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1	Optimisation of time /temperature treatment, for heat treated soft wheat flour
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24 Abstract

25 Chlorination of wheat flour in the EU countries has been replaced in recent years, to some extent, by heat treated flour which is used to produce high ratio cakes. Heat treated flour 26 27 allows high ratio recipes to be developed which generate products with longer shelf life, finer 28 texture, moist crumb and sweeter taste. The mechanism by which heat treatment improves the 29 flour is not fully understood, but it is known that during the heat treatment process, protein 30 denaturation and partial gelatinization of the starch granules occurs, as well as an increase in 31 batter viscosity. Therefore, it is important to optimize the flour heat treatment process, in 32 order to enhance baking quality. Laboratory preparation of heat treated base wheat flour 33 (culinary, soft, low protein) was carried out in a fluidised bed drier using a range of 34 temperatures and times. The gluten was extracted from the final product and its quality was tested, to obtain objective and comparative information on the extent of protein denaturation. 35 36 The results indicated that heat treatment of flour decreases gluten extensibility and partial gelatinization of the starch granules occurred. After heat treatment the gluten appeared to 37 retain moisture. The optimum time/temperature for the heat treatment of base flour was 38 120° C to 130° C for 30 min with moisture content of $\approx 12.5\%$. 39

40

41 *Keywords*: Heat treated flour; Baking; Bulk density; Gluten extensibility; Gelatinisation,
42 Viscosity.

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48 1- Introduction

49 Heat treated flour can be used in many applications in food processing such as cake, biscuit and wafer flours; beadings, batter flour, coatings; soup, sauces, baby food; thickeners for 50 specialities in industrial applications. Heat treated flour was first cited by Russo and Doe 51 52 (1970) who patented the process using a temperature range of 100° C to 115° C for a time of 53 60 min. Cauvain et al., (1976) suggested a heat treatment for whole wheat and semolina, 54 which was dried to a moisture content of 6% and then heated for a specific time/temperature, after which the grain was milled in the normal way. In 1979, Hanamoto and Bean patented a 55 method for producing heat treated flour, where the temperature was maintained at 71°C for 4-56 5 days. Guy and Mair (1993) first heat treated base flour gently to a moisture < 5% w/w, 57 58 when the gluten was not extensively denatured. After the initial drying process, the flour needed to be heated to a temperature of 130°C to 140°C for 30 minutes to achieve the 59 optimum performance. Nakamura et al., (2008) observed an increase in the volume of 60 Kasutera cake by dry heating of wheat flour at 120°C for 30 min. 61

By applying heat treatment to base flour and removing the moisture, it is possible to modify 62 its physical, rheological and bacteriological properties. The removal of moisture is an integral 63 64 part of achieving the overall changes required, but it is important that the flour moisture is 65 adjusted back to 12 % (wet basis), for optimum baking results such as decrease in bulk 66 density and an increase in cake height, which contributes to the overall quality such as 67 appearance and mouth feel. Cake batters made with heat-treated flour have been reported to 68 exhibit higher viscosity compared with batters made with untreated flour (Sahin, 2008). 69 Several studies on the effect of heat treatment on physical and chemical characteristics of 70 wheat flours have been published (Ozawa et al., 2009). Purhagen et al. (2011) studied the use 71 of normal and heat-treated barley flour along with waxy barley starch as anti-staling agents in

72 laboratory and industrial baking processes. Baked breads with heat-treated barley flour 73 differed from control breads with regard to water content, firmness and amylopectin 74 retrogradation. The addition of barley additives as native- or heat-treated flour or native- and heat-treated waxy starch resulted in increased water contents in the fresh bread as well as 75 76 increased water retention during storage compared to the control. The rheological behaviour 77 of high ratio cake batters prepared with untreated and heat-treated wheat flours were also 78 studied by Meza et al. (2011). They found that materials prepared with heat-treated flours exhibited greater stability, as indicated by slurry thixotropy and cohesive energy, and the 79 80 change in apparent viscosity and air content of foams and aerated emulsions on extended 81 mixing. The gel network generated in aerated emulsions prepared with heat-treated flours 82 was significantly stronger than those made with unheated flours. The objectives of this work 83 are to determine optimum time/temperature treatment of base flour to produce heat treated 84 flour by evaluating protein quality, gelatinization temperature, and peak viscosity of the flour, 85 and to evaluate baking height and bulk density of Madeira cake. Heat treated flour is more suitable than base flour for producing high ratio cakes because it improves quality in terms of 86 87 finer texture and lower bulk density.

88

2- Materials and Methods

89 2.1 Heat treated flour preparation:

The heat treated flour (batches of 400 grams) was prepared from a commercial base flour in a fluidised bed dryer (Sherwood Scientific Model MK11, UK), using a range of times (5 to 30 minutes) and temperatures (80 to 130° C), when the moisture content was reduced from 12.5% (wet basis), to moisture of < 8%.(wet basis) The moisture content of the heat treated flour was increased to the original moisture value using a Kenwood mixer (model KM199, UK), when required volume of water was sprayed onto the flour during mixing. Moisture

96	content analyses were carried out in duplicate ($\pm 0.01\%$) using a convective oven at 130 ^o C for
97	90 minutes. The temperature was adjusted via the control on the front of the dryer and the
98	velocity was set at maximum (air flow setting 9). The temperature and air velocity were
99	checked using a digital thermometer, (range -50° C to 1300° C $\pm 0.3\%$ rgd $+ 1\%$) and a hot
100	wire anemometer, (range 0.4 to 30 m/s \pm 3%) before the start of each experiment.
101	2.2 Cake batter preparation:
102	Madeira cake batter was prepared based on the ingredients and procedures used by Cook,
103	(2002) (Table 1).
104	1. All the ingredients were allowed to reach $20-21^{\circ}$ C before mixing.
105	2. Dry ingredients were sieved together using a 1 mm hand sieve.
106	3. The glycerine, egg and water were placed into the Hobart mixing bowl.
107	4. The sieved dry ingredients and shortening were added to the Hobart bowl.
108	5. The ingredients were mixed on speed 1 for 30 s, mixing was continued on speed 2 for
109	1 min.
110	6. The batter was then mixed on speed 3 for 7 min.
111	7. The batter was scaled into lined tins (350 g).
112	8. The cakes were baked in a conventional oven.
113	2.3 Baking equipment and procedure:

Both heat treated and untreated (base) flour were used to bake the Madeira cakes. A Glenlab convective oven (model OV125L, UK) was used thermostatically controlled to $\pm 0.75^{\circ}$ C over the temperature range 30°C to 250°C. Before each experiment, the oven was allowed to stabilize at the selected temperature for two minutes. For each experiment, a predetermined weight of batter (350 \pm 0.1g) using a Mettler balance (model PJ3000) was placed in the centre of the middle shelf. Each experiment was carried out in duplicate with a control. The

temperature of the oven was set at 175° C for 45 minutes and measured with a Type K Chromel TM AlumelTM thermocouple (1mm diameter) using a digital thermometer, accurate to $\pm 0.3\% + 1^{\circ}$ C in the range -50° C to 1000° C.

123 After baking, the cake was removed from the oven, and its height was measured along five 124 positions using digital callipers accurate to 0.01 mm. Bulk density was calculated by 125 averaging the results of ten slices of the cake, prepared using a Cookworks food slicer set to 126 15 mm thick, and the area measured with digital callipers and weighed on a two decimal 127 place balance. Viscosities were measured on a Brookfield RVDV-11+Pro Cone/Plate viscometer (Cone No51 radius 1.2cm), which was linked to a PC with RHEOCAL32 128 129 software installed. (Viscosity accuracy $\pm 1.0\%$ of full scale range, viscosity repeatability \pm 0.2% and temperature accuracy $\pm 1^{\circ}$ C over 100° C range). The water bath temperature was set 130 at 25° C and allowed to equate before analysis started. The gap between the cone and plate 131 132 was set electronically to 0.0127mm A Brookfield viscosity standard fluid was used to check the calibration before use. All 5 ml samples were checked in triplicate, (accuracy $\pm 2\%$ 133 134 S.D.217 mPa.s).

Gluten was extracted from the flour by the following method. Flour was hand washed 135 136 according to the standard method (Smiling, 1995). This was carried out by kneading the 137 dough sample approximately 10 min under cold running water until the water was clear and 138 then rested for 30 min before analysis began. The sample was then placed in a test tube and 139 centrifuged for 5 min at 5000 rpm, to remove air bubbles in the sample. After centrifugation, 140 the sample was removed gently from the test tube and 15g of gluten placed onto the grooved 141 base. Both the base and top was brushed lightly with paraffin oil to avoid adhesion when 142 removing samples. The top block was pushed firmly into the sample until the two blocks came together, then pressed for 40 min at 24° C (these parameters used for all samples) to 143

allow the gluten to relax. The gluten from the press was then removed and moulded into finestrips, which were used for analysis.

146 The gelatinisation temperatures and peak viscosity of heat treated flour were measured. The 147 Brabender viscograph measured the viscosity of flour slurry in a rotational stainless steel 148 bowl (75 rpm) as the temperature increased /decreased by 1.5°C. A measuring sensor, placed 149 in the slurry is deflected depending on the viscosity of the sample. Results are recorded on a 150 chart and are measured in Brabender Units (BU). The flour slurry was made up of $80g \pm$ 151 0.003 g (as is) of flour and 450 g \pm 0.5g of purified water. Slurry was placed in the bowl, heated to 92.5°C at 1.5°C per minute, and held at this temperature for 20 minutes. (Starting 152 temperature for the test is normally 45° C). The start of gelatinisation temperature is the 153 154 temperature at which viscosity increases by 20 BU. Individual starch granules gelatinise over 155 a temperature interval. As the temperature increases more starch granules swell then start to 156 break down and the peak viscosity starts to decrease. The temperature at peak viscosity (peak temp) is defined as the gelatinisation temperature over time. Peak viscosity is the BU at peak 157 158 temperature.

159

3- Results and Discussion

160 The optimum time/temperature profiles for the heat treatment of culinary flour are presented 161 in this section. A commercially heat treated flour was used as a control, and the laboratory 162 heat treated flours, to produce Madeira cakes. The parameters studied were cake height and 163 bulk density, in order to establish the optimum profile (time/temperature) for base heat 164 treated flour.

165

167 *3.1 Baking height:*

Base flour, heat treated at 80° C, increased slightly in baking height (3.8%). As the 168 temperature increased, the baking height also increased to 10.7% at 130° C. The results also 169 170 showed that time in the oven contribute to an increasing baking height. Heights were very similar when the base flour was heat treated at 120°C and 130 °C for 30 minutes. Guy and 171 172 Mair (1993) and Cauvain (1999) suggested that to achieve optimum improvement, the flour had to be heated gently to reduce moisture < 5% w/w before heating at temperatures between 173 130° C to 140° C, for 30 minutes. When flour drying temperatures increased above >140°C 174 there were cooked taints in the flour. 175

Figure (1) shows the baking result for the optimum time/temperature (30 min at 130^oC). Figure (2) shows the moisture level of the heat treated flour that is required to produce the optimum baking height. From the results, final flour moisture below 4 % gave the best result. Cauvain (1999) suggested that final flour moisture should be less than 8% and closer to 4%. The monitoring of moisture level could be a guide, when producing commercial heat treated flour, because of the improved quality of the Madeira cake. Figure (3) shows Madeira cakes produced from culinary flour, optimum heat treated and commercially heat treated flour.

The cake baked using culinary flour was similar in cake height to the control sample when removed from the oven, but collapsed when it cooled to room temperature, indicating that the product had no internal texture. The Madeira control cake was the benchmark for all the baking trials, which demonstrated a cake with a good height, a well defined central crack, and a good crust colour. The cake produced from optimum heat treated flour had a less defined central crack and the contour surface was similar to the control but more pitted.

Figure (4) shows bulk density of Madeira cakes made from heat treated base flour, at the different oven temperatures and times. Cakes were produced from base and commercially

heat treated flour (control). The base flour had a higher bulk density than the control and cakes produced from flour at $130 \,{}^{0}$ C, had also collapsed and therefore were more compacted. At the lower temperatures from 80^{0} C to 120^{0} C the cakes had not fully developed and therefore had not gained the height of the control, resulting in higher bulk densities. This was not the case for cakes produced from flour at 130^{0} C and control; they expanded to their normal height, giving a smaller bulk density, i.e., a more open structure which contributes to a more desirable mouth feel by the consumer.

198 *3.2 Batter viscosity:*

It is important to understand rheological characteristics of food material, for plant and 199 200 product design. The quality of cakes such as volume and texture can be attributed to cake 201 batter rheological properties. Figure (5) shows that the untreated, and heat treated flours at 80°C and 100°C have low viscosity values. Heat treatment of flour denatures most protein at 202 203 temperatures from 50° C to 80° C (Slade and Levin, 1995) and reduces their solubility in 204 water. Heating leads to disulphide bond linked aggregates and conformational (Attenburrow 205 et al., 1990) changes affecting mostly gliadins and low molecular weight albumins and 206 globulins (Guerrieri et al., 1996). Falcão-Rodrigues et al., (2005) suggested that the gliadins 207 have a significant role by imparting a higher viscosity. This was also confirmed by Attenburrow et al. (1990), Kokini et al. (1994) and (Kim et al., 2004), who suggested that due 208 209 to cross-linkage reactions, gluten viscosity increases on heating, because of the swelling and 210 gelatinization of starch granules, which also has a major factor on batter viscosity. A 211 combination of starch swelling, gelatinisation and protein denaturing, resulted in higher 212 viscosities in batters using heat treated flour compared to those using base flour.

213

215 *3.3 Brabender viscograph viscosities:*

In Table (2) the gelatinization temperature for the heat treated base flour is within a narrow 216 range of 57.2°C to 57.8°C, at two high temperature heat treatments. The base flour, which 217 218 was not heat treated, had a gelatinization temperature of 58.9°C. It also shows that the peak 219 viscosity progressively increases as the temperature/ time increases suggesting that a higher 220 number of starch granules have swollen, giving high peak viscosities. The control sample has 221 similar peak viscosity (320 BU). However, the culinary flour had a higher gelatinisation temperature of 58.9°C and a peak viscosity of 220 BU, suggesting a much lower level of 222 223 swollen starch granules present. In Figure (6) only one temperature was selected (100^oC) and 224 heat treated through a range of times. This behaviour is repeated again. As the time increases 225 there was an increase in peak viscosity and a change in gelatinization temperature. From the 226 results, heat treatment appears to have increased the ability of the starch granules to absorb 227 more water. To some degree, denatured proteins may have contributed to the level of 228 viscosity and also the mechanical damage to starch at milling. The proteins on the surface of 229 the starch are hydrophilic albumin and globulin and could be modified by chlorine and heat 230 treatment (Barlow et al., 1973). Seguchi (1984) showed that hydrophobicity of starch 231 granules increased with such treatment. Similar increases were observed when flours were 232 heat treated (Johnson et al., 1980). The modification of these proteins may allow the starch 233 granules to absorb more water. The culinary flour had a peak viscosity of 220 BU suggesting 234 that the starch granule surface had no modification and a barrier remained with less ability to 235 absorb water. Johnson et al. (1980) suggested the effects of heat treatment of starch improved 236 baking quality, viscosity being the controlling factor for the final cake volume.

237

239 *3.4 Gluten extensibilities:*

Gluten extensibility is a measure of the degree of protein denatutation in the flour due to the extent of heat treatment which may affect the viscosity of the cake batter, an important parameter in the quality of the baked product. Increased viscosity leads to an increase in baking height, which can result in a more attractive appearance.

244 The extensibilities show a decreasing extensibility (mm) possibly due to a reduction in gliadin due to heat treatment. This can be seen in Figures (7), (8) and (9) which represent the 245 246 extensibility of base flour, heat treated base flour (120°C for 30 min) and heat treated base flour (130[°]C for 30 min) glutens. The gluten samples had also lost their cohesion, indicating 247 248 that there was a reduction in gliadin level. These figures suggest that the heat treated base 249 flour contains more gluten than the non treated base flour. This may be due to the gluten 250 being easier to wash and retains less starch. This is in agreement with results obtained by 251 Ritchie (1985). During hand extraction, the heat treated flour was less cohesive than the 252 culinary gluten. Heat treated gluten broke down in small aggregates of gluten. Flours that were heat treated and then rehydrated to 12% moisture appear to retain their moisture and 253 254 give higher gluten weights Table (3). This may be a factor in the migration of moisture 255 during storage of the baked product. These results are based on heat treatment at two 256 time/temperature combinations, but may require a range of time/ temperature treatment and 257 different wheat varieties which may react differently to heat.

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Conclusions:

263 On the basis of this work the following conclusions can be drawn.

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- Heat treatment of base flour acts as an improver, by increasing Madeira cake height,
 and bulk density, whereas base flour without heat treatment produces poor high ratio
 cakes with high bulk density and low height.
- Optimum time/temperature (120°C to 130°C for 30 min) heat treatment of base flour
 with moisture 12.5% can produce good quality Madeira cake.
- Flour heat treatment affects viscosity which has an important role in final cake
 quality. Batter viscosity increase may be due to denatured gluten and partial starch
 granule gelatinization.
- Flour heat treatment decreased gluten extensibility and reduced cohesion which has
 positive effects on baking quality as the gluten appears to retain its moisture whereas
 base flour gluten retained its extensibility and cohesion.

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Figure 1: Effect of heat treatment on cake height made from flours produced at different temperatures and times.





Figure 2: Moisture level of the heat treated flour required to produce the optimum baking height. Shaded bars represent moisture content; black bars are equivalent baking height.









Figure 4: Madeira cake bulk densities, baked for 30 min.













 CERT





447	Figure 9: Effect	of base flour hea	at treated $(130^{\circ} \text{ C for } 30 \text{ min})$.



Table 1: Madeira cake recipe.

	Flour (Culinary)	200 g	
	Skimmed Milk Powder	14 g	
	Salt	5 g	
	Baking Powder	8 g	
	Castor Sugar	230 g	Q
	Water	140 g	
	Eggs	160 g	
	Glycerine	16 g	
	Shortening	120 g	
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Prepared Flour	Moisture%	Protein %	Peak	Gelatinisation Temp (°C)
			Viscosity	
			BU	
10 min at 120 ⁰ C	11.0	9.5	321	57.5
30 min at 120 ⁰ C	10.8	9.6	326	57.8
60 min at 120 ⁰ C	10.6	9.9	371	57.4
10 min at 130 ⁰ C	11.5	9.5	323	57.3
30 min at 130 ⁰ C	9.2	9.7	366	57.4
60 min at 130 ⁰ C	8.9	9.8	491	57.2
Control	12.2	9.4	320	57.8
Base	13.1	9.5	220	58.9
			P	>

Table 2: Flour analysis for treated, untreated and control flours.

Table 3: Gluten extracted from culinary and heat treated culinary flour (optimum temp 130°C for 30 min).

486	Culinar	y Flour	Heat treate	d Culinary
487	Wet wt	Dry wt	Wet wt	Dry wt
488	3.8g 3.5g	2.7 g 2.6 g	5.6g 5.3g	3.7 g 3.5 g
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497	Research Highlights:
498	• Heat treatment of flour improved cake quality such as baking height and volume.
499	Heat treatment of flour decreases gluten extensibility.
500	• Optimum time/temperature was 120° C- 130° C for 30 min with moisture content of
501	12.5%.
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