baumer Electric Ltd., measuring range, 30-130 mm; resolution 0.1 mm)
208 in a raster fashion. Data were collected at 2 mm intervals over a 160 mm × 160 mm area and
209 analysed using a MatLab™ script which calculated volume and AACC shape parameters
210 (AACC, 1999: see Appendix).

211

212 *Rheometry*

213 Oscillatory shear measurements were performed on a Bohlin CVO120HR controlled stress
214 rheometer (Malvern Instruments, London, UK) using sand-blasted parallel plates (25 mm
215 diameter and 1 mm gap) to prevent wall slip. A thin film of silicone oil (1 Pa s) was applied to
216 the exposed sample edges to prevent water loss. After loading, each sample was held for
217 3 min before testing to allow stress relaxation and temperature equilibration. All
218 measurements were made in duplicate.

219

220 The development of gel strength in the batter at temperatures of 80, 90 and 100°C was
221 studied using a protocol similar to that reported by Meza *et al.* (2011). This set of
222 temperatures crosses the range experienced by the batter during cooking as the cake
223 structure is formed and set by bubble expansion, starch gelatinization and protein
224 denaturation. Frequency sweeps were performed over the range 0.01–1 Hz. Stress sweeps
225 (0.1–5 Pa) were performed at the highest frequency (1 Hz) prior to each frequency sweep in
226 order to identify the region of linear viscoelasticity. The elastic modulus, G' , viscous modulus

227 G'' , complex modulus, $|G^*|$ and complex viscosity, $|\eta^*|$, were determined in the linear
228 viscoelastic region.

229

230 Steady shear rheology tests were performed at 20°C using the same tools and loading
231 technique over the shear rate range 0.05-50 s⁻¹, as described by Meza *et al.* (2012).

232

233 *Lab-scale heat treatment protocol*

234 The primary requirements of the method were that flour-air contact was high, temperature
235 changes could be effected quickly, and that a quantity of flour (approx. 300 g) sufficient for a
236 baking test could be obtained from a heat-treatment experiment. Fluidisation was initially
237 trialled but it proved impossible to fluidise flour satisfactorily due to its cohesiveness: at low
238 bed heights channelling occurred, and at high bed heights the flour formed a plug. Previous
239 workers such as Brekken *et al.* (1970) have reported the use of agitators within a fluidized
240 bed to combat this behaviour but this route was not pursued here. Neill *et al.* (2012) used a
241 high air velocity in their fluid bed dryer heat treatment so that the flour was elutriated and
242 captured on the bag filter (Neill and Magee, personal communication). Preliminary
243 experiments to the current study showed that it was possible to co-fluidise the flour with
244 sand, where the sand functioned as a thermal sink and was sized to enable rapid separation
245 by sieving, but this resulted in mechanical damage to the flour and poor baking performance.

246

247 A packed bed method was developed using 1 mm diameter glass ballotini as a thermal
248 regulator and structuring agent which allowed air to percolate through the mixture at the
249 required treatment temperature. Figure 1 illustrates the steps in the protocol. For a 300 g
250 flour test, 1000 g ballotini were preheated in an oven to the desired treatment temperature for
251 two hours. The flour charge was pre-dried in air at 80°C for 2 h in a separate oven. The
252 ballotini and flour were then combined and quickly transferred to the packed bed device. This
253 mixing of solids achieved rapid heating, reaching the target temperature in less than 20 s,

254 replicating the rise in the industrial device, while the packed bed configuration replicated
255 heated screws, which prolong the residence time at high temperature in the industrial
256 process.

257

258 The packed bed system was based on an Endecotts fluid bed dryer (Endecotts Ltd., London),
259 customized with an insulated 78 mm i.d. glass tube replacing the standard fluidization
260 chamber. The device was pre-heated by circulation of hot air for 20 min before addition of
261 the flour/ballotini charge. Hot dry air was passed through the bed at a superficial velocity of
262 0.01 ms^{-1} for the specified time. This velocity was lower than the ballotini fluidization velocity
263 and was selected to achieve percolation (removing volatiles, supplying oxygen and balancing
264 heat losses) with minimal elutriation. The temperature within and above the bed was
265 monitored during the treatment using K-type thermocouples. The maximum deviation from
266 the set temperature observed in these tests was 3 K, for a period of about 1 min. On
267 completion, the hot mixture was cooled (similarly quickly) by mixing with 1000 g of the same
268 ballotini initially at room temperature. The flour was then separated from the ballotini by
269 sieving for around 15 min with a 250 μm mesh. No discernible damage to the flour particles
270 was evident as a result of this protocol.

271

272

273 **Results and Discussion**

274 *Validation of lab-scale heat treatment protocol*

275 The efficacy of the lab-scale heat treatment method was assessed using flour from the 2010-
276 11 harvest, subjecting it to very different heat treatments:

277 (a) modest heating, 110°C for 15 min

278 (b) extended heating; at 130°C for 30 min

279 Test baking of the flours generated by these heat treatments gave volumes of (a)
280 $520.9 \pm 1.2 \text{ cm}^3$ and (b) $538.8 \pm 3.5 \text{ cm}^3$. The volume obtained for the untreated 2010-11 flour

281 was $526 \pm 2.4 \text{ cm}^3$, which is close to (a). The commercially heat-treated flour gave a volume
282 of $566 \pm 8.7 \text{ cm}^3$, which is greater than (b), as expected.

283

284 Table 3 summarises the quality parameters obtained from shape analysis of the test bake
285 cakes. The increase in volume and symmetry indices resulting from commercial heat
286 treatment is evident in the lab-scale data, while the uniformity indices within each set of
287 results is similar. Direct mapping of indices between the packed bed and commercial heat
288 treatment is not seen: the lab-scale method is expected to give an indication of the industrial
289 scale result. It is evident that improved baking performance, as observed with commercially
290 heat treated flour, can be achieved using the lab-scale heat treatment protocol.

291

292

293 *Effect of treatment time and temperature*

294 The effect of treatment time, t_{contact} , was investigated by holding the treatment temperature,
295 T_i , constant at 130°C and varying t_{contact} from 5 to 60 min (experiments 1-4, Table 4). The
296 effect of temperature was investigated by holding t_{contact} constant, at 15 min, and varying T_i
297 from 120 to 140°C (experiments 5-6, Table 4). The majority of the tests used the 2010-11
298 harvest flour and the remainder of the tests detailed in Table 4 results were verification trials
299 by repeating selected conditions with flours from two previous harvests (2009-10 and 2006-
300 07). The efficacy of heat treatment was assessed by test baking and rheological testing.

301

302 Figure 2 presents cross-section scans of the cakes baked from the lab-scale heat-treated
303 flours alongside those obtained for untreated and commercially heat-treated cakes. There is
304 noticeable asymmetry in the cake shape for all flours, which is due to uneven heat transfer in
305 the baking oven used in these tests. The heat flux across the shelf was measured in
306 separate tests and varied from the centre to the edges (Chesterton, 2011, data not reported).
307 This was a systematic feature common to all tests. The images in Figure 2 show that the
308 visual cake quality of the lab-scale heat-treated flour test bakes was generally intermediate

309 between the base flour and commercially heat-treated material. Colour reproductions of
310 Figure 2 show a difference in colour between the cakes baked with lab-treated flours and
311 those prepared from base and commercial heat-treated flour. This is an artefact arising from
312 differences in illumination conditions. Detailed studies of the materials would include precise
313 colour measurement as well as investigation of the texture of the baked cakes.

314

315 Figure 3 summarises the effect of treatment time and temperature on cake volume. Also
316 plotted on the figures are the values obtained for the untreated and commercially heat-
317 treated 2010-2011 flour. The conditions used for the latter are commercially sensitive. Figure
318 3(a) shows a significant effect of treatment time on baking performance at $T_f = 130^\circ\text{C}$: both
319 15 and 30 min of heat treatment improved the baking performance over the base flour. None
320 of the lab-scale tests gave cake volumes as large as the commercially heat-treated flour,
321 indicating that the test method is not able to reproduce the conditions in the plant perfectly.
322 The largest volume was obtained with $t_{\text{contact}} = 15$ min, and the value differed from the base
323 flour volume by a statistically significant amount. The existence of an optimal value of t_{contact}
324 around 15 min was observed for all three harvests at 130°C . Similar results have been
325 reported in other tests by the sponsor (Premier Foods, private communication). Neill *et al.*
326 (2012) reported optimal treatment conditions of $120\text{-}130^\circ\text{C}$ for 30 min, which represents a
327 longer period of heat treatment than this work.

328

329 Figure 3(b) indicates the existence of an optimal treatment temperature for $t_{\text{contact}} = 15$ min.
330 Treatment at 120°C gave a lower cake volume than 130°C , while increasing T_f to 140°C
331 showed a significant reduction of volume for the 2009-10 harvest flour. The reduction
332 observed for the 2010-11 harvest flour was not significantly different, highlighting how annual
333 variations in wheat growing conditions alter the performance of the flours. Both plots indicate
334 that under-treatment, by reducing either time or temperature (assuming 15 min at 130°C is
335 optimal), is more detrimental to the volume of cakes than over-treatment.

336

337 The data in Figure 3 are now compared in terms of equivalent treatment time at 130°C,
338 labeled t_{130C} . The use of an equivalent treatment time is frequently used in food processing
339 applications to compare processes with different time-temperature histories, particularly in
340 evaluating microbial deactivation (see Pyle *et al.*, 1997; Singh and Heldman, 2009). An
341 equivalent time is calculated by assuming a doubling of reaction rate for a 10 K increase in
342 temperature (i.e. to 140°C) and the rate halving with a 10 K decrease to 120°C. This
343 assumes that the heat treatment process is chemical reaction controlled. The data from
344 Figure 3 are replotted in Figure 4 presents with the abscissa as t_{130C} . The sensitivity of the
345 result to the assumption that the rate doubles every 10K is indicated by the error bars in t_{130C} ,
346 showing the value of t_{130C} calculated with (i) $k_{140}/k_{130} = 2.5$ and (ii) $k_{140}/k_{130} = 1.5$. This
347 presentation format confirms the existence of an asymmetric optimum, with cake volume
348 increasing noticeably with t_{130C} until 15-20 min and decreasing slowly thereafter. The optimal
349 time was consistently around 15 min for the harvests tested here, but the cake volumes
350 differed between harvests. This consistency in treatment time is not entirely unexpected as
351 the flours used were commercial flours gristed to suit a given process as closely as the
352 available wheat supply could provide at the time.

353

354 Figure 4 indicates that several experiments produced cake volumes that were lower than the
355 average volume for the 2010-2011 flour. The range of volumes for this material was
356 $\pm 8.9 \text{ cm}^3$, leaving only one experiment (experiment 9, 2006-2007 harvest) with a volume
357 statistically lower than the base flour. This result is likely to be due to differences between
358 harvests, although there is some uncertainty associated with the effect of storing the flour
359 frozen until 2011, when the tests were performed.

360

361 The quality indices for experiments 1-10 in Table 4 are plotted against equivalent treatment
362 time in Figure 5. The volume index values in Figure 5(a) show an initial increase with t_{130C}

363 followed by a decrease, with a peak between 15 and 30 min, mirroring the trend in Figure 4.
364 The variation of volume index with t_{130C} was not as pronounced as the cake volume: it is not
365 as accurate as it is based on only 3 measurements for each sample. Comparing the volume
366 index with the untreated case showed that all lab-scale heat-treatment tests improved baking
367 performance, and in most cases the volume index was comparable to the commercially heat-
368 treated value.

369

370 The symmetry index measures the cake peakedness, *i.e.* the relative height of the cake
371 centre to the cake shoulders. The symmetry index data in Figure 5(b) show a gradually
372 increasing trend with t_{130C} , *i.e.* the cakes become more peaked, possibly reaching a plateau
373 at $t_{130C} = 30$ min. A low symmetry index indicates a flat cake, which is undesirable, but too
374 high a value is also undesirable. Flours with $t_{130C} < 20$ min gave values similar to the
375 commercially heat-treated flour, while the values for $t_{130C} > 20$ min indicate over-peaked
376 cakes.

377

378 The uniformity index indicates the difference between shoulder heights and is a measure of
379 the centrality of the cake peak. Lower values (ideally zero) are preferred. The base and
380 commercially heat-treated uniformity index values were significantly different from zero,
381 indicating that the cake peaks were off-central (see Figure 2). This was the result of the oven
382 used for these experiments, reported above. Interestingly, short lab-scale heat-treatment
383 (<20 min) improved the uniformity of the cakes (Figure 5(c): also Figure 2, cakes (1)-(4)). The
384 reason is not yet known. Longer lab-scale heat-treatment (>20 min) gave similar or larger
385 uniformity indices to the base and commercially heat-treated flours, indicating lopsided
386 cakes.

387

388 The treatment condition that produced the largest volume cake in Figure 5 was $t_{\text{contact}} =$
389 15 min and $T_f = 130^\circ\text{C}$, and gave volumes comparable to commercially heat-treated cakes
390 (Figure 4). The commercially heat-treated average volume was not exceeded, which is

391 attributed to the additional pin-milling stage used in the commercial process. Previous lab-
392 scale heat-treatment experiments have found an additional pin-milling step necessary to
393 improve flour to the level achieved in the commercial process (Premier Foods, personal
394 communication). Cauvain and Muir (1974) investigated the effect of particle size on baking
395 quality and found that milling did not change the poor quality of untreated flours, but resulted
396 in a substantial improvement in baking quality of heat-treated flours. The lack of pin-milling in
397 lab-scale heat-treatment studies has been proposed as a reason why lab-produced flours
398 were not comparable to commercially heat-treated flours.

399

400 *Oscillatory shear – the weak gel model*

401 Measurements of the elastic and viscous moduli, G' and G'' , respectively, allow the complex
402 modulus, G^* , to be calculated. In the weak gel model (Gabriele *et al.*, 2001) this is related to
403 the test frequency ω by:

$$404 \quad |G^*| = \sqrt{(G')^2 + (G'')^2} = A_F \omega^{1/z} \quad [1]$$

405 where z is the interaction factor and A_F is the gel strength. The former can be interpreted as
406 the number of flow units interacting with one another in a three-dimensional structure to give
407 the observed deformation response, while A_F can be interpreted as the strength of the
408 interaction between flow units. For all the materials tested the z parameter showed little
409 variation with time and temperature variations, and no correlation with treatment time,
410 temperature, or cake quality after test baking.

411

412 Figure 6(a) shows the effect of treatment time, for $T_f = 130^\circ\text{C}$. A_F values were consistently
413 higher than the untreated flour value, indicating a stronger gel network. The A_F values were
414 at least as high as the commercially heat-treated values at 100°C , and when treated for
415 15 min and 30 min at 130°C the flours gave A_F values higher than the commercially heat-
416 treated one (at all temperatures: 80, 90 and 100°C). The A_F data at 100°C followed the trend
417 observed in cake volume: increasing from 5 to 15 min of treatment at $T_f = 130^\circ\text{C}$, then

418 decreasing with extended treatment time. The 2006-07 harvest flour was treated for 5 min at
419 130°C and gave a comparable result to the 2010-11 harvest.

420

421 Figure 6(b) shows the effect of temperature for t_{contact} at 15 min. At 90°C and 100°C the lab-
422 scale heat-treated flours gave A_F values higher than the base flour. Treatment for 15 min at
423 $T_f = 130^\circ\text{C}$ gave the highest A_F at all temperatures (80, 90, 100°C) and also had the largest
424 cake volume. Only treatment at 130°C gave an A_F value higher than for commercially heat-
425 treated flour, with the treatments at 120°C and 140°C being comparable to it.

426

427 Figure 6(c) compares heat treatment ($T_f = 130^\circ\text{C}$, $t_{\text{contact}} = 15$ min) for different harvests. At
428 each temperature (80-100°C) the A_F values for the lab-treated flours were higher than the
429 base and commercially heat-treated values, but within the uncertainty of the commercially
430 heat-treated data. There is some variation in A_F values between harvests, which in all cases
431 lies within experimental error.

432

433 The A_F values obtained at 100°C are plotted against $t_{130\text{C}}$ in Figure 7 and show a similar trend
434 to that between $t_{130\text{C}}$ and cake volume in Figure 4. The largest A_F values are found at $t_{130\text{C}}$
435 ~15 min, as with the cake volume. Since both A_F and cake volume correlate with flour
436 quality, measurement of A_F provides a potential proxy for successful heat-treatment. The
437 correlation between A_F and cake volume is shown in Figure 8. The plot shows a positive
438 correlation, but is not strong ($R^2 = 0.3$) due to the inherent variability in both the A_F and cake
439 baking methods. However, since there is a large variability in the cake baking method, this
440 result indicates that A_F can provide an alternative measure, as the problems of accuracy and
441 reproducibility in cake quality determination are eliminated. The rheological tests require
442 relatively small samples, effectively the amount needed to prepare a reproducible volume of
443 batter, and provide an avenue for identifying the region of optimal conditions to be confirmed
444 later on by cake baking.

445

446 The results from steady shear rheology tests performed at 20°C did not show a consistent
447 correlation with baking results, confirming that heat treatment was affecting the behaviour of
448 the batters in the starch gelatinization/protein denaturation stages of baking. The apparent
449 viscosity-shear rate plots exhibited shear-thinning behaviour, as reported for similar materials
450 by Meza *et al.* (2011). Batters mixed for 2, 6 and 10 minutes prepared with flours heat-
451 treated for 15 min or longer at 130°C gave identical viscosity-shear rate plots, as reported for
452 commercial heat-treated flours by Meza *et al.* (2011), whereas these plots differed for batters
453 prepared with base flour or flour heat-treated for 5 min at 130°C. These qualitative
454 observations support the findings of the oscillatory tests at higher temperature, and are not
455 reported in detail here for brevity.

456

457 The objective of this work was to develop a heat treatment test. The mechanisms
458 responsible for the changes in flour performance have not been investigated, partly as this
459 would require quantification of protein and starch content and functionality, texture, colour,
460 protein extraction, and crumb deformation. We believe that the results of such studies can
461 now be linked to the process with greater confidence as this method allows the flour to
462 experience the thermal and environmental conditions more closely.

463

464

465 **Conclusions**

466 A method of replicating industrial heat-treatment on a laboratory scale is presented which
467 was subsequently used to determine the effect of treatment time and temperature on the
468 quality of flour produced. The latter was determined by test baking and quantified using
469 measures of cake volume and shape. The former was found to correlate with the A_F
470 parameter of the weak gel model (Meza *et al.*, 2011; Gabriele *et al.*, 2001), suggesting that

471 measurement of this parameter could provide a proxy for determining flour quality after heat
472 treatment.

473

474 The study showed that a packed bed in which flour was mixed with glass ballotini and air was
475 passed upward through the bed, mimicked the industrial heat-treatment process effectively.
476 Preheating the ballotini allowed a rapid temperature change to be imposed. A secondary
477 effect of the ballotini was that they broke up the cohesive mass of flour, therefore aiding air
478 flow through the bed. An evenly distributed air flow is important for replicating the industrial
479 process.

480

481 A series of treatment conditions were used to determine the optimal time and temperature for
482 heat-treatment. Test baking showed that the optimal heat-treatment condition was around
483 130°C for 15 min, as the resultant cake gave the largest volume and best quality,
484 approaching commercial heat treatment results despite the absence of a milling step.

485

486 The gel strength analysis, based on oscillatory rheometry testing, advocated by Meza *et al.*
487 (2011) was used to assess small volumes of cake batters. Data from several harvests
488 confirmed that the gel strength parameter A_F correlated with heat-treatment in a similar way
489 to cake volume. The weak gel model allows ready quantification of the gel strength for
490 comparison with other samples or a reference. Determination of the weak gel model A_F
491 parameter is proposed as an alternative to test baking (which is time consuming and subject
492 to inherent variability and subjective assessment) for optimising heat treatment, or at least for
493 identifying the optimal region for cake baking testing.

494

495

496 **Acknowledgments**

497 A CASE Ph.D. Studentship for AKSC from the Food Processing Faraday and support from
498 Premier Foods are all gratefully acknowledged.

499 **Nomenclature**

500	A_F	gel strength, weak gel model (Gabriele <i>et al.</i> , 2001)	-
501	G'	elastic modulus	Pa
502	G''	viscous modulus	Pa
503	$ G^* $	magnitude of the complex modulus	Pa
504	k_{T_1}	rate of reaction at temperature T_1	s^{-1}
505	t_{130C}	effective treatment time at 130°C	min
506	$t_{contact}$	time of heat-treatment	min
507	T_f	temperature of flour during heat-treatment	°C
508	z	number of gel units, weak gel model (Gabriele <i>et al.</i> , 2001)	-
509			
510	$ \eta^* $	complex viscosity	Pa s
511			
512			

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569 **Tables**

570 *List of table captions*

571 Table 1 Flours tested in this investigation. Significant figures indicate measurement
572 accuracy.

573 Table 2 Batter formulations used for 2006-07 harvest (in wt % and in baker%*
574 Quantities reported to one decimal place: experimental variation lay within
575 this level of precision.

576 Table 3 Comparison of cake volume and quality indices obtained for lab-scale heat
577 treatment flours with untreated and commercially heat treated flours.
578 Indices based on AACC method 10-91 (AACC, 1999). Standard deviations
579 based on six replicates. 2010-11 harvest flour.

580 Table 4 Experimental conditions used to test the effect of time and temperature on
581 the quality of heat-treated flour.

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586

587 Table 1 Flours tested in this investigation. Significant figures indicate measurement
 588 accuracy.

Harvest	Flour	Water (wt%*)	Ash (wt%)	Protein (wt%)	Protein (dry basis) (wt% d.b.)
2010-2011	base	12.60	0.69	9.13	9.73
	heat-treated	6.99	0.77	8.57	9.21
2009-2010	base	12.0	0.68	9.29	10.56
	heat-treated	6.10	0.76	8.81	9.38
2006-2007	base	12.58	0.69	7.98	9.13
	heat-treated	6.99	0.77	8.57	9.21

589

590 * Mass fractions are wet basis unless otherwise stated

591

592

593

594 Table 2 Batter formulations used for 2006-07 harvest (in wt % and in baker%*).

595 Quantities reported to one decimal place: experimental variation lay within

596 this level of precision.

Ingredient	Base flour		Heat-treated flour	
	wt%	<i>baker%</i>	wt%	<i>baker%</i>
Caster sugar	35.8	133	35.8	142
Flour (2006-2007)	26.9	100	25.2	100
Water (tap)	14.4	54	16.1	64
Whole liquid eggs	13.8	51	13.8	55
Skimmed milk powder	3.9	14	3.9	15
Margarine	2.8	10	2.8	11
Baking powder	1.0	4	1.0	4
Emulsifier	0.8	3	0.8	3
Salt	0.6	2	0.6	2

597

598 * baker % is ratio to flour content

599

600 Table 3 Comparison of cake volume and quality indices obtained for lab-scale heat
 601 treatment flours with untreated and commercially heat treated flours.
 602 Indices based on AACC method 10-91 (AACC, 1999). Standard deviations
 603 based on six replicates. 2010-11 harvest flour.
 604

Treatment	Cake volume (cm ³)	Volume index (mm)	Symmetry index (mm)	Uniformity* index (mm)
Base, no heat treatment	526 ± 2.4 ^c	94.8 ± 5.5 ^b	11.3 ± 4.4 ^b	8.1 ± 1.5 ^a
Commercially heat-treated	566 ± 8.7 ^a	110.8 ± 5.5 ^a	15.6 ± 4.4 ^a	8.1 ± 1.5 ^a
Packed bed				
(a) modest (110°C for 15 min)	520.9 ± 1.2 ^d	101.1 ± 5.5 ^{a,b}	12.6 ± 4.4 ^{a,b}	0.0 ± 1.5 ^b
(b) extended (130°C for 30 min)	538.8 ± 3.5 ^b	112.6 ± 5.5 ^a	18.4 ± 4.4 ^a	1.6 ± 1.5 ^b

605 * lower values more desirable.

606 Letters ^{a, b, c, d} denote outcome of ANOVA testing. Letters indicate samples belonging to
 607 same population, at the $p = 0.05$ significance level. Letters in order largest-smallest.

608

609

610

611 Table 4 Experimental conditions used to test the effect of time and temperature on
612 the quality of heat-treated flour.

613

Expt	$t_{contact}$ (min)	T_f (°C)	Harvest
1	5	130	10-11
2	15	130	10-11
3	30	130	10-11
4	60	130	10-11
5	15	120	10-11
6	15	140	10-11
7	15	130	06-07
8	15	130	09-10
9	5	130	06-07
10	15	140	09-10

614

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616 **List of Figure captions**

617

618 Figure 1 Schematic of heat treatment protocol

619 Figure 2 Cross sections of one cake sample from each test bake set of untreated (base),
620 commercially heat-treated, and packed-bed heat treatment of flours detailed in
621 Table 4. Numbers in parentheses are the experiment number in Table 4.

622 Figure 3 Effect of (a) treatment time ($T_f = 130^\circ\text{C}$) and (b) temperature ($t_{\text{contact}} = 15$ min)
623 on cake volume. Error bars indicate the range within replicates ($n = 4$). Dashed
624 horizontal lines show results obtained for untreated (base) flour and commercially
625 heat treated flour.

626 Figure 4 Effect of equivalent treatment time on cake volume for different harvests. Error
627 bars in $t_{130\text{C}}$ values indicate the range of $t_{130\text{C}}$ values calculated using k_{140}/k_{130}
628 $= 1.5$ and $k_{140}/k_{130} = 2.5$.

629 Figure 5 Effect of equivalent contact time on cake quality indices based on AACC
630 approved method 10-91 (AACC, 1999). (a) volume index, (b) symmetry index, (c)
631 uniformity index. Horizontal loci show values obtained for untreated (dashed) and
632 commercially heat-treated (dot-dashed) 2010-2011 flour reported in Table 3.

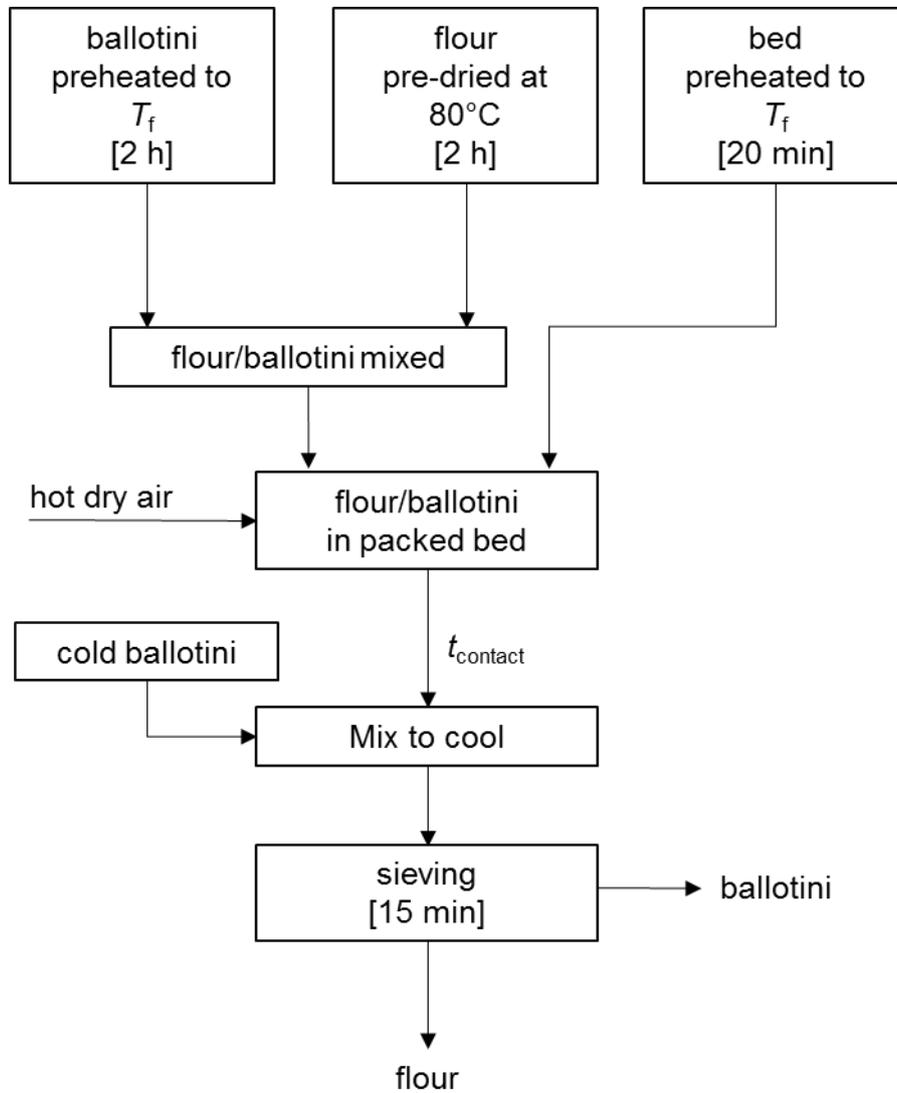
633 Figure 6 Gel strength for cake batters measured at 80, 90 and 100°C : (a) effect of t_{contact}
634 for $T_f = 130^\circ\text{C}$, (b) effect of T_f for $t_{\text{contact}} = 15$ min, (c) effect of flour harvest for
635 $t_{\text{contact}} = 15$ min, $T_f = 130^\circ\text{C}$. Flours are from the 2010-11 harvest unless otherwise
636 indicated.

637 Figure 7 Effect of time and temperature, expressed as $t_{130\text{C}}$, on gel strength, A_F ,
638 measured at 100°C . Horizontal loci indicate the values obtained for commercially
639 heat-treated (dashed) and base flour (dotted) for the 2010-11 harvest. Error bars
640 on x-axis show range of $t_{130\text{C}}$ values calculated using $k_{140}/k_{130} = 1.5$ and
641 $k_{140}/k_{130} = 2.5$.

642 Figure 8 Correlation of cake volume with gel strength measured at 100°C. Dashed grey
643 line shows line of best fit.

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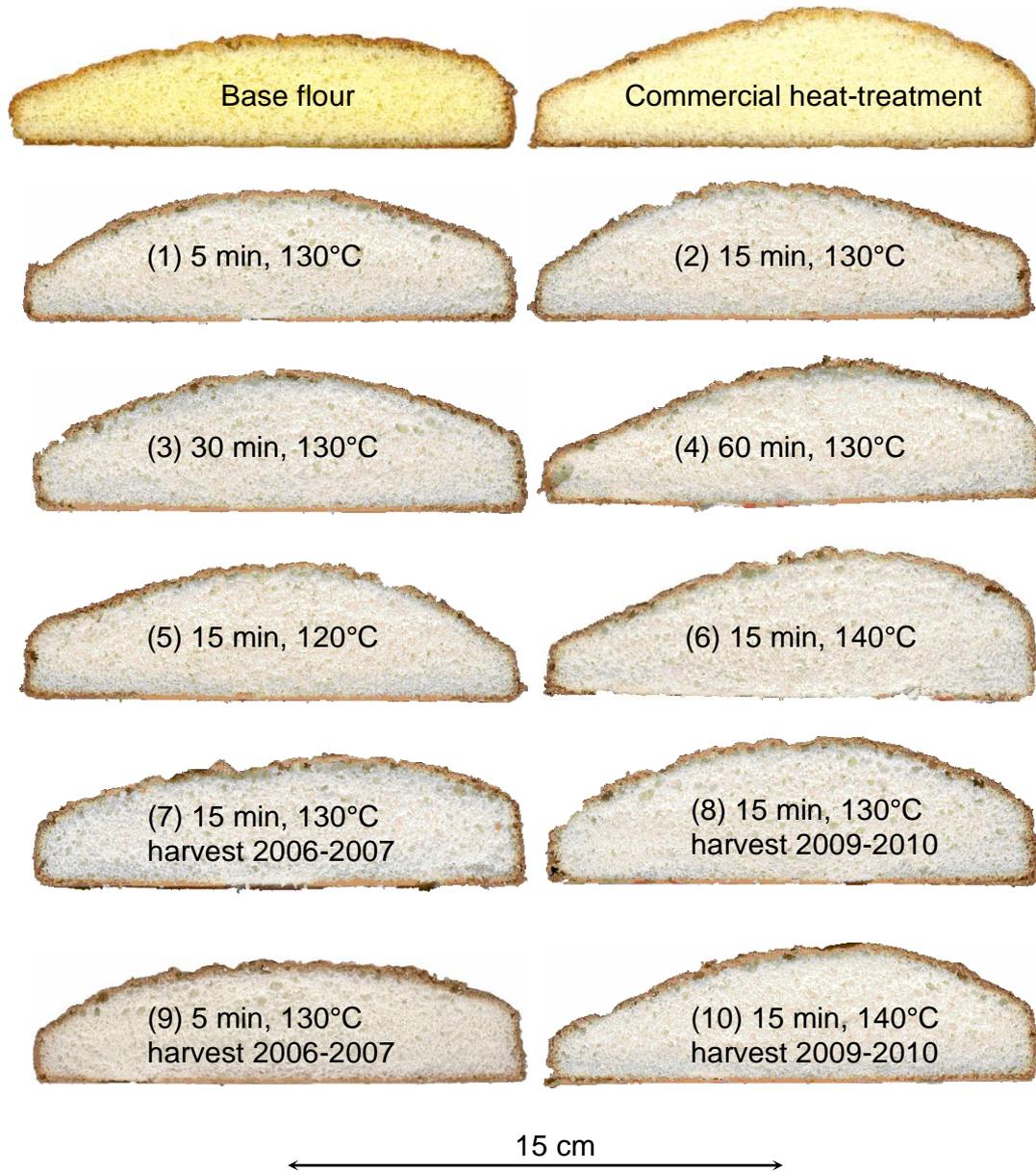


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656 Figure 1 Schematic of heat treatment protocol

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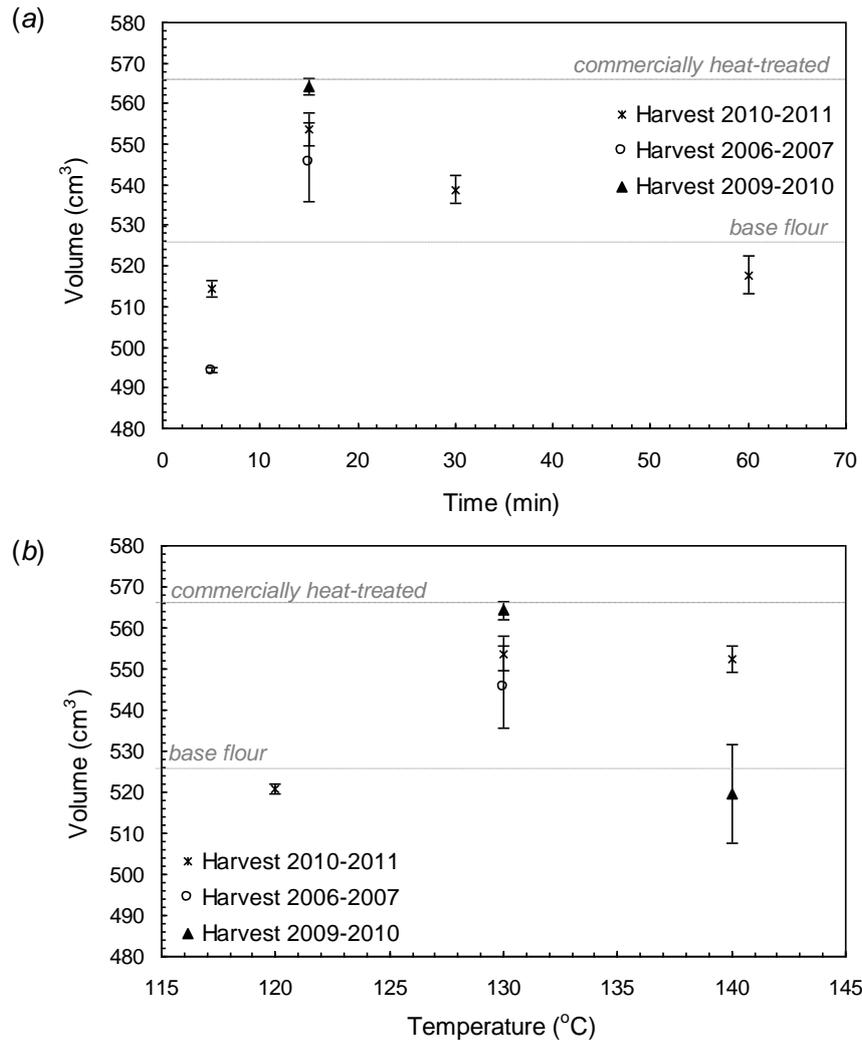
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662 (base), commercially heat-treated, and packed-bed heat treatment of flours
663 detailed in Table 4. Numbers in parentheses are the experiment number in
664 Table 4.

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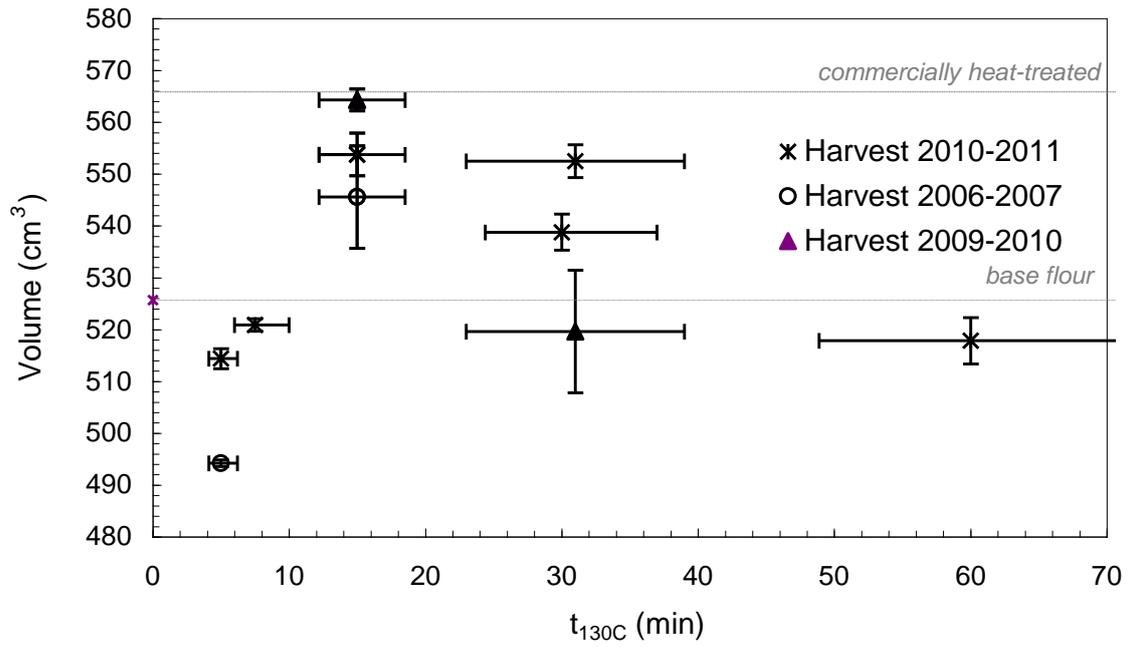
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671 4). Dashed horizontal lines show results obtained for untreated (base) flour

672 and commercially heat treated flour.

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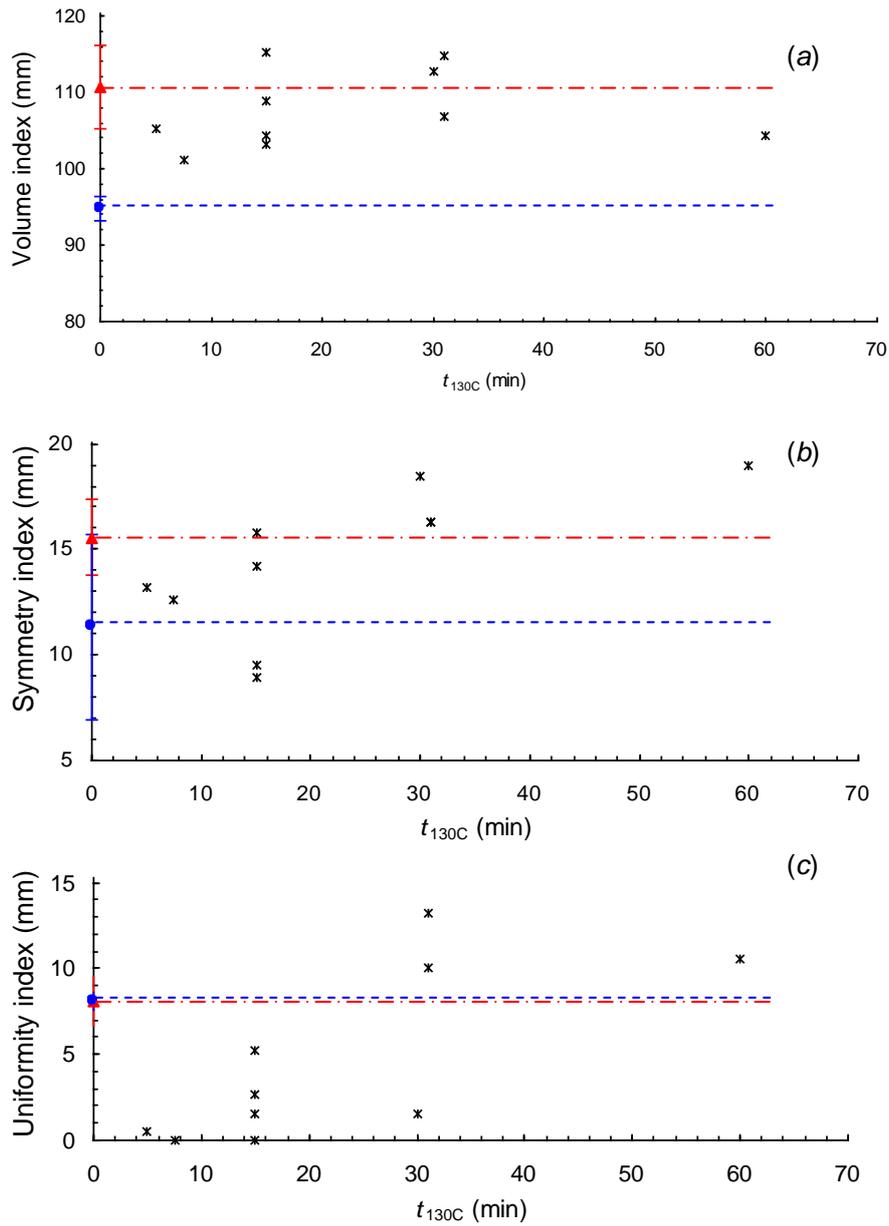
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676 Figure 4 Effect of equivalent treatment time on cake volume for different harvests.

677 Error bars in t_{130C} values indicate the range of t_{130C} values calculated using

678 $k_{140}/k_{130} = 1.5$ and $k_{140}/k_{130} = 2.5$.

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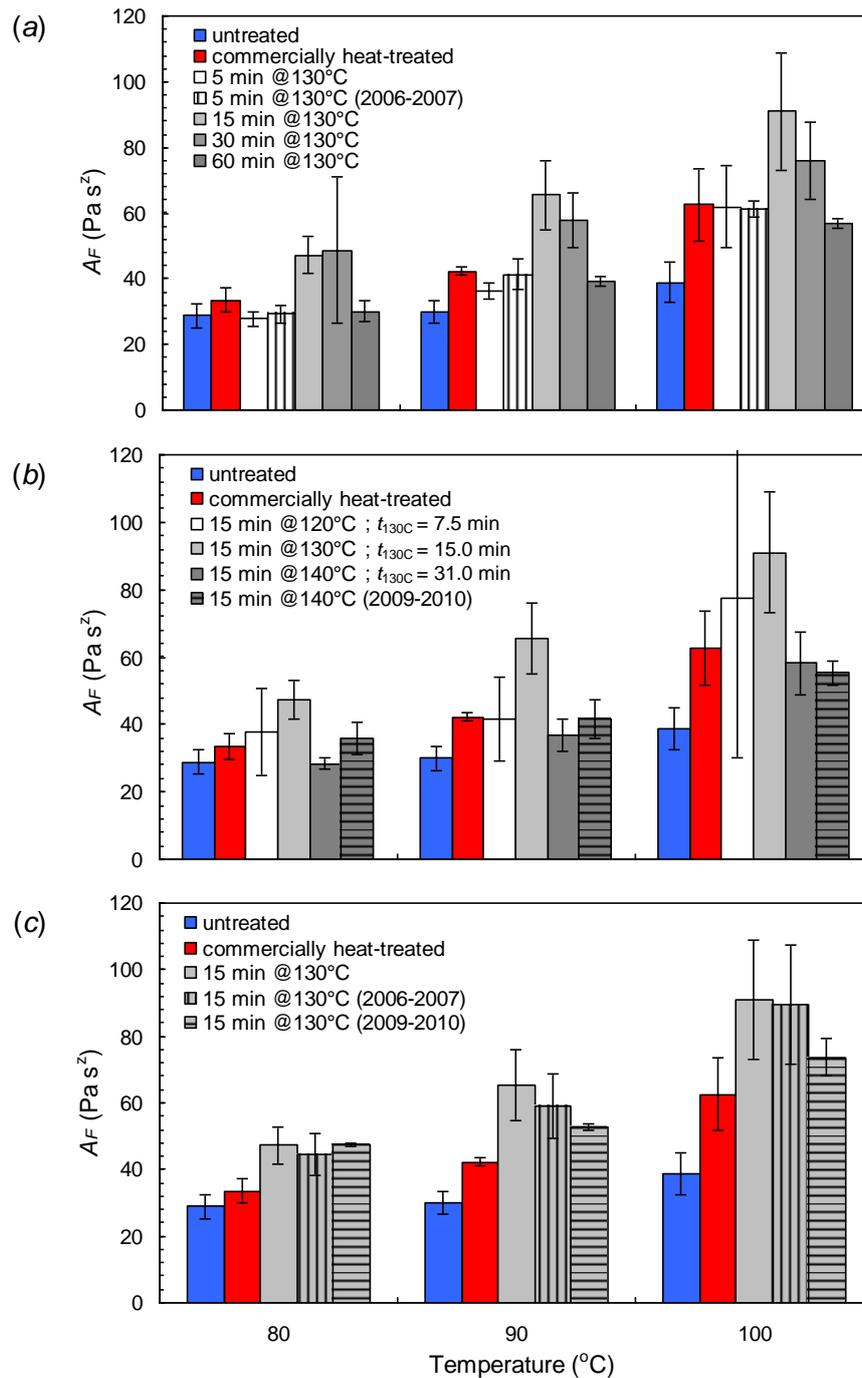


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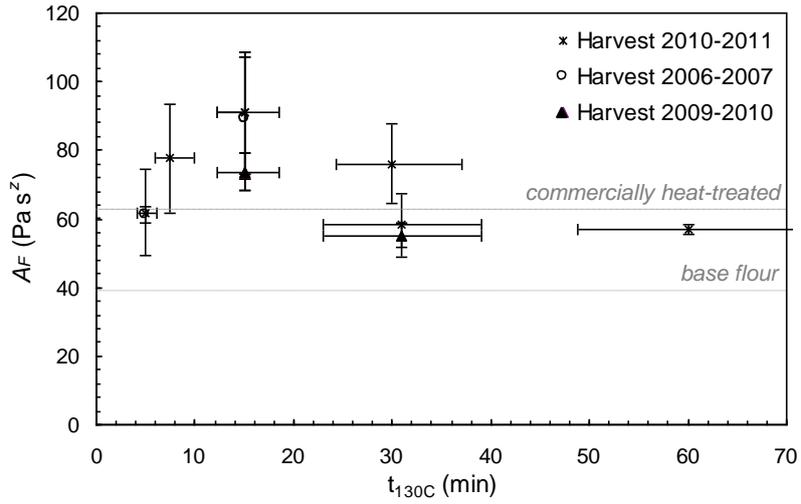
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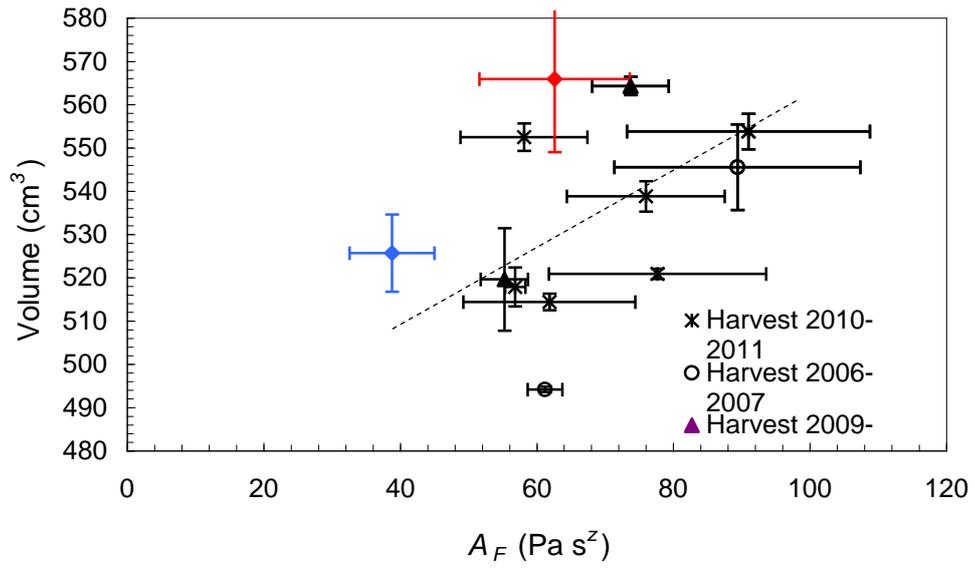
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701 $k_{140}/k_{130} = 1.5$ and $k_{140}/k_{130} = 2.5$.

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706 Figure 8 Correlation of cake volume with gel strength measured at 100°C. Dashed

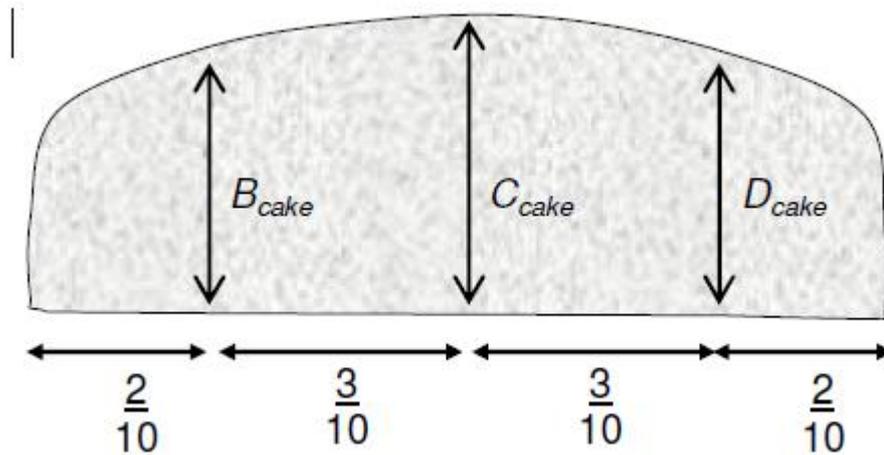
707 grey line shows line of best fit.

708

709

710 Appendix. AACC cake shape parameters

711 Figure A1 Cross section through cake showing measurements.



712

713 The height of the cake is measured at the three positions on a diameter shown above. The

714 indices are calculated from

715
$$\text{Volume index} = B_{cake} + C_{cake} + D_{cake} \quad \text{A1}$$

716
$$\text{Symmetry index} = 2C_{cake} - B_{cake} - D_{cake} \quad \text{A2}$$

717
$$\text{Uniformity index} = B_{cake} - D_{cake} \quad \text{A3}$$

718 The volume index gives an indication of the overall size of the cake. The symmetry index

719 assesses how peaked the cake is, while the uniformity index reflects how central the cake

720 peak is.