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

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Abstract

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

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

Introduction

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

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

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

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

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

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

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

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

The optimal time and temperature for heat treatment in stage 2 can vary with harvest year as a result of annual variation in both wheat supply and properties. Hence the optimal conditions and grist have to be established each year, requiring a campaign of testing. Currently the only method of assessing the quality of heat-treated flour is to test bake, using a set laboratory recipe incorporating high levels of sugar and liquid, designed to test the robustness of the flour. Such tests are time consuming, require specialist operators, and are subject to inherent variability. Furthermore, assessment of the ‘quality’ of a cake is non-trivial. Parameters such as volume and height are recorded quantitatively, but aspects such as shape, evenness and texture are assessed qualitatively by a trained operator. Neill *et al*. (2012) studied heat treatment of a flour using a fluidized bed to deliver between 5 and 60 minutes of heat treatment at 120°C and 130°C. They assessed the effect of heat treatment by Brabender viscosity measurements, gluten extensibility, starch gelatinisation and test baking of Madeira cake. Quantifiable improvement in cake quality was observed and they reported an optimal heat treatment as 30 min at 130°C. They did not report results for different harvests. Thomasson *et al*. (1997) heat treated flour by placing a layer of flour on a tray in an oven and reported an optimal treatment as 30 min at 125°C. Different harvests were again not considered.

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

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

We then address two important issues in heat treatment:

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

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

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

Materials and Methods

Flours

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

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

Ingredients

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

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

Baking method

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

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

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

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

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

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

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

Cake properties

Cross-sectional images

Cakes were bisected and scanned using a HP Scanjet 3570c device. Cakes were placed face-down on the scanner and covered with black cloth to increase the contrast between the image and background. Cake images were then removed from the background using Photoshop CS5 software for presentation.

Volume measurement

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

Rheometry

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

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

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

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

Lab-scale heat treatment protocol

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

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

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

Results and Discussion

Validation of lab-scale heat treatment protocol

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

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

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

Test baking of the flours generated by these heat treatments gave volumes of (*a*) 520.9 $\pm$ 1.2 cm^3 and (*b*) 538.8 $\pm$ 3.5 cm^3. The volume obtained for the untreated 2010-11 flour was 526 ± 2.4 cm^3, which is close to (*a*). The commercially heat-treated flour gave a volume of 566 ± 8.7 cm^3, which is greater than (*b*), as expected.

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

Effect of treatment time and temperature

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

Figure 2 presents cross-section scans of the cakes baked from the lab-scale heat-treated flours alongside those obtained for untreated and commercially heat-treated cakes. There is noticeable asymmetry in the cake shape for all flours, which is due to uneven heat transfer in the baking oven used in these tests. The heat flux across the shelf was measured in separate tests and varied from the centre to the edges (Chesterton, 2011, data not reported). This was a systematic feature common to all tests. The images in Figure 2 show that the visual cake quality of the lab-scale heat-treated flour test bakes was generally intermediate between the base flour and commercially heat-treated material. Colour reproductions of Figure 2 show a difference in colour between the cakes baked with lab-treated flours and those prepared from base and commercial heat-treated flour. This is an artefact arising from differences in illumination conditions. Detailed studies of the materials would include precise colour measurement as well as investigation of the texture of the baked cakes.

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

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

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

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

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

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

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

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

The treatment condition that produced the largest volume cake in Figure 5 was $t_{contact}$ = 15 min and T_f = 130°C, and gave volumes comparable to commercially heat-treated cakes (Figure 4). The commercially heat-treated average volume was not exceeded, which is attributed to the additional pin-milling stage used in the commercial process. Previous lab-scale heat-treatment experiments have found an additional pin-milling step necessary to improve flour to the level achieved in the commercial process (Premier Foods, personal communication). Cauvain and Muir (1974) investigated the effect of particle size on baking quality and found that milling did not change the poor quality of untreated flours, but resulted in a substantial improvement in baking quality of heat-treated flours. The lack of pin-milling in lab-scale heat-treatment studies has been proposed as a reason why lab-produced flours were not comparable to commercially heat-treated flours.

Oscillatory shear – the weak gel model

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

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

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

Figure 6(*a*) shows the effect of treatment time, for T_f = 130°C. A_F values were consistently higher than the untreated flour value, indicating a stronger gel network. The A_F values were at least as high as the commercially heat-treated values at 100°C, and when treated for 15 min and 30 min at 130°C the flours gave A_F values higher than the commercially heat-treated one (at all temperatures: 80, 90 and 100°C). The A_F data at 100°C followed the trend observed in cake volume: increasing from 5 to 15 min of treatment at T_f = 130°C, then decreasing with extended treatment time. The 2006-07 harvest flour was treated for 5 min at 130°C and gave a comparable result to the 2010-11 harvest.

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

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

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

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

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

Conclusions

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

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

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

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

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Nomenclature

A_F	gel strength, weak gel model (Gabriele *et al*., 2001)	-
G'	elastic modulus	Pa
G''	viscous modulus	Pa
$\lvert G^* \rvert$	magnitude of the complex modulus	Pa
k_{T_1}	rate of reaction at temperature T_1	s^{-1}
t_{130C}	effective treatment time at 130°C	min
$t_{contact}$	time of heat-treatment	min
T_f	temperature of flour during heat-treatment	°C
z	number of gel units, weak gel model (Gabriele *et al*., 2001)	-
$\lvert \eta^* \rvert$	complex viscosity	Pa s

References

AACC (1999) AACC method 10-91: use of layer cake measuring template. *American Association of Cereal Chemists Inc.*

Brekken, R. A., Lancaster, E. B. & Wheelock, T. D. (1970) Fluidisation of flour in a stirred, aerated bed, Part I: general fluidisation characteristics. *Chemical Engineering Progress Symposium Series,* 66, 81-90.

Cauvain, S. P. & Muir, D. D. (1974) Report 61: High-ratio yellow cakes: the effect of flour particle size. Flour Milling and Baking Research Association.

Chesterton, A.K.S. (2011) Ph.D. Dissertation, University of Cambridge.

Chesterton, A.K.S, Pereira de Abreu, D.A., Moggridge, G.D., Sadd, P.A. and Wilson, D.I. (2013) Evolution of cake batter bubble structure and rheology during planetary mixing, *Food & Bioproducts Processing,* 91, 192-206.

Coulter (1994) *LS Series Product manual,* Miami, Florida, Coulter Corporation.

Doe, C. A. & Russo, J. V. (1968) Flour treatment process. British Patent GB1110711.

Gabriele, D., de Cindio, B. & D'Antona, P. (2001) A weak gel model for foods. *Rheologica Acta,* 40**,** 120-127.

Gomez, M., Oliete, B., Garcia-Alvarez, J., Ronda, F. & Salazar, J. (2008) Characterization of cake batters by ultrasound measurements. *J. Food Engineering,* 89**,** 408-413.

Guerrieri, N., Alberti, E., Lavelli, V. & Cerletti, P. (1996) Use of spectroscopic and fluorescence techniques to assess heat-induced molecular modifications of gluten. *Cereal Chemistry,* 73**,** 368-374.

Guerrieri, N. & Cerletti, P., 1996. Effect of high-temperature short-time treatment of wheat flour on gluten vitality and structure. *Cereal Chemistry* 73, 375–378

Indrani, D. & Rao, V.G., 2008. Functions of ingredients in the baking of sweet goods. In: Sumnu, S.G., Sahin, S. (Eds.), Food Engineering Aspects of Baking Sweet Goods. CSR Press, Boca Raton, Florida, USA

Mangels, C. E. (1934) Some effects of heat exposure on wheat starches. *Cereal Chemistry,* 11**,** 86.

McGee, H. (2004) *On food and cooking: the science and lore of the kitchen*.

Meza, B. E., Chesterton, A. K. S., Verdini, R. A., Rubiolo, A. C., Sadd, P. A., Moggridge, G. D. & Wilson, D. I. (2011) Rheological characterisation of cake batters generated by planetary mixing: Comparison between untreated and heat-treated wheat flours. *Journal of Food Engineering,* 104**,** 592-602.

Niell, G, Al-Muhtaseb, A.H. and Magee, T.R.A. (2012) Optimisation of time/temperature treatment for heat treated soft white flour, *J. Food Engineering*, 113, 422-426.

Nicholas, E. M., Boynton, G. E., Russo, J. V. & Totty, R. (1974) Flour treatment process. British Patent GB1499986.

Ozawa, M., Kato, Y. & Seguchi, M. (2009) Investigation of dry-heated hard and soft wheat flour. *Starch-Starke,* 61**,** 398-406.

Ozawa, M. & Seguchi, M. (2006) Relationship between pancake springiness and interaction of wheat flour components caused by dry heating. *Food Science and Technology Research,* 12**,** 167-172.

Pyle, D.L., Fryer, P.J., and Rielly, C.D. (1997) *Chemical Engineering for the Food Industry*, publ. Springer.

Sahin, S. (2008) Cake batter rheology. In Sumnu, G. U. & Sahin, S. (Eds.) *Food engineering aspects of baking sweet goods*, publ.Taylor & Francis, Boca Raton.

Sevenou, O., Hill, S. E., Farhat, I. A. & Mitchell, J. R. (2002) Organisation of the external region of the starch granule as determined by infrared spectroscopy. *International Journal of Biological Macromolecules,* 31**,** 79-85.

Singh, R.P. and Heldman, D.R. (2009) *Introduction to Food Process Engineering*, publ. Elsevier.

Thomasson, C.A., Miller, R.A. and Hoseney, R.C. (1995) Replacement of chlorine treatment for cake flour, *Cereal Chemistry*, 72, 616-620.

www.talkingretail.com/products/product-news/premier-foods-bakes-new-growth-for-ambient-cakes (2009) Premier Foods bakes new growth for ambient cakes. Metropolis International Group Ltd.

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

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

* Mass fractions are wet basis unless otherwise stated

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

Ingredient	Base flour		Heat-treated flour	
	wt%	*baker%*	wt%	*baker%*
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*

* baker % is ratio to flour content

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

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}

* lower values more desirable.

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

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

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

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Figure 1 Schematic of heat treatment protocol

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

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

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

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

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

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

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

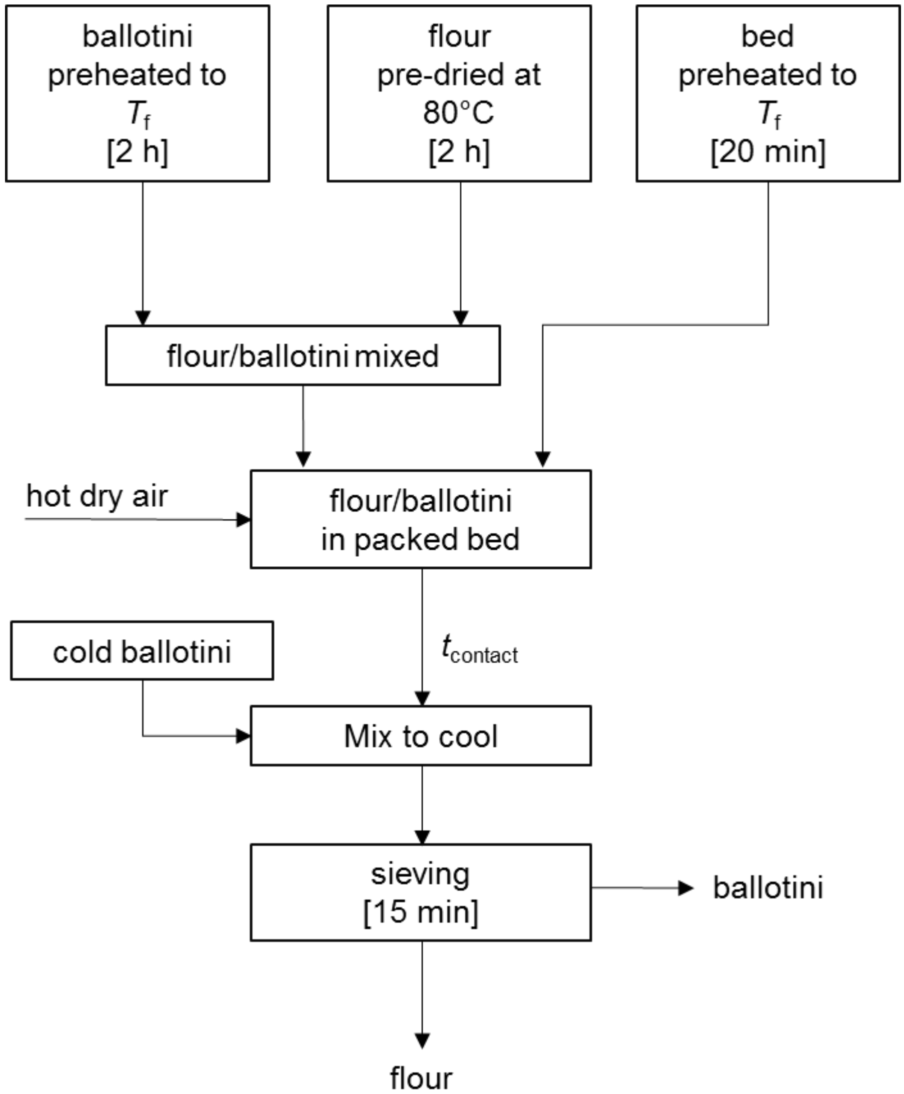
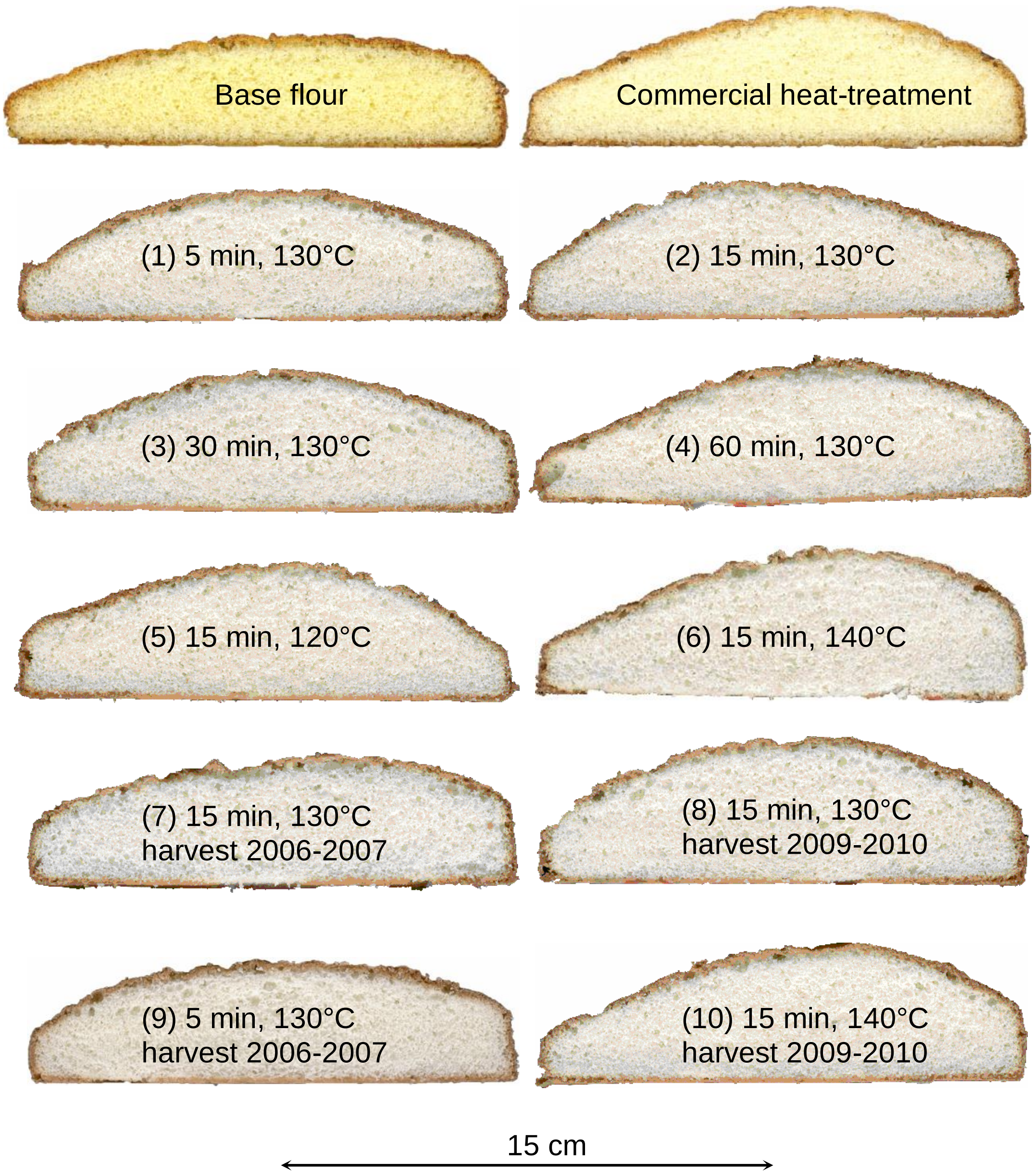


Figure 1 Schematic of heat treatment protocol



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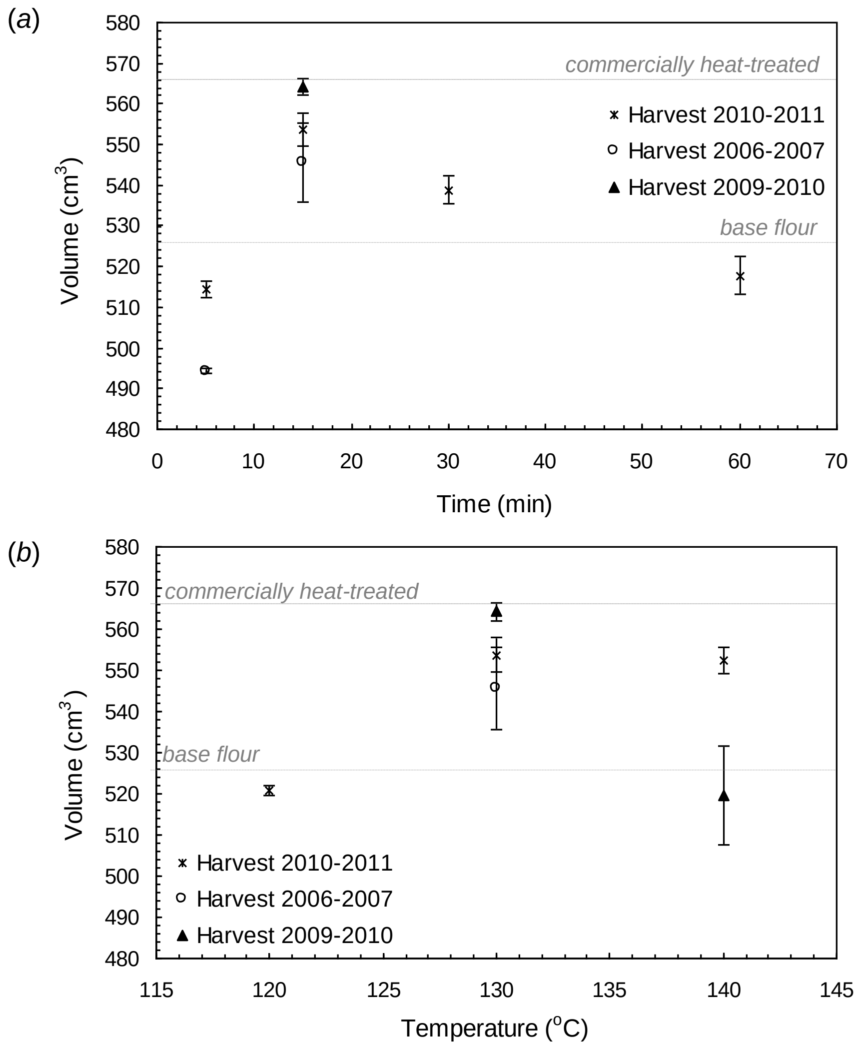

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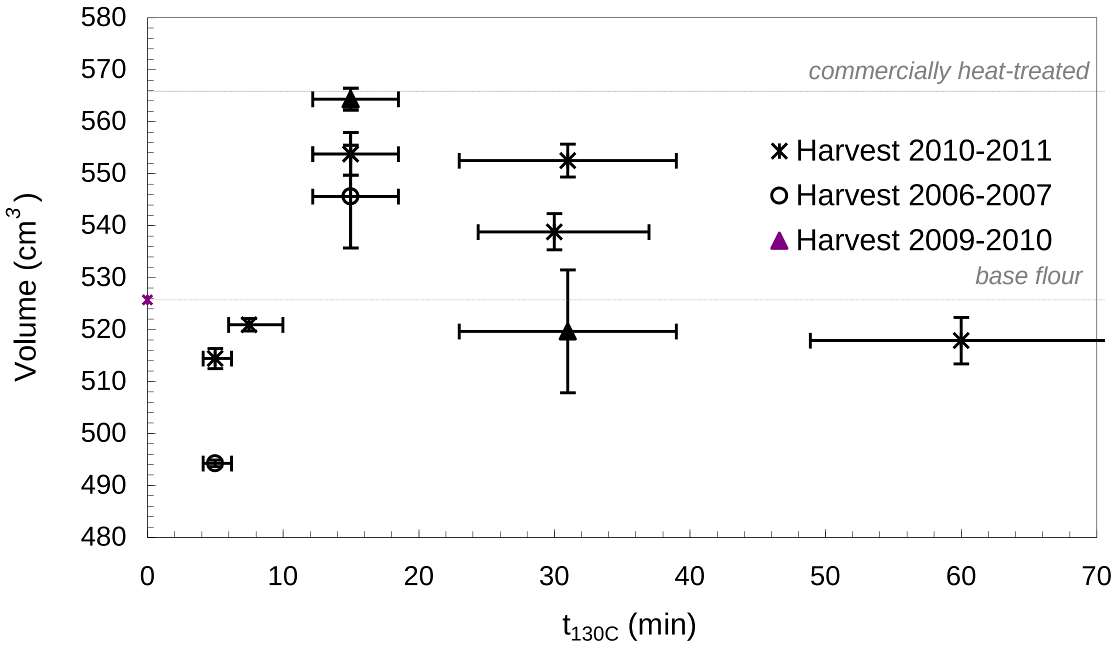

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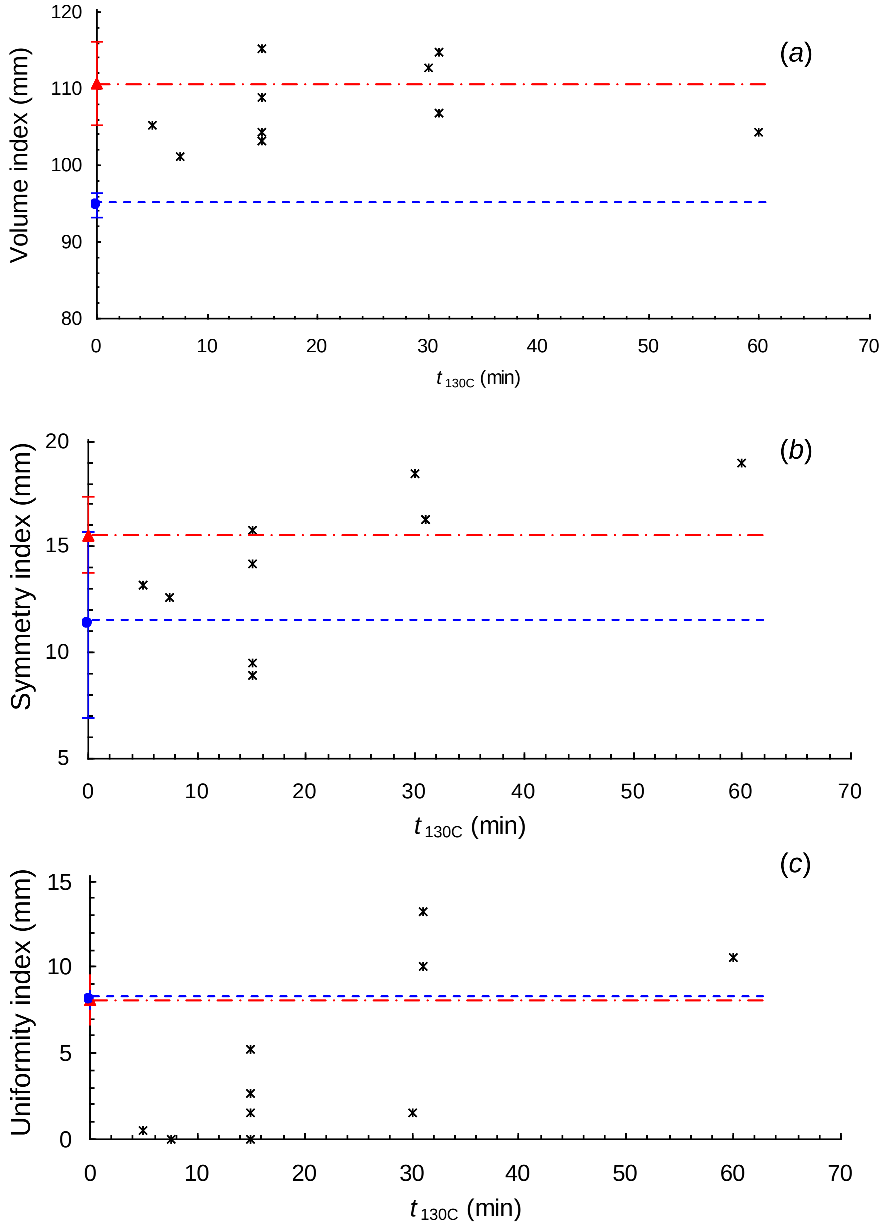

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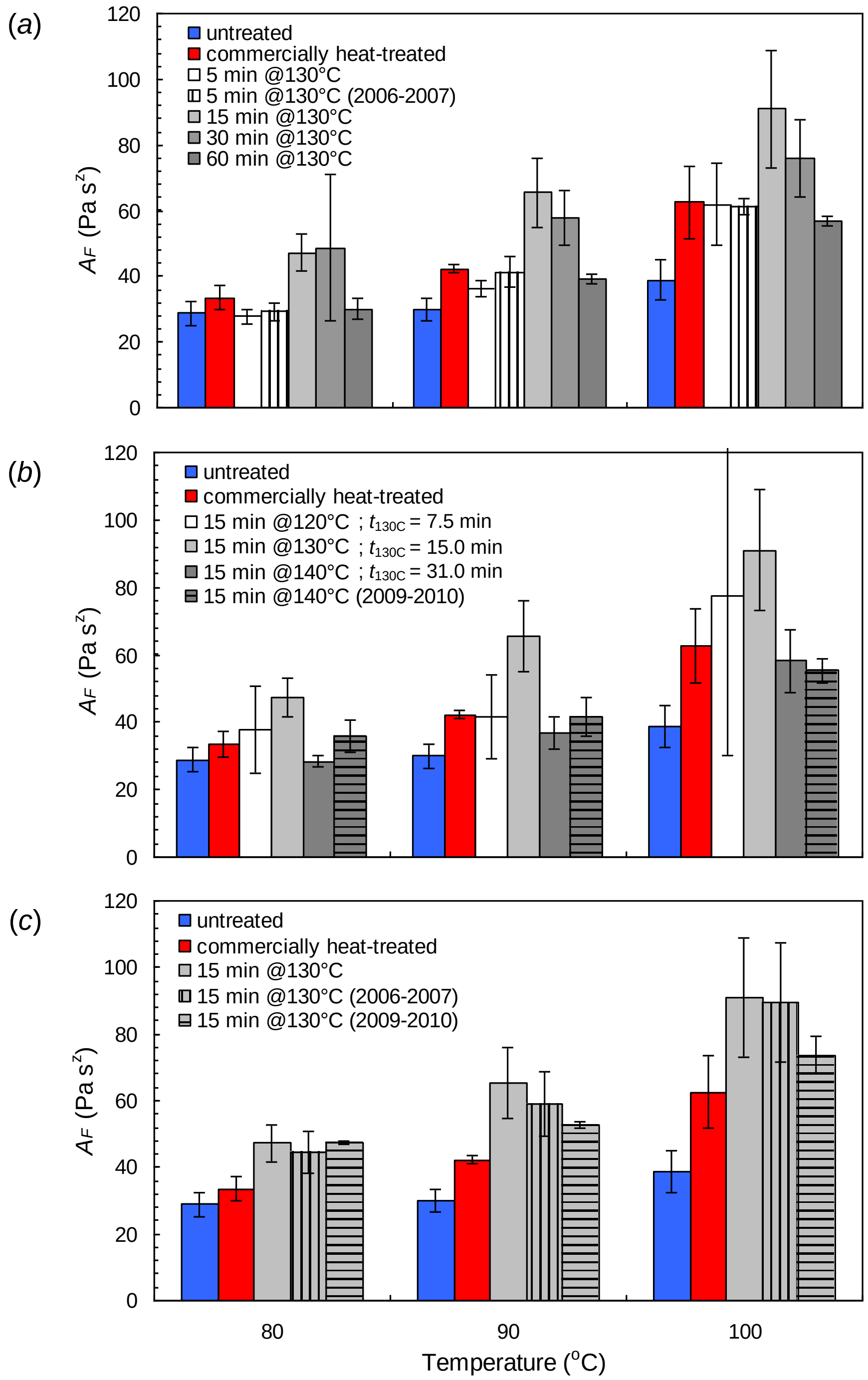

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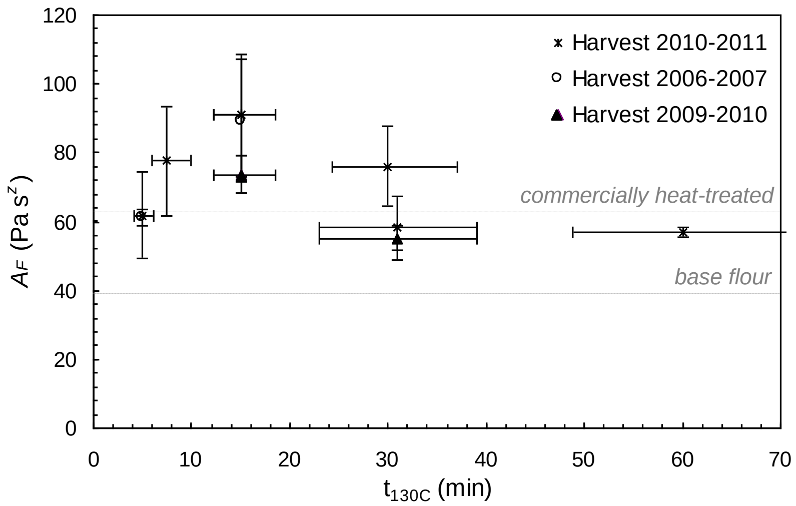

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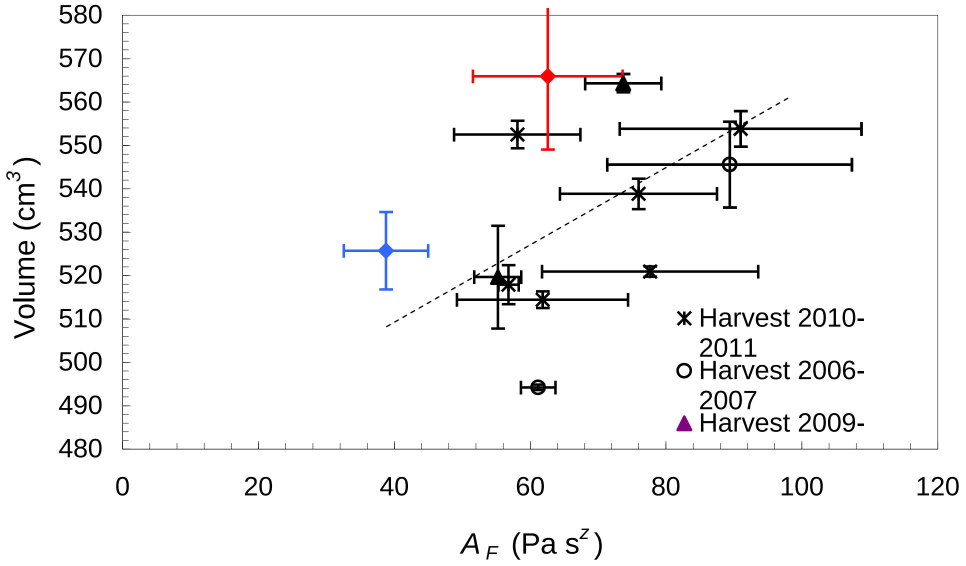

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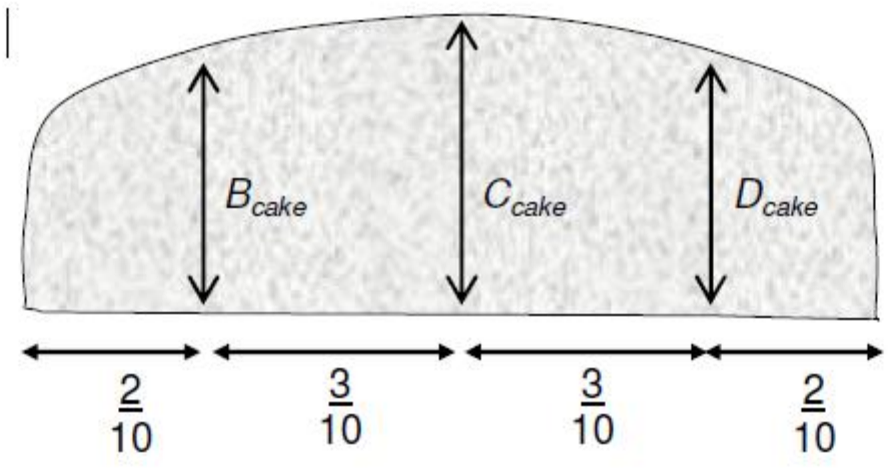
Appendix. AACC cake shape parameters

Figure A1 Cross section through cake showing measurements.



The height of the cake is measured at the three positions on a diameter shown above. The indices are calculated from

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

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

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

The volume index gives an indication of the overall size of the cake. The symmetry index assesses how peaked the cake is, while the uniformity index reflects how central the cake peak is.