GAS CELLS IN BREAD DOUGH

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Linda Trinh

School of Chemical Engineering and Analytical Science
Contents

Contents .......................................................................................................................... 2
List of Tables .................................................................................................................. 5
List of Figures .................................................................................................................. 6
Nomenclature .................................................................................................................. 12
   Abbreviations .......................................................................................................... 12
   Notation .................................................................................................................... 12
Abstract ......................................................................................................................... 14
Declaration ...................................................................................................................... 16
Copyright Statement ...................................................................................................... 16
Acknowledgements ........................................................................................................ 17
Chapter 1 - Introduction: Understanding bread dough aeration ..................................... 18
Chapter 2 - Bread: Its history, significance and manufacture ........................................... 23
   2.1 An introduction to bread ...................................................................................... 23
      2.1.1 The significance of bread ............................................................................ 23
      2.1.2 Industrial breadmaking - the Chorleywood Bread Process ....................... 25
      2.1.3 Breadmaking ingredients .......................................................................... 27
   2.2 Aeration throughout breadmaking ....................................................................... 33
      2.2.1 Entrainment and disentrainment of gas ....................................................... 33
      2.2.2 Parameters during mixing affecting cells in bread dough ......................... 36
      2.2.3 Measuring aeration throughout breadmaking ............................................ 39
      2.2.4 Quantifying aeration using a population balance model ......................... 45
   2.3 Summary of bread’s history, significance and manufacture .................................. 48
Chapter 3 - Experimental methodologies ...................................................................... 49
   3.1 Outline .................................................................................................................. 49
   3.2 Materials used ..................................................................................................... 49
   3.3 Equipment used .................................................................................................. 50
      3.3.1 Tweedy 1 mixer .......................................................................................... 52
      3.3.2 Nikon Metris 225/320kV X-ray CT ................................................................. 54
      3.3.3 Farinograph .................................................................................................. 56
      3.3.4 Micro-Dough lab ......................................................................................... 56
      3.3.5 C-Cell .......................................................................................................... 56
      3.3.6 Texture Analyser ......................................................................................... 57
   3.4 Analytical techniques used .................................................................................. 58
      3.4.1 Determining flour water absorption in the Farinograph .............................. 58
      3.4.2 Determining flour water absorption on the Micro-Dough Lab .................. 58
      3.4.3 Measuring dough density ............................................................................ 58
      3.4.4 Determining the gas free dough density ..................................................... 60
      3.4.5 Determining dough voidage ....................................................................... 60
4.11 The effect of sugar on finished bread .................................................. 107
  4.11.1 Method: The effect of sugar on finished bread .................................. 107
  4.11.2 Results and discussion: The effect of sugar on finished bread .......... 109
  
  4.12 Summary .......................................................................................... 124

Chapter 5 - Aeration in bread dough with and without sugar .................... 126

  5.1 Introduction ....................................................................................... 126
  5.2 Aeration of bread dough with and without sugar methodology .......... 126
    5.2.1 Experimental methodology ......................................................... 127
    5.2.2 Pressure step change modelling methodology ............................ 129
  5.3 Results and discussion ...................................................................... 129
    5.3.1 Experimental results .................................................................... 130
    5.3.2 Pressure step change modelling results ..................................... 140
  5.4 Summary .......................................................................................... 143

Chapter 6 - X-ray computerised tomography of bread dough during mixing and proving ...... 145

  6.1 Introduction ....................................................................................... 145
  6.2 X-ray CT of non-yeasted dough with and without sugar .................... 146
    6.2.1 Method: X-ray CT of dough with and without sugar ...................... 146
    6.2.2 Results and discussion: X-ray CT of dough with and without sugar .... 147
  6.3 X-ray CT of non-yeasted non-sugar containing bread dough throughout mixing with a pressure step change ........................................... 153
    6.3.1 Method: X-ray CT of bread dough without sugar throughout mixing with a pressure step change .......................................................... 153
    6.3.2 Results and discussion: X-ray CT of bread dough without sugar throughout mixing with a pressure step change .......................................................... 154
  6.4 X-ray CT of bread dough during proving .......................................... 164
    6.4.1 Method: X-ray CT of bread dough during proving ........................ 165
    6.4.2 Results and discussion: X-ray CT of bread dough during proving ........ 167
  6.5 Summary .......................................................................................... 185

Chapter 7 - Conclusions and further work ................................................ 187

  7.1 Conclusions ..................................................................................... 187
  7.2 Further work .................................................................................... 188

Chapter 8 - References ............................................................................. 191

Chapter 9 - Appendices ............................................................................. 201
List of Tables

Table 2.1 Techniques used for imaging cell size distribution ........................................ 41
Table 3.1 Experimental materials and their procurement sites ........................................ 49
Table 3.2 Experimental equipment, their makes and information on use of the equipment 51
Table 3.3 Flours and their corresponding water absorption values. These were determined
from the Micro-Dough Lab, Farinograph and packaging. The final column gives the water
absorption value used in experiments within this thesis, based on these measurements .... 62
Table 3.4 Formulation used to determine the gas free dough density of a dough made from
flour a .................................................................................................................. 63
Table 3.5 Formulation used to determine the gas free dough density of a dough made from
flour c .................................................................................................................. 63
Table 3.6 Characterisation of flours a and c ................................................................. 64
Table 4.1 Dough formulations used to assess the voidage and gas free dough density of
different sugar content strong flour doughs. ...................................................... 70
Table 4.2 Dough formulations used to assess the voidage and gas free dough density of
different sugar content weak flour doughs ...................................................... 71
Table 4.3 Strong flour dough formulations used in the Farinograph ............................ 76
Table 4.4 Weak flour dough formulations used in the Farinograph ............................ 76
Table 4.5 Dough formulations used for torque measurements during mixing in the Tweedy 1
mixer .................................................................................................................. 82
Table 4.6 Dough formulations and the mixing speeds used to assess aeration of different
sugar content doughs at different mixing speeds .............................................. 86
Table 4.7 Strong flour dough formulations used to assess the effect of sugar on dough
adhesiveness .................................................................................................. 87
Table 4.8 Texture analyser settings used for dough adhesiveness test .......................... 88
Table 4.9 Texture analyser settings used for uniaxial extension of dough .................... 91
Table 4.10 Texture analyser settings used for dough uniaxial extension tests .............. 93
Table 4.11 Strong flour dough formulations used for ESEM experiments ..................... 98
Table 4.12 Strong flour dough formulations used to observe the dynamic dough density
during proving at 38ºC ..................................................................................... 102
Table 4.13 Weak flour dough formulations used to observe the dynamic dough density
during proving at 38ºC ..................................................................................... 103
Table 4.14 Formulations of baked bread loaves ......................................................... 107
Table 5.1 Strong flour dough formulations used to derive entrainment and disentrainment
values from a population balance model on the aeration of bread dough .......... 127
Table 5.2 Weak flour dough formulations used to derive entrainment and disentrainment
values from a population balance model on the aeration of bread dough .......... 127
Table 5.3 Weak flour dough formulations used to derive entrainment and disentrainment
values from a population balance model on the aeration of bread dough .......... 127
Table 5.4 RMSEs for the no breakup population balance fitted to the experimental pressure
step change data .............................................................................................. 140
Table 6.1 Dough formulations used for comparison of 0% and 15% sugar dough via X-ray CT
....................................................................................................................... 146
Table 6.2 Formulations and mixing times used for comparison of pressure-vacuum and
constant pressure mixed doughs via X-ray CT .............................................. 154
Table 6.3 Sample volumes and resolutions for X-ray scans ...................................... 154
Table 6.4 Dough formulation used for X-ray CT of bread dough proving .................... 165
Table 6.5 Percentage error at different thresholding levels throughout dough proving .... 169
List of Figures

Figure 1.1 A selection of some point of difference UK bread products. Products illustrated include Kingsmill 50:50, for those who want the goodness of wholegrain without its texture, Hovis British Farmers Loaf, a loaf made with 100% British wheat, Hovis seed sensation, containing seven seed varieties, Kingsmill Little Big Loaf, full sized bread slices but in a smaller pack size and Warburtons Sandwich thins, who want less bread in their sandwiches. ....................................................................................................................... 18

Figure 1.2 A selection of bakery snacks available in UK supermarkets. Illustrated include traditionally seasonal products such as hot cross buns, products released for a specific event, such as the red nose day doughnuts, world breads such as croissants and brioche, and traditional UK bakery snacks such as scones and pancakes to name a few. ............................... 19

Figure 2.1 The molecular structure of the disaccharide, sucrose. Sucrose consists of the two monosaccharides, α-D-glucose and α-D-fructose, linked by a glycosidic bond. ................................................................. 32

Figure 2.2 Unpublished voidage measurements obtained from X-ray CT. The results show the voidage measured from X-ray CT and the double cup method, illustrating the large disparity between the measurements. .............................................................. 45

Figure 3.1 Inside the Tweedy mixer .................................................................................. 52

Figure 3.2 Outside the Tweedy 1 mixer with mixer lid in position ........................................ 52

Figure 3.3 Tweedy 1 mixer set up. This shows some of the equipment used alongside the Tweedy mixer to control dough mixing and record mixing parameters ................................. 53

Figure 3.4 Nikon Metris 225/320KV X-ray CT. The source, manipulator stage and detector positions can be adjusted to optimise scan settings ......................................................... 55

Figure 3.5 Nikon Metris 225/320KV X-ray CT set up. This shows the equipment housed in a customised bay, whilst control of the equipment is located outside the bay for safety purposes. ........................................................................................................ 55

Figure 3.6 Texture analyser. A Stable Microsystems instrument used within the food industry to quantify the texture and physical properties of products. A range of attachments to the arm and testing bed can be used depending on the property being measured. ............................... 57

Figure 3.7 (a) Double cup system, used to measure the dough density through calculations involving weight measurements of dough in a fluid and air (b) Double cup ...................... 59

Figure 3.8 Protein content vs. water absorption values of flours. 78 values flours are illustrated, including 75 flours in literature and 3 flours used throughout this thesis. ............... 62

Figure 3.9 Density of doughs made from flour a and c, following mixing at a range of headspace pressures for 180 s. The data points are the mean of six measurements from an identical batch of dough. Error bars have been omitted as they are smaller than the markers used. Linear regression trend lines are shown. ...................................................... 63

Figure 3.10 Gas free dough density of flours from literature and flours a and c ...................... 64

Figure 4.1 Density of different sugar content strong flour doughs following 180 s of mixing at different mixer headspace pressures. The data points illustrated are the mean of six measurements. Error bars have been omitted as they are smaller than the symbols used. Linear regression lines are shown. ................................................................. 72

Figure 4.2 Density of different sugar content weak flour doughs following 180 s of mixing at different mixer headspace pressures. The data points illustrated are the mean of six measurements. Error bars have been omitted as they are smaller than the symbols used. Linear regression lines are shown. ................................................................. 72

Figure 4.3 Voidage of different sugar content strong flour doughs, following 180 s of mixing at mixer headspace pressures ranging from 0.25 bara to 2.5 bara ........................................ 73

Figure 4.4 Voidage of different sugar content weak flour doughs, following 180 s of mixing at mixer headspace pressures ranging from 0.25 bara to 2.5 bara ........................................ 74

Figure 4.5 A typical Farinogram with significant features annotated ................................. 75

Figure 4.6 (a) 0% sugar strong dough Farinogram (b), 0% sugar weak dough Farinogram .......... 79

Figure 4.7 (a) 5% sugar strong dough Farinogram (b), 5% sugar weak flour dough Farinogram 79
Figure 4.8 (a) 10% sugar strong dough Farinogram (b), 10% sugar weak dough Farinogram . 79
Figure 4.9 (a) 15% sugar strong dough Farinogram (b), 15% sugar weak dough Farinogram . 79
Figure 4.10 Mixing time to Farinogram peaks in different sugar content doughs .................. 80
Figure 4.11 Dough consistency at significant times indicated by the Farinogram .................. 80
Figure 4.12 Torque of different sugar content doughs over mixing time whilst mixing at atmospheric pressure. The torques presented are based on a moving mean of 19 s, taken from the mean of three experiments. ................................................................. 83
Figure 4.13 Time to reach peak torque in different sugar content doughs. The data points illustrated are the mean of three measurements. ................................................................. 84
Figure 4.14 Peak torque in different sugar content doughs whilst mixing at atmospheric pressure for 240 s. The data points illustrated are the mean of three measurements. The error bars presented are one standard deviation on either side of the mean. .................. 84
Figure 4.15 End torque in different sugar content doughs, following mixing at atmospheric pressure for 240 s. The data points illustrated are the mean of three measurements. The error bars presented are one standard deviation on either side of the mean. .................. 84
Figure 4.16 Work done at end torque in different sugar content doughs, following mixing at atmospheric pressure for 240 s. The data points illustrated are the mean of three measurements. The error bars presented are one standard deviation either side of the mean. ................................................................. 85
Figure 4.17 Mean mixing speed during 4 minute mixing sessions of different sugar content doughs. Each data point is an average of 684 data points from three batches of dough. The error bars presented are one standard deviation either side of the mean. .................. 85
Figure 4.18 The density of 0% and 15% sugar strong flour dough mixed at different speeds under atmospheric pressure for the 180 s. The data points presented are the mean of six measurements from a single dough. Error bars have been omitted as they are smaller than the markers used. Linear regression trend lines are shown. ................................................................. 87
Figure 4.19 Set up for rolling of the dough to obtain the desired thickness ....................... 88
Figure 4.20 Texture analyser probing dough sample to assess adhesiveness .................... 88
Figure 4.21 Time and force required for application of 0.98 N of force with a probe to different sugar content doughs and then to return the probe to starting position. The data points illustrated are the mean of measurements from three samples from one dough batch. ................................................................. 90
Figure 4.22 Penetration depth of a probe when applying 0.98 N of force to different sugar content doughs and distance travelled by the probe on returning to the starting position. The data points illustrated are the mean of measurements from three samples from one dough batch. ................................................................. 90
Figure 4.23 Distance travelled by a probe to apply 0.98 N of force to different sugar content doughs. Each data point illustrated is the mean of three measurements from one dough batch ................................................................. 90
Figure 4.24 Two piece cylindrical mould used for shaping dough for uniaxial dough extension experiments ................................................................. 92
Figure 4.25 Dough sample prepared for uniaxial extension system. Due to the similarity in colour of the dough and background, the dough has been marked out in the image. The areas where cyanoacrylate was applied is highlighted in yellow. ................................................................. 92
Figure 4.26 Uniaxial extension system ................................................................. 93
Figure 4.27 Uniaxial extension system in action ................................................................. 93
Figure 4.28 Force involved with time during uniaxial extension of a dough piece at speeds of 6 mms⁻¹. Each point illustrated is a moving average of 60 points. ................................................................. 95
Figure 4.29 Distances involved with time during the extension of a dough piece at speeds of 6mms⁻¹. Each point illustrated is a moving average of 60 points. ................................................................. 95
Figure 4.30 Force required to snap different sugar content dough strips during uniaxial extension of the dough at a speed of 6 mm s⁻¹. The data points illustrated are the mean of six measurements. The error bars shown are one standard deviation either side of the mean. ................................................................. 96
Figure 4.31 Distance different sugar content dough strips were extended during uniaxial extension at a speed of 6 mm s\(^{-1}\) before snapping. The data points illustrated are the mean of six measurements. The error bars shown are one standard deviation either side of the mean. ................................................................. 96

Figure 4.32 The relationship between the force applied to a dough strip to cause it to snap and the distance the dough strip is extended before snapping, during uniaxial dough extension tests in different sugar content doughs ................................................................. 97

Figure 4.33 The engineering strain at snapping point on different sugar content doughs during uniaxial extension tests. The data points illustrated are the mean of six measurements. The error bars shown are one standard deviation either side of the mean. 97

Figure 4.34 The stress at snapping point on different sugar content dough strips as they snap during uniaxial extension tests. The data points illustrated are the mean of six measurements. The error bars shown are one standard deviation either side of the mean. 98

Figure 4.35 ESEM micrograph of 0% sugar dough mixed for 230 s with a step change from 1 bara to 0.5 bara at 110 s (a) at 600 x magnification (b) at 1200 x magnification............. 100

Figure 4.36 ESEM micrograph of 15% sugar dough mixed for 230 s with a step change from 1 bara to 0.5 bara at 110 s (a) at 600 x magnification (b) at 1200 x magnification............... 101

Figure 4.37 Change in density of different sugar content (a) strong doughs (b) weak doughs, during proving at 38°C. The results presented are the mean of four data measurements taken from two batches of dough. The error bars have been omitted for clarity. ................. 105

Figure 4.38 Proving time required for different sugar content strong and weak flour doughs, to reach their minimum density. The data points are the mean of four measurements, taken from two batches of dough. Error bars are one standard deviation either side of the mean. ........................................................................ 105

Figure 4.39 Minimum density reached by different sugar content (a) strong doughs (b) weak doughs when proving at 38°C. The data points are the mean of four measurements, taken from two batches of dough. Error bars have been omitted for clarity, as they were smaller than the markers used .................................................................................. 106

Figure 4.40 The percentage weight loss of different sugar content loaves as a result of baking. Each data point is the mean of three measurements from separate loaves. Error bars are one standard deviation either side of the mean. ........................................................................ 110

Figure 4.41 The specific volume of different sugar content loaves. Each data point is the mean of three measurements from separate loaves. The error bars are one standard deviation either side of the mean. ................................................................. 111

Figure 4.42 Density of different sugar content doughs mixed in the breadmaker. Each data point is the mean of six measurements from separate loaves. The error bars are one standard deviation either side of the mean. ................................................................. 112

Figure 4.43 Change in density during proving at 38°C of different sugar content strong flour doughs mixed in a breadmaker. The results presented are the mean of four data measurements taken from two batches of dough. The error bars have been omitted for clarity. ........................................................................ 112

Figure 4.44 The relationship between the density of different sugar content doughs immediately after mixing and their minimum density obtained during proving in breadmaker and Tweedy loaves. .................................................................................. 113

Figure 4.45 The relationship between the density of different sugar content doughs immediately after mixing and their density as finished bread in breadmaker and Tweedy loaves. Linear regression lines are shown. ........................................................................ 114

Figure 4.46 The relationship between the minimum density of different sugar content doughs obtained during proving and their density as finished bread in breadmaker and Tweedy loaves ........................................................................ 114

Figure 4.47 The force required to compress different sugar content loaves by 8 mm. Each data point is the mean of twelve measurements from three separate loaves. The error bars are one standard deviation either side of the mean. ........................................................................ 115
Figure 4.48 The relationship between the force required to compress bread 8 mm and the density of the bread in breadmaker and Tweedy loaves. Linear regression lines are shown. 115
Figure 4.49 The number of cells per mm² in different sugar content (a) breadmaker loaves (b) Tweedy loaves. Each data point is the mean of nine measurements from three separate loaves. The error bars presented are one standard deviation of the mean. 116
Figure 4.50 The mean cell diameter present in different sugar content (a) breadmaker loaves (b) Tweedy loaves. Each data point is the mean of nine measurements from three separate loaves. The error bars presented are one standard deviation of the mean. 117
Figure 4.51 Raw and analysed C-cell cell intensity images of slices of breadmaker produced bread of different sugar contents. 118
Figure 4.52 Raw and analysed C-cell cell intensity images of slices of Tweedy produced bread of different sugar contents. 119
Figure 4.53 The relationship between the cell number density and specific volume in breadmaker and Tweedy loaves. Linear regression lines are shown. 120
Figure 4.54 The relationship between mean cell diameter and the specific volume of breadmaker and Tweedy loaves. Linear regression lines are shown. 120
Figure 4.55 The cell size distribution in different sugar content finished breadmaker loaves. Each data point is measured from nine slices of bread from three separate mixes. 121
Figure 4.56 The number of cells of different sizes per slice in breadmaker bread. Each data point is the mean of nine slices of bread from three separate mixes. 122
Figure 4.57 The cell size distribution in different sugar content finished Tweedy loaves. Each data point is measured from nine slices of bread from three separate mixes. 122
Figure 4.58 The number of cells of different sizes per slice in Tweedy bread. Each data point is the average of nine slices of bread from three separate mixes. 123

Figure 5.1 (a) Voidage at 1 bara of strong and weak flour doughs containing 0%, 7.5% and 15% sugar (b) Gas free dough density of strong and weak flour doughs containing 0%, 7.5% and 15% sugar. 130
Figure 5.2 Experimental and model voidages during pressure-vacuum mixing. \( P_1 = 1 \) bara, \( P_2 = 0.5 \) bara. Each data point is from a separate dough mix. (a) strong flour, 0% sugar (b) weak flour, 0% sugar (c) strong flour, 7.5% sugar (d) weak flour, 7.5% sugar (e) strong flour, 15% sugar (f) weak flour, 15% sugar. 131
Figure 5.3 Experimental and model voidages during pressure-vacuum mixing. \( P_1 = 1 \) bara, \( P_2 = 0.25 \) bara. Each data point is from a separate dough mix. (a) strong flour, 0% sugar (b) weak flour, 0% sugar (c) strong flour, 7.5% sugar (d) weak flour, 7.5% sugar (e) strong flour, 15% sugar (f) weak flour, 15% sugar. 132
Figure 5.4 Experimental and model voidages during mixing with a pressure step increase from 1 bara to 2 bara. Each data point is from a separate dough mix. (a) strong flour, 0% sugar (b) weak flour, 0% sugar (c) strong flour, 7.5% sugar (d) weak flour, 7.5% sugar (e) strong flour, 15% sugar (f) weak flour, 15% sugar. 133
Figure 5.5 Initial model voidages of strong and weak flour doughs containing 0%, 5% and 15% sugar. 134
Figure 5.6 (a) Entrainment rate of strong and weak flour doughs containing 0%, 5% and 15% sugar (b) Disentainment coefficient of strong and weak flour doughs containing 0%, 5% and 15% sugar. 135
Figure 5.7 Ratio of entrainment rate to disentainment coefficient for strong and weak flour doughs containing 0%, 7.5% and 15% sugar. 136
Figure 6.1 Dough set up for X-ray imaging of static samples. 137
Figure 6.2 Orthoslice of (a) 0% sugar dough and (b) 15% sugar dough mixed in a Tweedy 1 for 230 s and subjected to a pressure change from 1 to 0.5 bara at 110 s. 138
Figure 6.3 Dough voidage, measured by the double cup method and Avizo via X-ray CT, for dough containing 0% sugar and 15% sugar, which were each mixed for 230 s in the Tweedy 1 with a pressure step decrease from 1 bara to 0.5 bara at 110 s. 139
Figure 6.4 Number of cells above cut-off per cm³ for dough containing 0% and 15% sugar, which were each mixed for 230 s in the Tweedy 1 with a pressure step decrease from 1 bara to 0.5 bara at 110 s. ........................................................................................................149
Figure 6.5 Mean cell volume above cut-off per cm³ for dough containing 0% and 15% sugar, which were each mixed for 230 s in the Tweedy 1 with a pressure step decrease from 1 bara to 0.5 bara at 110 s. ........................................................................................................150
Figure 6.6(a) The number density per unit volume of different cell volumes (b) The frequency of different cell volumes in a 0% and 15% sugar dough following mixing for 230 s in a Tweedy 1, with a pressure step decrease from 1 bara to 0.5 bara at 110 s. .........................151
Figure 6.7 Cumulative number of cells % above cut-off in 0% and 15% sugar dough following mixing for 230 s in a Tweedy 1, with a pressure step decrease from 1 bara to 0.5 bara at 110 s ........................................................................................................152
Figure 6.8 Cumulative voidage in 0% and 15% sugar dough following mixing for 230 s in a Tweedy 1, with a pressure step decrease from 1 bara to 0.5 bara at 110 s (a) per unit volume (b) percentage ........................................................................................................153
Figure 6.9 Orthoslices of dough with highlighted region size of 60 pixels square (a) Dough mixed for 110 s at constant pressure of 1 bara. Pixel resolution is 10.8 µm. (b) Dough mixed for 230 s at constant pressure of 1 bara. Pixel resolution is 8.6 µm. (c) Dough mixed for 230 s in total: first 110 s at 1 bara, and final 120 s at 0.5 bara. Pixel resolution is 7.1 µm. .......156
Figure 6.10 X-ray measured dough voidage of pressure-vacuum mixed dough and constant pressure mixed dough........................................................................................................157
Figure 6.11(a) The number density per unit volume (b) The frequency of different cell volumes within dough throughout pressure-vacuum mixing. The doughs were mixed at 1 bara for the first 110 s and 0.5 bara thereafter. ........................................................................................................158
Figure 6.12 Cumulative cell percentage for different volume cells within dough throughout mixing at 1 bara for the first 110 s and 0.5 bara thereafter, and throughout a constant pressure of 1 bara for 230 s. ........................................................................................................159
Figure 6.13 Cumulative voidage for different cell volumes within dough throughout mixing at 1 bara for the first 110 s and 0.5 bara thereafter, and the control, which was mixed at a constant pressure of 1 bara........................................................................................................160
Figure 6.14 Number of cells per unit volume at different mixing times, with X-ray CT results and fitting of the no breakup population balance model of aeration to the experimental results. Dough was either mixed at a constant pressure of 1 bara or pressure-vacuum mixed for a total of 230 s, with a pressure step decrease from 1 bara to 0.5 bara at 110 s. .........161
Figure 6.15 Dough voidage outside mixer at different mixing times, with X-ray CT and double cup measurements, and fitting of the no breakup population balance model of aeration to the experimental results. Dough was either mixed at a constant pressure of 1 bara or pressure-vacuum mixed for a total of 230 s, with a pressure step decrease from 1 bara to 0.5 bara at 110 s ........................................................................................................162
Figure 6.16 Mean cell volume at different mixing times, with X-ray CT results and fitting of the no breakup population balance model of aeration to the experimental results. Dough was either mixed at a constant pressure of 1 bara or pressure-vacuum mixed for a total of 230 s, with a pressure step decrease from 1 bara to 0.5 bara at 110 s ........................................................................................................163
Figure 6.17 Dough set up for X-ray imaging during proving .............................................165
Figure 6.18 Change in dough volume with proving time. The dough was mixed in a conventional plenary kitchen mixer and proved at 21.5°C. .................................................168
Figure 6.19 Change in dough voidage with proving time. The dough was mixed in a conventional plenary kitchen mixer and proved at 21.5°C. .................................................169
Figure 6.20 Orthoslice at (a) 0 min (b) 10 min (c) 20 min (d) 30 min (e) 40 min (f) 50 min (g) 60 min (h) 70 min (i) 85 min (j) 90 min (k) 110 min 9l) 145 min, during proving at 21.5°C ...172
Figure 6.21 Two dimensional images of dough slices with some cells highlighted and tracked throughout dough proving from 0-145 minutes at 21.5°C ........................................173
Figure 6.21 Two dimensional images of dough slices with some cells highlighted and tracked throughout dough proving from 0-145 minutes at 21.5°C ........................................174
Figure 6.21 Two dimensional images of dough slices with some cells highlighted and tracked throughout dough proving from 0-145 minutes at 21.5°C .................................................................175
Figure 6.21 Two dimensional images of dough slices with some cells highlighted and tracked throughout dough proving from 0-145 minutes at 21.5°C .................................................................176
Figure 6.21 Two dimensional images of dough slices with some cells highlighted and tracked throughout dough proving from 0-145 minutes at 21.5°C .................................................................177
Figure 6.21 Two dimensional images of dough slices with some cells highlighted and tracked throughout dough proving from 0-145 minutes at 21.5°C .................................................................178
Figure 6.22 Surfacegen images showing the volume of the dough (blue) and the cells within it (red) during proving at 0 minutes, 30 minutes, 60 minutes and 140 minutes from left to right .................................................................................................................................179
Figure 6.23 Number density of cells at different times whilst proving at 21.5°C ............181
Figure 6.24 Mean cell volume at different times whilst proving at 21.5°C .................181
Figure 6.25 Number density of cells per unit volume at different times whilst proving at 21.5°C .................................................................182
Figure 6.26 Cumulative % voidage of different cell volumes at different times whilst proving at 21.5°C .................................................................................................................................182
Figure 6.27(a) Mean Feret shape of cells at different times whilst proving at 21.5°C. The data points are the mean of all cells present at the proving time. Error bars are one standard deviation on either side of the mean. A linear regression line is shown. (b) Feret shape distribution of cells per unit area at different times whilst proving at 21.5°C. The mean cell Feret shape are based on group sizes of 0.5. The volume is the dough volume at the proving time. (c) Mean cell Feret shape distribution of cells at different times whilst proving at 21.5°C. The mean cell Feret shape are based on group sizes of 0.5. The volume is the dough volume at the proving time.(c) Mean cell Feret shape at different times whilst proving at 21.5°C .................................................................184
Nomenclature

Abbreviations
2D Two Dimensional
3D Three Dimensional
4D Four Dimensional
AACC American Association of Cereal Chemists
ANOVA Analysis of Variance
BU Brabender Units
CBP Chorleywood Bread Process
CLSM Confocal Laser Scanning Microscopy
CT Computerised Tomography
DATEM Diacetyl Tartaric Acid Esters of Monoglyceride
ESRF European Synchrotron Radiation Facility
ESEM Environmental Scanning Electron Microscopy
MRI Magnetic Resonance Imaging
NMR Nuclear Magnetic Resonance
RMSE Root Mean Square Error
RPM Revolutions Per Minute
SSL Sodium Stearoyl-2-Lactylate
SEM Scanning Electron Microscopy
TA Texture Analyser

Notation

$C_d$ Probability of a cell being disentrained during a disentrainment event
$d$ Cell diameter (m)
$n$ Number of mol of gas (kg mol)
$N_{in}$ Number of cells entrained per unit volume of dough per revolution
$P$ Pressure
$P_1$ Extra mixing pressure
$P_2$ First mixer headspace pressure (bara)
$P_2$ Second mixer headspace pressure (bara)
$P_b$ Pressure inside cell (N m$^{-2}$)
$P_∞$ Pressure outside cell (N m$^{-2}$)
$r$ Cell radius (m)
$R$ Universal gas constant (J mol$^{-1}$ K$^{-1}$)
$T'$ Absolute temperature (K)
$V$ Volume
$V'$ Compressed volume
$V_{in}$ Standard entrained cell volume (mm$^3$)
$W$ Mass (g)
$We$ Weber number
<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\sigma$</td>
<td>Surface tension (N m$^{-1}$)</td>
</tr>
<tr>
<td>$\rho$</td>
<td>Density (g cm$^{-3}$)</td>
</tr>
<tr>
<td>$\rho_{gf}$</td>
<td>Gas free dough density (g cm$^{-3}$)</td>
</tr>
<tr>
<td>$\tau$</td>
<td>Shear stress (N m$^{-2}$)</td>
</tr>
<tr>
<td>$\Phi$</td>
<td>Voidage</td>
</tr>
<tr>
<td>$\Phi_{\text{step}+}$</td>
<td>Voidage immediately after the step change</td>
</tr>
<tr>
<td>$\omega_d$</td>
<td>Disentrainment frequency per unit volume</td>
</tr>
<tr>
<td>$\zeta$</td>
<td>Number of mixer revolutions</td>
</tr>
<tr>
<td>$\zeta_0$</td>
<td>Number of mixer revolutions at initial state</td>
</tr>
</tbody>
</table>
Abstract

Gas cells make up a significant proportion of bread’s volume and are responsible for a number of bread’s characteristics, making their size distribution throughout bread an important quality parameter. The number and size of cells affect the texture and volume of bread, the quantity of sauce mopped up, and how bright the bread appears. Gas cells are incorporated into bread dough during mixing and manipulated throughout the breadmaking process to obtain the desired cellular structure. Due to the fragile nature of bread dough, obtaining accurate quantitative data on its cellular structure is challenging. This thesis investigates the cellular structure of bread, as well as assessing the effect of sugar during breadmaking. Magnetic resonance imaging (MRI), microscopy and X-ray computerised tomography (X-ray CT) have been used throughout research in bread dough to visualise dough’s cellular structure. A non-destructive and non-invasive method giving a high resolution is X-ray CT, in particular when using a synchrotron light source. However, time on a synchrotron beamline is highly competitive, and can require applications more than two years in advance. Running costs of experiments from a synchrotron beamline are also high. This thesis details an alternative X-ray set-up to accurately visualise dough’s cellular structure using a conventional and therefore more easily accessible X-ray source.

Three X-ray CT experiments were conducted to investigate dough’s cellular structure throughout mixing, during proving and in different sugar content doughs. The resolution of the scans varied from 7-11 µm.

Industrial bread dough mixing is often conducted at a high pressure initially to improve oxygen availability, followed by a period of partial vacuum to favourably manipulate the cell size distribution. Using X-ray CT, dough cell size distribution was measured at different points throughout pressure-vacuum and constant pressure mixing. A simplified population balance model was fitted to the measured cell size distributions and the validity of the assumptions within the simplified model explored. It was shown that the dynamic changes in the cell size distribution within bread dough could be accurately measured during pressure step change mixing with a non-synchrotron X-ray source. Pressure-vacuum mixing was shown to give a finer cell distribution than constant pressure mixing and the observed decrease in cell number density was found to be much more short lived than the decrease in cell size. The model was found to provide a reasonably accurate characterisation of pressure-vacuum mixing.

X-ray CT was also used to monitor dough’s changing cellular structure during proving by taking scans every 5 minutes over 145 minutes. Dough voidage increased from 3% to 66%, resulting in a volume increase from 544 mm$^3$ to 1293 mm$^3$. Cell growth was quickest between 40 and 140 minutes, where a steady increase in volume and significant changes in the cell structure occurred. A change in voidage distribution was observed, with greater proportions of gas located in larger cells over time. In addition, over the course of proving cell numbers dropped, a 156-fold increase in mean cell volume occurred, and mean cell Feret shape increased from 1.59 to 1.91. This in-situ method of X-ray imaging of bread dough provides
higher resolution images than comparable data from conventional X-ray sources. In addition, the method has proved to be effective in obtaining high resolution and high contrast 3D images of the cellular structure of dough. This technique will help those wanting to investigate cellular changes in the dough dynamically, but without the waiting time and applications that are required with synchrotron X-rays.

On investigating the effect of sugar during breadmaking, sugar was found to increase the gas free dough density and dough voidage, change the dough's rheology, increase its proving time and produce denser bread. Application of a population balance model on the experimental results indicate that the decrease in steady state voidage as the sugar content increases is a result of an increase in disentainment. This was reflected in the X-ray CT of sugared vs. non-sugared doughs through fewer and smaller cells present in sugared doughs. This is likely to be a result of a weaker dough structure, making cell rupture more likely. The Chorleywood Bread Process (CBP) is used industrially worldwide for the production of bread in less time and using inferior ingredients compared to the traditional bulk fermentation process, making it more cost effective. These results show that simply extending the pressure vacuum mixing used for the production of standard bread loaves in the CBP to sugared doughs should be avoided as aeration of sugared doughs differs to non-sugared doughs. The results suggest that to do so would be detrimental to the product quality.
Declaration

No portion of the work referred to in the thesis has been submitted in support of an application for another degree or qualification of this or any other university or other institute of learning.

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Staff within Manchester University that have not been directly involved in my work have also provided support which I am especially grateful for. I appreciate Patrick Hill’s generosity in allowing me to use the electron microscope and his time and assistance in running the experiments. The workshop staff were always so quick to respond to problems I had with equipment, and never failed to get to the heart of the problem.

During the first year of my PhD I ran some experiments at the Hollings Campus at Manchester Metropolitan University, providing me with essential water absorption data for my flours, and an opportunity to practise experimental techniques. Weili Li and the technical staff were very supportive, and I’d like to express my gratitude towards them.

My friends in chemical engineering have been very supportive throughout my studies, and it’s reassuring to know when we go through tough times that this is typical of all PhD students! Playing bridge has been a nice way to get away from it all. Thank you!

Thank you also to all my friends outside of university who have provided emotional support and listened to me vent my frustration. Andrew Birkin, in particular, has put up with a lot of this, and supported me unconditionally. Thanks! I’d also like to thank my family for their unconditional support in my studies and career.

Last but not least, I would like to express my gratitude towards the EPSRC. This project would not have been possible without their financial support.
Chapter 1 - Introduction: Understanding bread dough aeration

Bread is a versatile and filling food, and is popular worldwide. 99% of UK households buy bread and the equivalent of almost 12 million loaves are sold daily (Anon, 2012a). 80% of UK bread is produced in large plant bakeries, 17% in in-store bakeries and the remaining 3% in craft bakeries (Anon, 2012a). The main players in the plant bakeries are Allied Bakeries, Hovis and Warburtons. However, the competition to offer cheaper products than own brand bakeries has resulted in the closure of two Hovis bakeries in 2013. Plant bakeries have been advised to cut costs and offer a point of difference in their products if they are to compete against cheaper own brand bakeries (Ruddick, 2012). Bakers are aware of the issue, as reflected by the point of difference products with value adding attributes on the market, some of which are illustrated in Figure 1.1. Examples of point of difference products include (1) white loaves made with 50% wholegrain flour. These offer the desired taste and texture of white bread with added nutritional benefits. (2) Crustless loaves. This is a convenient product for consumers (often children) who dislike bread crusts. (3) Loaves made from 100% British wheat. This appeals to consumers wanting to live more sustainably and support the British industry.

Figure 1.1 A selection of some point of difference UK bread products. Products illustrated include Kingsmill 50:50, for those who want the goodness of wholegrain without its texture, Hovis British Farmers Loaf, a loaf made with 100% British wheat, Hovis seed sensation, containing seven seed varieties, Kingsmill Little Big Loaf, full sized bread slices but in a smaller pack size and Warburtons Sandwich thins, who want less bread in their sandwiches.
The UK bakery market is worth almost £3.46 billion with bakery snacks and world breads being the driving growth (Anon, 2012a). Two of the factors driving the trends in bakery include healthy eating concerns and indulgence (Anon, 2012a). The concerns over healthy eating are balanced by the desire for pleasure seeking indulgent products. Pleasure seeking products include continental breakfast products, bakery snacks and international products. Continental breakfast products are categorised in ‘bakery snacks’, the second largest bread category, only behind ‘bread’ (Anon, 2012a). There are more than 30 sub categories within bakery snacks. Some of these are illustrated in Figure 1.2. Research has shown that indulgence is desirable in bakery snacks to boost the enjoyment factor of the weekend after a week focused with healthy intentions (Anon, 2012a).

Figure 1.2 A selection of bakery snacks available in UK supermarkets. Illustrated include traditionally seasonal products such as hot cross buns, products released for a specific event, such as the red nose day doughnuts, world breads such as croissants and brioche, and traditional UK bakery snacks such as scones and pancakes to name a few.

Breadmaking processes vary worldwide. The process may involve mechanical dough development, a long fermentation, the addition of an improver to develop the dough in less time or the production of a fermenting ‘sponge’ to which other ingredients are later added. The Chorleywood Bread Process (CBP) is a mechanical dough development process. It originated in the UK as a solution to producing acceptable quality bread using UK flours, which have inferior breadmaking qualities compared to imported flours. In the UK, large plant bakeries and many in-store bakeries produce bread using the CBP. It had also been adopted in
a number of countries worldwide. One reason for the widespread uptake of the CBP is that flour can be processed into a loaf of bread, wrapped, sliced and be ready for sale all within three and a half hours. This is largely due to the lack of a lengthy bulk fermentation stage. This lack of fermentation also saves money, as storage space under controlled conditions is not required. Cost savings are also made by using cheaper lower protein wheat. The CBP also creates a light and soft bread of consistent quality. Disadvantages of the CBP include the addition of extra ingredients to create bread of acceptable quality, a lack of flavour, and a change in texture to traditionally made breads.

To achieve bread of saleable quality, it is important that the number and distribution of cells present in the crumb is optimum for the characteristics expected of the bread. These cells originate from gas pockets entrained during mixing and are manipulated throughout the breadmaking process to achieve bread of desirable characteristics. A high volumned loaf with a fine crumb structure is a desirable characteristic. As bread is sold by weight, a larger volume is indicative of more bread and a light texture. Another quality characteristic is bright bread, in particular in white breads. The orientation of cells revealed on a slice affect how much light is reflected from them and thus how bright the bread appears. Soft bread is another key quality characteristic that is perceived by consumers as indicative of fresh bread. These quality parameters are largely determined by the cell distribution in bread, and affect its eating qualities, from how “heavy” the bread is, to how well it slices and how well it mops up a sauce.

This thesis sets out to better understand the breadmaking process as a series of aeration stages. Within this aim exists a further aim of developing a method of accurately obtaining the cell size distribution in bread dough at different stages of breadmaking, with the intention of using the results to validate the assumptions made in an existing published population balance model on aeration during mixing. In addition, it was mentioned previously that the two main driving forces in bakery snacks are the indulgence factor and healthy eating. Low sugar snacks are often perceived as healthy whilst high sugar snacks are viewed as indulgent. This thesis assesses the effect of sugar in bread dough to better understand its effects on bread’s structure. This information is useful for developing new bakery products.

Searches through scientific databases to investigate the research conducted on bread dough aeration found a lack of research on the effect of sugar on aeration in breadmaking. However, a glance at supermarket bakery aisles reveals that sweetened bread products make up a large portion of the UK bread market. Examples of these products include fruit loaf, brioche, doughnuts, Chelsea buns and panettone. The breads are sweetened with some sugar, and usually either or both dried fruit and icing, or like doughnuts, filled with a sweet paste, such as jam or custard. Aside from the textural and flavour pleasures in consuming dried fruit, icing and jam, the additional sweetness they give the product is likely to be required due to high quantities of sugar in dough making it sticky. A sticky dough is likely to be dense due to its cohesiveness, and difficult to process due to its adhesiveness. Assessment of the percentage to flour (Baker’s percentage) of sugar present in sweetened breads in UK
supermarkets found the majority to contain less than 5% sugar. Brioche was found to contain 13% sugar based on flour weight, but a number of other ingredients were also present, such as milk, butter and eggs, such that as a percentage of all the ingredients present the sugar content was considerably less. Understanding how sugar contributes to the creation of aerated structures in these products may help reduce the sugar content while retaining the desirable characteristics of these products, and can also help in the production of more indulgent breads containing increased quantities of sugar. This thesis reports on a study into the interrelationship of bread dough aeration with added sugar. As dough aeration and rheology are interdependent (Campbell, and Martin; 2012; Chin, 2009), and small changes in cell concentration can affect dough rheology (Chin et al., 2005; Scanlon et al., 2008), sugar’s effect on dough rheology was also investigated. Rheology is the study of how a material flows and can be used to characterise and predict dough’s behaviour at different stages of breadmaking (Dobraszczyk et al., 2001; Campbell and Martin, 2012). Studying dough rheology is also important as sensory detection of texture is dependent upon its rheological properties (Daubert and Foegeding, 2003), which changes throughout breadmaking (Dobraszyck et al., 2001).

Dough aeration is measured in terms of dough voidage and the distribution of gas within the dough (Campbell, 1991). The latter is more challenging due to the requirement of a non-destructive method. Gas cells in bread are important as they make up a large proportion of bread’s volume and affect bread’s brightness and also its mechanical properties on consumption, and therefore the hedonic response to the bread. They are worth studying as different cell size distributions mean different bread properties and therefore determine the quality and success of the product. By understanding the link between the cell size distribution and properties of bread, this can help improve existing product and developing new products. In this thesis, both voidage and cell size distribution are measured using X-ray CT. This non-invasive method alongside visualisation analysis software enabled quantitative information on the 3D cellular structure of dough to be derived. Using X-ray CT, the 3D sample volume was reconstructed, giving voxel dimensions as low as 7 µm. The method allowed comparison of cell volume distribution during pressure-vacuum mixing to the cell volume distribution at constant pressure mixing. This gave insight into the effect of pressure-vacuum mixing on the cell volume distribution and if the observations agree with the theory of how pressure-vacuum mixing works, as well as improving the accuracy of a population balance model for aeration during mixing. The in situ X-ray scans during proving allowed observation of the behaviour of individual cells, cell mechanics and change in cell volume and shape distribution. These are a few of the parameters which can be obtained via X-ray CT using conventional X-ray equipment.

The scope of this thesis is as follows: Chapter 2 offers a background to this thesis. It details the history and significance of bread, and the CBP. As aeration is key to bread’s quality, the aeration process is explained and methods that have been used to measure it are discussed. Chapter 3 provides a background to the experiments conducted. It describes the materials
used and explains why they were chosen, describes the equipment used and details the analytical techniques. The flours and doughs used in later experimental chapters are also characterised here. Chapter 4 presents a range of tests in ten short experimental sections to investigate the effect of sugar during the breadmaking process. These include assessing the dough voidage and gas free dough density, its microstructure, specific mixing parameters and some rheological parameters. In Chapter 5 a population balance model is utilised to compare the aeration of varying sugar content bread doughs in terms of entrainment and disentainment during mixing. Different size and direction step changes are investigated for three different sugar content doughs to determine why the voidage varies within these doughs. Chapter 6 continues to investigate sugared doughs through X-ray CT. Chapter 6 consists of three experimental sections: in the first a comparison of the cellular structure of a sugar containing and non-sugar containing dough is made. Next, the cellular structure of dough is investigated throughout pressure-vacuum mixing, and the results used to assess the validity of a population balance model. The work of this Section has been published in the journal Chemical Engineering Science and can be found in the appendices. Finally, on further development of the X-ray CT technique, it was then used to investigate the cellular structure of bread dough in situ during proving. The voidage distribution and cell distribution were obtained throughout proving and the change in cell shape was quantified. Finally, Chapter 7 concludes this thesis and makes recommendations for how research in this area should be taken forward.
Chapter 2 - Bread: Its history, significance and manufacture

The introduction outlined the purpose of this thesis, to report novel findings of the interrelationship between aeration processes in bread manufacture and the role of added sugar in sweetened breads. This chapter presents an in depth review of the relevant literature across the scope. The review starts in Section 2.1 presenting an overview of bread in its historical, social and technological context. Section 2.2 details the aeration mechanisms, the factors affecting them and the different methods that can be used to measure aeration throughout the breadmaking process.

2.1 An introduction to bread

This section is an overview of breadmaking and is split into three sections. The first gives a history of bread and details its significance historically and today. Following this, in Section 2.1.2, the breadmaking process is described with emphasis on the industrial CBP. Finally, in Section 2.1.3, common breadmaking ingredients are listed and their purpose described.

2.1.1 The significance of bread

The importance of bread is reflected by its use as currency in the Pharaonic period in Egypt, and the use of the word “bread” as an equivalent to food by Christians who recite the Lord’s prayer, asking for their “daily bread”. Bread is made from grain and water, and cooked into a solid mass. There are records of bread consumption from the Neolithic period. During the Neolithic period, unleavened breads were made from a mixture of cracked grains and water. Over time a preference developed to pound the grains, until eventually unleavened bread was made from ground grains and water. Leavened bread is believed to have come about after the Egyptians forgot about a batch of dough. This resulted in it becoming inoculated with yeast from the air. The dough was cooked and a more flavoursome, lighter bread was discovered. Despite the inconvenience of making leavened bread and the reduced product mass due to yeast metabolism, the art of making leavened bread quickly spread worldwide, a reflection of its popularity. The popularity of bread has been long standing, although preferences vary worldwide and amongst individuals.

Bread has played a role in survival for Vikings, warriors and explorers under difficult conditions (Sonoma, 2008). Cracker-like breads are low in moisture making them light and compact, thus allowing them to be taken on long journeys without becoming mouldy. Aside from being a form of nourishment, bread has a religious significance. On Good Friday Christians traditionally hang a loaf of bread in the house to ward off evil (Davidson, 2006), and the consumption of hot cross buns is associated with Easter. The cutting of the cross in the bun was believed to expel the devil (Ingram and Shapter, 1999). These actions are still carried out today although due to tradition rather than beliefs. Nowadays, tradition also means ‘luxury’ breads are consumed at special occasions. These include stollen in Germany for Christmas, hot cross buns in England for Easter, challah for the Jewish Sabbath and pan de
mureto for day of the dead in Mexico. The need for bread on special occasions emphasises the significance of bread in society. Bread is not just consumed on special occasions, however. It is also a staple and viewed as a necessary everyday food, as reflected by the amalgamation of ordinary people and politicians to form the Anti-Corn Law League in 1838 in protest against rising corn prices which caused bread prices to rise (Anon, 2010a).

During Medieval times in Britain, the noble employed workers with the sole task of baking. It was not just nobility that enjoyed bread, however. Bread was enjoyed across all classes and the type of bread consumed was indicative of status. Nobility consumed white bread, tradesman consumed wholemeal bread, and the poor consumed bran bread (Anon, 2012b). These breads not only varied in appearance but also in firmness, with the noble consuming the softest and whitest bread. Even during the Second World War the realisation of the nutritional benefits of wholemeal bread over white did not sway the consumer preference towards wholemeal bread. It wasn’t until 1984 when ascorbic acid was added to wholemeal bread resulting in a softer texture that its popularity began to increase. For many years, white bread was perceived as a superior product, and despite the ban of toxic whitening substances, bakers continued to add these to their bread. This and the time taken for wholemeal bread to become popular illustrates the consumer preference of appearance and texture over health benefits. With this in mind, in 1956 laws were introduced, whereby the use of all flour for breadmaking other than wholemeal had to be fortified with thiamine, calcium, iron and nicotinic acid.

Nowadays, the importance of bread is reflected by its consumption worldwide. Not only is it an affordable form of nourishment for most but it is also a good source of carbohydrates. The Department of Health recommends a third of the calorific intake is derived from starchy carbohydrates such as bread, pasta, potatoes and rice. Depending on the country, the quantity and variety of bread consumed varies. In oriental countries, rice makes up a large portion of the carbohydrate intake. However, traditional steamed bread and the growing in popularity western breads are also consumed. In the Middle East, unleavened breads are the bread of choice. In the UK, bread makes up a large proportion of the starch consumed, and the preference is white bread which makes up 76% of the bread sold with 50% of bread being sold as sandwiches (Anon, 2012d). Bread has always been a popular product in the UK. The Great War was the beginnings of female employment. This meant women had less time to make bread, increasing the importance of industrially made bread. It became essential that bread could be made in less time to meet the increasing demands. In the 1960s the lengthy traditional bulk fermentation process was abandoned in preference for the CBP. The CBP is a mechanical dough development process with a substantially lower bread production time than the bulk fermentation process.

Industrially made bread has several advantages over homemade bread, including convenience, consistency, a longer shelf life, and a regular shape. However, the flavour of industrially made bread and quality of ingredients is often inferior to homemade bread (Blythman, 2007). The rising popularity of cookery shows and in particular the baking series,
The Great British Bake-Off, has increased the popularity of baking in the UK (Jones, 2012). This has contributed towards an improved general knowledge of baking and in particular awareness of the small number of essential ingredients required to produce bread, compared to that used in industry. This awareness is likely to be responsible for the increased popularity of artisan bakeries, which produce bread consumers perceive as superior in flavour and texture compared to industrially produced bread. The growing popularity of artisan bread reflects the need to tighten the gap between artisan breads and industrially produced breads.

2.1.2 Industrial breadmaking - the Chorleywood Bread Process

Breadmaking is a series of aeration stages (Campbell et al., 1998), where each stage involves the manipulation retention of the gas cells in a suitable form until the product is baked (Grenier et al., 2003) to obtain the desired characteristics. There are three main stages in breadmaking: mixing, proving and baking. These essential breadmaking stages vary from one product to another.

To begin with, the weighed ingredients, usually flour, water, yeast, salt, vegetable fat and dough improver, are brought together into a homogenous dough through shearing actions. During mixing, the dried ingredients become hydrated. For flours produced from a gluten containing grain, hydration of the protein is particularly important for development of the viscoelastic properties of the dough. Mixing also inputs heat and mechanical energy into the dough, through stretching and pulling actions. This helps develop the dough, and form a gluten network within the dough. When the dough is developed, it becomes a viscoelastic material, capable of holding expanding cells.

As the dough is mixed, it is folded over itself, trapping pockets of gas between the layers of the dough. This is the beginning of the gas cells in bread. The cells in bread can make up 80% of its volume and are essential in determining the characteristics of bread. Mixing is the stage of breadmaking where bakers have the most control over the cells in bread. Different mixing actions, mixer blades, mixing time, mixing speed, mixing pressure and the scale of the equipment can be varied to achieve a different cell volume distribution. As new cells can only be created from these existing cells, mixing becomes a key stage of breadmaking. It is believed that 90% of the final bread quality depends upon mixing (Cauvain, 2000). This is also due to mixing being required to develop the dough in mechanical dough development processes such as the CBP.

The second stage of the breadmaking process is known as proving or fermentation. During proving, the dough is left in a warm and humid environment to increase in volume. In order to achieve a high loaf volume and fine crumb structure, coalescence should be retarded. During proving the increase in volume is a result of yeast carbon dioxide production. Yeast metabolise the sugar in the dough and produce carbon dioxide as a waste product via an enzymic reaction. The carbon dioxide diffuses into the cells and causes them to expand. The optimum temperature for yeast metabolism is 35-40°C (Cauvain, 2012) with higher temperatures reducing proving times. Gujral and Singh (1999) found that proving
temperatures of up to 50°C are used for industrial breadmaking. The humidity during proving is also an important parameter. A relative humidity of 85% is used to prevent the surface of the dough from drying out. Depending on the desired bread characteristics, the proving time varies. Generally, a shorter proving time results in a finer crumb and a longer proving time results in a coarser crumb (Zghal et al., 1999) due to the greater degree of coalescence which occurs on extending the proving time.

During the final stage of breadmaking, the dough is subjected to heat. Depending on the bread being made, the cooking method will vary. For example, mantou are steamed, chapattis are heated over a skillet, puris are fried and focaccias are baked. Baking is the most common method of cooking bread. The heat transforms the dough into an edible product. The high temperatures cause water to evaporate from the product, resulting in loss of weight from the product. Evaporation also occurs from the product surface, forming a dry, firm and crispy crust. The heat deepens the colour of the product into a golden brown colour, develops flavour and aroma through Maillard reactions (non-enzymic reactions occurring in food when reducing sugars react with proteins, peptides, amino acids or amines) and changes the texture of the product through starch gelatinization and protein coagulation.

During baking, the product is subjected to temperatures of 200-230°C. Heat is transferred to the dough via conduction and convection. The initial increase in temperature, compared to proving temperatures, results in an increase in yeast carbon dioxide production, up to temperatures of 55°C (Zhang et al., 2007). This causes a final expansion of the dough volume by one third (Dobraszczyk et al., 2003) through an increase in cell volume. This activity is termed oven spring. It occurs until the crust sets and resists expansion of the unbaked portion of the dough. Despite the resistance created by the crust setting, the cells within the unbaked portion of the loaf continue to expand (Zhang et al., 2007) leading to coalescence, which gives bread its interconnected cell structure. Local expansion eventually ceases as a result of starch gelatinization increasing dough viscosity and decreasing dough extensibility.

Although bread can be made in a number of ways, industrially there are two main methods of making bread: the bulk fermentation process and the CBP. The bulk fermentation process is the more time consuming of the two. The dough is mixed at a low speed for approximately 10 minutes. Following this, it undergoes the first of its three proving sessions. The first session lasts 2-4 hours with mixing part way to subdivide and redistribute the cells, helping achieve a finer crumb structure in the final loaf. The dough surfaces are then smoothed down and the dough divided into pieces of the desired weight to undergo intermediate proof. Finally, the dough is shaped and undergoes its final proof, before being baked. The key feature of the bulk fermentation process is the long proving times. Compared to the short proving session in the CBP, the bulk fermentation process is beneficial for flavour development and protein structure, enabling better retention of gas and retardation of coalescence during proving.

Despite the textural and flavour benefits of the bulk fermentation process, the CBP is generally used in preference. Advantages of the CBP include a higher yield through less
proving and the addition of more water, and cost savings through being able to use lower quality wheat and less space and time needed, a result of the lack of proving. In the UK, approximately 80% of the flour is derived from lower protein British wheats (Anon, 2012c), saving money through lack of imports and the use of a cheaper, lower quality flour (the most substantial ingredient in terms of quantity). However, to enable processing, the use of these wheats must be balanced through the addition of extra water and yeast, and unpopular additives.

Mixing in the CBP occurs at a high speed, imparting 42 kJ kg\(^{-1}\) of energy on the dough in less than 3 minutes. Through the rapid input of energy into the dough and optimisation of the cell distribution, the need for lengthy proving is eliminated. The CBP also uses pressure-vacuum mixing to ensure a fine cell distribution within the dough. The dough is first mixed at a positive pressure to increase oxygen availability to the dough. This facilitates the action of the dough improver, ascorbic acid, which helps develop the dough and produce a whiter loaf. A partial vacuum is then drawn towards the end of mixing. This removes excess air from the dough and expands the existing cells, allowing the subdivision of cells caused by further mixing to result in a fine cell distribution.

2.1.3 Breadmaking ingredients
As mentioned in Section 2.1.1, the number of ingredients required to produce bread is small. The minimal ingredients for leavened bread are flour, water, yeast and salt. Industrial bread often contains more than a dozen ingredients, however. These ingredients are added to ensure the efficient processing of the ingredients into a desirable product. Depending on the product being made, different characteristics are sought and thus the ingredients and methodology vary. However, there are ingredients common to industrially produced breads. In this section, breadmaking ingredients are listed and their purpose in the formulation described.

Flour
Although a variety of cereal grains can be used in baking, wheat flour is most commonly used as it is the only cereal capable of delivering a highly aerated structure in the baked loaf (Campbell and Herrero-Sanchez, 2001). This is due to the unique viscoelastic properties of doughs produced from wheat flour, allowing growth and retention of carbon dioxide in the dough without cell rupture. These properties arise from the wheat storage protein gluten which comprises mainly a mixture of water insoluble glutenin and gliadin. The quantity of gluten and ratio of glutenin to gliadin affects the breadmaking quality of wheat flour, dramatically changing the dough rheology (Xu et al., 2007). Glutenin is responsible for the strength and elasticity of the dough, and therefore cell retention. Gliadin is responsible for the extensibility and viscosity of the dough, and therefore cell expansion. The ratio of glutenin to gliadin is important for the dough’s viscoelasticity. In wheat flour this ratio is approximately 1:1. In other cereal grains the ratio differs, and less gluten is present, making bread from these grains less desirable and the production of highly aerated loaves difficult. Cauvain and Young (2006) noted that protein quality is not as essential in the CBP compared
to other breadmaking processes, due to the high level of oxidants used and the short processing time. This enables use of lower quality European wheats containing approximately 3% less protein than the hard winter wheats of North America and Canada. The quality of bread produced from lower quality wheats can also be boosted by adding vital wheat gluten. Flours containing a higher proportion of protein have improved viscoelastic properties and produce breads of a larger volume and better quality (Cauvain and Young, 2006), making them desirable for breadmaking. A protein content of 11% or more is considered high and the flour termed strong. Strong flours are reputed to absorb more water, give less aeration and produce doughs of a lower gas free dough density (Campbell et al., 1993; Campbell et al., 2001; Dobraszczyk et al., 2001; Chiotellis and Campbell, 2003b; Chin, 2003; Sroan and MacRitchie, 2008). An investigation of 74 flours into the relationship between protein content and water absorption, presented in Figure 3.8, does not illustrate a relationship between these two parameters, however. This is likely to be due to the small number of flours in relation to the number of flours that exist, and the other parameters affecting water absorption, discussed later in this thesis.

The degree of enzyme activity in the flour is important since the flour starches need to be broken down into sugars through yeast enzymic action to provide a sugar the yeast can metabolise. Too much enzyme activity results in too much sugar and not enough starch present, giving the final bread a poor texture, and too little results in low carbon dioxide production, giving a loaf of low volume.

Flour also contains 1-1.5% lipids. The lipids present and their quantity varies across different flours. These surface active components will differ in polarity and saturation. In the quantities present they do not affect dough rheology (Sroan and MacRitchie, 2009), but do affect the volume and crumb structure (MacRitchie and Gras, 1973; Sroan and MacRitchie, 2008) by affecting the stability of the liquid lamellae around the gas cells. Sroan and MacRitchie (2008) noted that the stability of the liquid lamellae is one of two factors that determine the degree of expansion of the dough through controlling disproportionation and coalescence. The other factor is the dough’s rheology. However, despite the occurrence of disproportionation being widely accepted, no evidence has been published proving it takes place.

Water

Water binds the dry ingredients together to form a dough. It is important that the optimal quantity of water is used to ensure the ingredients are properly bound and optimally dispersed. The CBP requires more water than other breadmaking processes due to the partial vacuum mixing drying out the dough (Cauvain and Young, 2007), and a softer dough being necessary for processing in the machinery (Cauvain and Young, 2006). The optimal water content for breadmaking varies from one flour to the next, and can be determined using a Farinograph. Factors believed to affect the water absorption of the flour include the quantity of damaged starch present, and the flour’s protein, moisture, pentosan and bran content (Cauvain and Young, 2006). Using 2% less water than the optimum results in reduced carbon
dioxide production in the dough during proving, producing a loaf of lower volume (Rasanen et al., 1997). Findings from Chin et al. (2005) agree with this. It is not just the carbon dioxide production that is affected by insufficient water but also retention of carbon dioxide (Peighambardoust et al., 2010) due to inadequate gluten hydration.

On the other hand, an excessive level of water results in a more extensible and very sticky dough (Spies, 1997), causing processing difficulties.

Although the quantity of water added to bread dough varies, depending on the flour used, dough formulation and processing method, 60% of the flour weight is the general proportion added. However, it is not just the water content in foods that affect its properties but also the water activity, $a_w$, which depends upon the form in which the water exists in the product, and is determined by the ingredient formulation. Water in food exists in three general forms: (1) free, unbound water, (2) free, immobilised water, and (3) chemically bound water.

Water activity is an important parameter for determining moisture movement within the dough and can affect microbial growth, enzyme activity, and chemical activity within the dough (Pennington and Baker, 1990). It is a significant parameter in the investigation of the effect of sugar in bread dough. By definition, water activity is the vapour pressure of water in the product divided by the vapour pressure of pure water at the same temperature. Water activity ranges from 0 to 1 and can also be referred to as the amount of free water in the product or the amount of water not bound to food molecules. Bread dough and bread have a water activity of approximately 0.95 (Czuchajowska et al., 1989; Dairy Research and Information Centre, 2013). On investigating the effect of sugar on the water activity of bread Czuchajowska et al. (2004) found this figure decreases on addition of sugar. Sugar has a hygroscopic nature (Tlapale-Valdivia et al., 2010) and is known to cause a significant reduction in the water activity of food products, due its solubility and relatively small molecular size (Pennington and Baker, 1990).

**Yeast**

Yeast are microorganisms. They require warmth, moisture and food to reproduce. In the CBP, the yeast species, *Saccharomyces cerevisiae*, is commonly used. There are an unlimited number of *Saccharomyces cerevisiae strains*, and several thousand varieties are used in baking (Anon, 1996a). These strains differ in sugar tolerance, growth rate and sensitivity to different chemicals. Manufacturers will select the yeast strain or combination of yeast strains based on the suitability to the breadmaking process and product being made. Yeast used in the British bread industry is typically fast acting because of the short timescale of the CBP.

*Saccharomyces cerevisiae* are facultative anaerobes. Facultative anaerobes can metabolise both aerobically and anaerobically, favouring aerobic metabolism if sufficient quantities of oxygen are present. In the CBP, *Saccharomyces cerevisiae* is added at 2.5% of the flour weight. This is greater than the quantity which would be added in traditional breadmaking and is necessary to ensure adequate carbon dioxide production due to the lack of proving in the CBP.
It is well known that yeast produce carbon dioxide which causes the cells in bread dough to expand. The carbon dioxide is produced as yeast ferment glucose. Unless sugar is added, glucose is entirely derived from flour. Wheat flour contains approximately 1% of fermentable carbohydrates which are readily available for yeast fermentation. The remainder is derived from amylase action on the flour starches.

Yeast production of carbon dioxide occurs in several stages. Equation (1) is the net chemical reaction of yeast fermentation of glucose,

\[
\text{C}_6\text{H}_{12}\text{O}_6 \rightarrow 2\text{C}_2\text{H}_5\text{OH} + 2\text{CO}_2
\]

where \(\text{C}_6\text{H}_{12}\text{O}_6\) is glucose, \(\text{C}_2\text{H}_5\text{OH}\) is ethanol and \(\text{CO}_2\) is carbon dioxide. Ethanol and carbon dioxide gas are the main products of yeast metabolism. In addition to these, yeast also produce flavour and aroma compounds, and hydrogen peroxide, which results in a more elastic dough (Mirsaeerghazi \textit{et al.}, 2008).

The concentration of other ingredients in the dough can inhibit yeast carbon dioxide production. Too much sugar, salt, alcohol or fat reduces the rate at which the bread rises (Gujral and Singh, 1999; Chin \textit{et al.}, 2005; Spaull and Bruce-Gardyne, 2003; McGee, 2004). The authors recommend increasing the quantity of yeast present if high quantities of the above ingredients are present.

Yeast is available in different forms for baking. These include compressed, dried, creamed, and liquid form. In industrial breadmaking, liquid or creamed yeast are commonly used due to ease of pumping.

**Salt**

Salt serves a number of functions in bread. It tightens the gluten network and increases dough stability through allowing hydrophobic and hydrophilic interactions to occur between gluten molecules (Belitz, 2007). Salt flavours the bread, reduces the action of proteolytic enzymes which otherwise damage the gluten (Davidson, 2006) and increases the bread’s volume (McGee, 2004; Cauvain, 2000). Salt is normally added at the start of mixing, and it is important that the particle size is small enough to completely dissolve during mixing. However, some bakers choose to delay the addition of salt, resulting in more extensible dough through a greater degree of protein hydration (Cauvain, 2000).

**Vegetable fat**

Fat is added to bread via two routes. The first is as a component of flour, where fat is typically present at 1-2%, and the second is as an ingredient itself. Vegetable fat is used over animal fat, allowing vegans to consume the product. Vegetable fat consists of a mixture of different triacylglycerols of which the quantity and combination are responsible for its physical properties.

Typically, vegetable fat is added in quantities of up to 5% of the flour weight, with more fat required when using wholemeal flours (Cauvain and Young, 2007). The addition of fat has
numerous benefits in bread. The first being it delays staling by coating starch granules and postponing moisture release. Secondly, fat lubricates dough, helping it pass more easily through equipment (Anon, 2012c). Thirdly, the crumb colour is improved due to greater light reflection, a result of an increased number of small cells surviving baking. Finally, fat crystals stabilise cells by forming a layer around them with protein (Li et al., 2004). This allows the cells to expand without rupturing and prevents coalescence, through transfer of interfacial material to the cell surface during baking (Brooker, 1996). This enables the production of a high volume bread with a fine crumb (McGee, 2004; Cauvain and Young, 2006). To ensure the last two benefits, it is crucial that 5% of the fat is solid at proving temperature (Dobraszczyk et al., 2001; Gan et al., 1995; Brooker, 1996). The use of lower melting point fats results in the fat crystals melting during proving, reducing the availability for adsorption to cells. In addition, shortenings containing many small fat crystals are superior to those containing larger fat crystals (Brooker, 1996; Cauvain and Young, 2006). They increase the number of small cells that survive baking, and thereby contribute towards the crumb quality. Thus, the crystal structure of the solid proportion of lipids present is an important parameter (Pareyt et al., 2011). Although fat is beneficial for the quality of bread, too much fat stops the formation of the long gluten strands necessary for dough expansion (Davidson, 2006).

**Dough improver**

Dough improvers are chemicals which are added to the traditional dough mix to give bread characteristics which are desirable and otherwise difficult to achieve. Dough improvers consist of surfactants, oxidants and reductants.

Surfactants are molecules which lower the surface tension between a hydrophobic and hydrophilic interface by congregating at the interface. In bread, this may be the gas-water interface or the fat-water interface. This stabilises the cells. Benefits of surfactants in bread include retarding retrogradation, improving loaf fineness and increasing loaf volume, without affecting the gas free dough density (Campbell et al., 2001; Junge et al., 1981). As the surface tension is reduced, the thermodynamic benefit of coalescence is also reduced, thus delaying coalescence (Campbell et al., 2001). Surfactants used in breadmaking include diacetyl tartaric acid esters of monoglyceride (DATEM) and sodium stearoyl-2-lactylate (SSL).

Oxidants are used to improve the gas retention properties in the dough through enhancing gluten reformation (Anon, 1996c). This is done through increasing the number of sulfhydryl links, resulting in increased dough strength and bread of a greater volume. The oxidants used worldwide range and examples include ascorbic acid, potassium bromate, azodicarbonamide and potassium iodate. The oxidant used depends upon what is permitted in each country and the stage of breadmaking for which the oxidising properties are required. In the UK, ascorbic acid is used at a concentration of 75-300ppm. The occurrence of its oxidising action during mixing is optimal, due to competition with yeast for oxygen. Chemically, ascorbic acid is a reducing agent. It requires oxygen and ascorbic acid oxidase to convert into the oxidising agent, dehydroascorbic acid. Thus, the effectiveness of ascorbic acid depends largely on the oxygen incorporated from the air during mixing. Ascorbic acid aids dough development and
increases loaf volume (Belitz et al., 2004; Cauvain, 2000; Anon, 2002), as well as improving the crumb structure and softness (Anon, 2002).

Reducing agents are used to weaken the dough or to change its rheology, so the dough can be moulded without any structural damage. They may also be used when a very high protein flour causes difficulty in fully developing the dough. In reducing the energy required to develop the dough, the final dough temperature can also be reduced. Achieving an optimal dough temperature following mixing is another reason reducing agents may be used. An example of a reducing agent is L-cysteine.

Other dough improvers include acetic acid and lactic acid, which extend the shelf life of bread. However, too much acid can have detrimental effects on the dough, due to the acids reacting with flour proteins and yeast, inhibiting yeast metabolism (Gujral and Singh, 1999).

Sugar
Sucrose is the most common sugar added to bread. Sucrose, also known as saccharose, is usually extracted from sugar beet or sugar cane. It is a disaccharide comprising the two monosaccharides α-D-glucose and α-D-fructose, shown in Figure 2.1.

![Figure 2.1](image)

**Figure 2.1** The molecular structure of the disaccharide, sucrose. Sucrose consists of the two monosaccharides, α-D-glucose and α-D-fructose, linked by a glycosidic bond.

Addition of sugar may be in either the crystalline or liquid form. When added as a solid, the sugar dissolves in the water during mixing. At 25°C 65 g of sucrose dissolves in 100 g water and the solubility increases at higher temperatures (Belitz et al., 2004). Sugar is not essential in bread, although its presence can be beneficial. Benefits include: an attractive brown colour, sweetness, tenderisation and moisture preservation through moisture absorption (McGee, 2004), and acceleration of yeast carbon dioxide production with 1-2% sugar.

Too much sugar, however, has a negative effect on bread. At concentrations greater than 10%, sugar weakens the gluten network; sugar binds with the water, preventing gluten from binding to it (McGee, 2004). If insufficient quantities of water are present, yeast cells may also dehydrate. Yeast activity is also inhibited by high sugar levels. Adding more yeast
(Cauvain and Young, 2006), or allowing the dough more time to rise can solve this problem (Spaull and Bruce-Gardyne, 2003). However, extending the proving time defeats the main advantage of the CBP over the bulk fermentation process.

2.2 Aeration throughout breadmaking

A significant proportion of bread’s volume is gas. As a result, the characteristics of bread are largely determined by its gas cells. Controlling the aeration of bread dough during each stage of breadmaking is therefore important in achieving the desired characteristics in bread. Their volume, orientation and quantity play an important role in the texture of the bread (Campbell et al., 1999) and their eating qualities, such as their ability to mop up a sauce, which is dependent upon surface area. The space they occupy and their influence on the surrounding matrix also affects the crumb structure (Cauvain et al., 1999). During the different breadmaking stages, gas is entrained and disentrained. Retention of entrained gas is important and relies on the cells being stable. Cell stability is determined by their mechanics, which are described in Section 2.2.1. This section also explains how gas is entrained and disentrained in the dough, as well as giving examples of other aerated foods. Section 2.2.2 describes how adjusting the mixing parameters can affect bread dough aeration. In Section 2.2.3 methods of measuring the aeration of bread dough are described. Finally, this section is summarised in Section 2.2.4.

2.2.1 Entrainment and disentrainment of gas

Aeration of foods is a popular process, mainly due to its textural benefits (Campbell and Mougeout, 1999), and also due to the financial benefits of selling the consumer more air for their money. As well as bread, aerated foods include beer, ice cream, chocolate (Aero, Milky Way, Wispa), and soufflés, to name a few. Aerated chocolate can be marketed to the calorie conscious as light, and aerated drinks have the appeal of fizziness.

The method of aeration varies depending on the product. Methods used to aerate food include (1) steam generation, which causes thermal expansion of gas within cells, (2) whipping of low-medium viscosity liquids to traps air, (3) use of chemical raising agents, (4) rapid dry heating to cause surface blistering, and (5) gas injection. Bread dough is a high viscosity paste and is aerated through mixing and fermentation.

The structure of bread depends on the creation of gas cells during mixing and the effect of subsequent processing on manipulation and retention of these cells. The cells are incorporated during mixing, as the rough layers of dough fold over itself, enveloping pockets of gas with diameters on the micrometre scale. These gas pockets become the gas cell nuclei for the carbon dioxide generated by yeast to diffuse into and expand. However, not all cells will expand. A large number of cells incorporated during mixing are not visible in the final bread, as they do not grow or are physically unstable (Vliet, 1999). Shimiya and Nakamukra (1997) suggested that cells smaller than 100 µm in diameter during baking dissolved into the aqueous phase and concluded that cells must reach a critical size to expand. This is why it is vital that a sufficient number of gas cells of optimal size distribution are created during
mixing. Another is yeast cannot create new gas cells (Baker and Mize, 1941). Equation (2), the Laplace-Young equation, explains this:

$$P_b - P_\infty = \frac{2\sigma}{r} \quad (2)$$

where:
- $P_b$ = pressure inside the cell (N m$^{-2}$)
- $P_\infty$ = pressure outside the cell (N m$^{-2}$)
- $\sigma$ = surface tension (N m$^{-1}$)
- $r$ = cell radius (m)

The pressure inside the cell is greater than the pressure outside the cell, and the smaller the cell, the greater its internal pressure. If yeast were to generate a new cell, the starting radius would be zero, so the yeast would have to generate an infinite amount of pressure to create a new cell.

Initially the gas cells are largely full of nitrogen due to nitrogen’s low solubility in water, compared to carbon dioxide and oxygen. During proving, yeast generate carbon dioxide, creating a concentration gradient which aids the diffusion of carbon dioxide into cells. Carbon dioxide is high soluble, and diffuses into the aqueous phase until saturation is achieved.

As yeast ferment, the carbon dioxide concentration increases, expanding cells through the pressure generated inside them. The relationship between these parameters is governed by the ideal gas law (3):

$$PV = nRT \quad (3)$$

where:
- $P$ = absolute pressure (N m$^{-2}$)
- $V$ = volume of gas (m$^3$)
- $n$ = number of mol of gas (kg mol$^{-1}$)
- $R$ = universal gas constant (8.31 J mol$^{-1}$ K$^{-1}$)
- $T$ = absolute temperature (K)

It is not just the growth of the cells that is important, but also the rate at which the cells grow. To obtain a regular crumb, it is crucial that the cells grow at roughly the same rate (Vliet, 1999).

Once cells are entrained in the dough, there are a number of forces which determine whether they break up. The ratio of forces acting on breaking the cell to the surface tension which stabilises it is given by the Weber number.

$$We = \frac{rd}{\sigma} \quad (4)$$

where:
\[ We = \text{Weber number} \]
\[ \tau = \text{Shear stress (N m}^{-2}) \]
\[ d = \text{cell diameter (m)} \]
\[ \sigma = \text{Surface tension (N m}^{-1}) \]

Depending on the processing factors and the ingredients used, the maximum stable cell diameter will vary. Cell breakup occurs when the forces tending to rupture the cell are greater than the forces stabilising it.

The rheology of the dough is also key in retaining the cells and achieving high quality bread. A high viscosity dough is required to prevent the ascent of cells and an extensible dough is required to prevent cell rupture and to allow the dough to rise on baking.

Campbell and Shah (1999) demonstrated that both entrainment and disentrainment of gas occurs throughout mixing. Disentrainment occurs as the mixer blade cuts through the dough and ruptures existing cells (Campbell, 1991). In proving and baking, gas continues to be disentrained. This generally occurs once the cells have significantly increased in volume and are no longer stable and break up.

Cell coalescence is the fusion of cells caused by rupture of the dough film between them. Rupture of a dough film is caused by development of weak spots resulting from local thinning (Vliet et al., 1992; Vliet, 1999). The resistance of these dough films against biaxial extension determines the stability of the dough films against coalescence. Excessive coalescence causes a large loss of gas, resulting in a large loss in loaf volume. Coalescence is the main instability mechanism that occurs at the end of proving and during early stages of baking (Vliet, 1999) due to increased carbon dioxide production by yeast and the formation of steam causing cell expansion. Coalescence does not occur during mixing due to the cells being too small.

It is mostly accepted that cell disproportionation occurs at the later stages of proving and during baking. Cell disproportionation is the diffusion mechanism through which gas is driven from smaller cells to larger cells by Laplace pressure, the difference between the internal and external pressure of the cell. As the Laplace pressure is greater in smaller cells than larger cells, and gas solubility is greater at higher pressures, gas diffuses across the concentration gradient. This self-accelerating process results in the growth of large cells at the expense of smaller cells. The rate of disproportionation increases as the smaller cells shrink and their internal pressure increases. However, it has also been argued that disproportionation is unlikely, as gas from the cells would have to diffuse across the more concentrated dough matrix (Dobraszczyk et al., 2001).

Dobraszczyk et al. (2001) found the strain hardening properties of a material to affect cell rupture. The authors found that doughs with a higher strain hardening value have a greater failure strain, meaning they will inflate to a larger volume before rupturing. Strain hardening is therefore an important parameter for determining the stability of expanding cells. Strain
hardening is responsible for the increase in stiffness as strain increases. It stabilises regions of the cell wall that have been subjected to localised thinning, prolonging time to rupture.

### 2.2.2 Parameters during mixing affecting cells in bread dough

Mixing is the stage where bakers have the most control over the gas cells in bread dough. Bakers vary the different parameters to optimise the gas cell distribution in order to achieve the desired final product. The factors during mixing that affect the cells in bread doughs can be divided into two groups. The first is the ingredients and the second the processing. In Section 2.1.3, brief descriptions of the ingredient factors affecting the cells in bread were given. This section describes the processing factors.

**Gases**

The use of different mixer headspace compositions produce bread of differing characteristics, due to the different solubilities of gases and the dough chemistry. Gases which have been used to fill the mixer headspace include oxygen, nitrogen, carbon dioxide, a mixture of these gases in the form of air and in other proportions. Doughs can also be mixed in the absence of gas, such as under vacuum, although mixing entirely under vacuum produces extremely dense dough (Baker and Mize, 1937). In addition, the gas composition can be changed during mixing, to achieve the desired effect on cell size distribution, as is done in the CBP. This alters the cellular structure to that which would have been obtained from mixing under a single gas composition (Martin et al., 2008).

For the UK manufacture of bread via the CBP, oxygen is essential for the oxidation of ascorbic acid for dough development, helping the dough expand without rupture (Baker, 1941). Oxygen also improves the crumb structure and bread volume. In Cauvain and Young (2006), it was shown that Collins (1986) found on mixing different ratios of oxygen and nitrogen an increase in bread volume, up to a concentration of 60% oxygen. Mixing under an atmosphere of pure oxygen, however, is detrimental towards the crumb quality. It produces a coarse bread, resembling that of vacuum mixed doughs (Chamberlain and Collins, 1979). This is due to the yeast carrying out large quantities of aerobic respiration and producing more carbon dioxide. This causes the cells present to coalesce or breakup (Baker and Mize, 1941).

As with oxygen, mixing dough under an atmosphere of pure carbon dioxide results in a dense dough, and produces loaves of low volume with a coarse crumb (Martin et al., 2008). The authors also noted that the absence of carbon dioxide limits dough development. However, the high solubility of carbon dioxide gives the advantage of gas transfer to and from the cells and dough matrix on altering the pressure, thus enabling more control of the gas distribution.

Nitrogen is an essential gas during mixing, required for fine crumbed bread and yeast metabolism. Mixing under an atmosphere of pure nitrogen, however, has undesirable effects. It results in the production of a soft and sticky dough which is difficult to handle and dense when baked (Chin, 2003; Baker and Mize, 1937). This is due to the absence of oxygen.
The effect of different gases and their proportions illustrate the importance of mixer atmosphere in the processing of bread and determining its characteristics. In the CBP, air is used as the mixer atmosphere for the majority of the mixing process. Air contains a combination of gases which produce bread of acceptable quality. There is also no cost associated with air, compared to use of alternative gases.

**Pressure**

The pressure in the mixer affects the number and volume of cells in the dough. This is due to gas solubility being affected by pressure. Solubility of gases increases at higher pressures, thus at higher pressures there is an increased quantity of gas in the mixer headspace and thus the dough. On the other hand, at lower pressures, fewer cells are incorporated into the dough, resulting in less surface area for carbon dioxide diffusion during proving causing loss of a greater quantity of carbon dioxide to the atmosphere (Campbell *et al.*, 1998; Shah *et al.*, 1999). This produces denser bread (Chin *et al.*, 2005) with a coarser crumb (Martin *et al.*, 2008).

The two extremes of pressure are a high pressure and vacuum. The number of cells in dough following mixing is proportional to the mixing pressure (Campbell, 1998). In the CBP, mixing begins at a positive pressure under air. This increases the oxygen concentration in the mixer (Chamberlain and Collins, 1979) and the quantity of gas entrained in the dough, resulting in the formation of a greater number of new cells within the dough (Campbell, 1991). Mixing at a lower pressure, results in fewer cells and thus lower gas retention (Campbell *et al.*, 1998; Campbell, 1991). This can result in a finer texture, due to there being fewer cells initially and so coalescence less likely to occur. Mixing under vacuum results in a coarse bread of low volume (Baker and Mize, 1937). The gas cells are few and large. This is due to the removal of gas, which is necessary in creating the gas nuclei during mixing. The lack of oxygen also affects the chemistry of the dough. Pressure-vacuum mixing, however, produces a fine crumb. It causes the expansion of cells under the reduced pressure, allowing them to be subdivided into more cells (Hoseney, 1985). As mixing continues, the cell volume tends towards its new steady state.

The mixing pressure does not just affect the gas solubility and availability, it also affects the time to dough development and the dough consistency. When mixing at higher pressures, oxygen availability is increased, reducing the mixing time and work input required to achieve dough development (Chin and Campbell, 2005a). The dough consistency changes depending on the mixing pressure, due to lower pressures drying out the dough. Therefore, the lower the mixing pressure, the more water that must be added to the dough.

These factors highlight the importance of mixing pressure in obtaining desirable dough characteristics.
Dough development

One of the aims of mixing is dough development. When a dough is fully developed, all the flour particles should be hydrated (Hoseney, 1985), the dough should be viscoelastic with its gas content at the dough’s maximum capacity and gas cells should be retained without rupture. To develop a dough, energy must be input to the dough.

Every flour has a critical amount of work required for dough development that will produce the best bread. Mixing speed and time are the two main factors which determine the work input on dough. Kilborn and Tipples (1972) found however, that whilst the mixing speed can be used to compensate for the mixing time, the adverse is not the case. They found that each flour has a minimum mixing speed, known as the critical mixing speed, below which dough development cannot be achieved, regardless of the level of work input. This is due to mixing simultaneously aligning and misaligning molecules within dough. Mixing below the critical mixing speed facilitates misalignment of concatenations (Dobraszczyk et al., 2001) and produces a soft and sticky dough lacking spring. Bread made from this dough is inconsistent, has a reduced volume, and is generally of lower quality. It is agreed that increasing mixing speed for a set mixing time increases dough aeration through greater efficiency, producing loaves of greater volume (Campbell et al., 1993, Campbell et al., 1998; Campbell et al., 2001, Chin, 2003; Chin and Campbell, 2005b). In addition, mixing at higher speeds means less time is needed to reach peak dough development. To continue mixing once peak development is reached is termed unmixing. It is detrimental and results in the dough reverting to having the appearance of underdeveloped dough (Kilborn and Tipples, 1972; Cauvain, 2000). Unmixing induces stress on gluten molecules, overstretches them and causes them to disintegrate (Spaull and Bruce-Gardyne, 2003). This produces a wet and sticky dough (Hoseney, 1985) of lower extensibility and elasticity (Calderón-Domínguez et al., 2008). A reduction in dough extensibility reduces its ability to withstand the increasing pressure of growing cells.

As mentioned above, of the two main factors that affect dough development, mixing speed is the more important factor. The mixing time should be chosen around the critical mixing speed. Factors to consider when deciding upon the mixing time include: (1) a mixing time corresponding to dough development, (2) mixing conditions to allow entrainment of an optimum cell volume distribution, and (3) an optimal dough temperature at the end of mixing for optimal further processing.

With this section in mind, it is vital that bakeries optimise the mixing speed and time for each dough formulation they use, in order to fully develop each dough.

Mixing Scale

Dough aeration increases on scale-up, resulting in an increased quantity of gas in the dough (Campbell and Shah, 1999; Chin, 2003; Martin et al., 2004b). Martin et al. (2004b) found that by changing the mixing scale whilst maintaining other parameters, the dough contained the same numbers of cells with a different volume of gas. The effects of mixing scale are
important to bear in mind, as due to cost, convenience and availability, mixing in this PhD took part in a scaled down version of the industrial Tweedy mixer. These differ in scale by a factor of 600.

**Mixer Design**

The mixer design affects the mixing action. The mixing action is dependent on the shape of the mixer bowl, the blade geometry, the mixing speed and the rheology of the dough. The mixing action is important for dough development, as this is how the energy is transferred into the dough and also determines the dough rheology, which in turn affects dough aeration. Mixing actions which rub, cut or tear the dough damage it and should only be used if this damage is corrected later on in the breadmaking (Baker, 1941). Desirable mixing actions include folding, stretching and squeezing the dough. The type of mixing action used affects the density of the dough (Peighambardoust et al., 2010) and the cell volume distribution in the dough (Campbell et al., 1991).

Therefore, the quantity of gas incorporated, dough rheology and dough development depends on the mixer action and design. This again emphasises the importance of each detail during mixing on the cellular structure of the dough.

**2.2.3 Measuring aeration throughout breadmaking**

Bread dough aeration is an important quality parameter. Studies using a range of methodologies have been conducted investigating bread dough aeration at the different stages of breadmaking. These are detailed in this section. As the gas contribution towards the voidage is located in cells and its distribution is important, both voidage and cell size distribution in dough have been investigated. Whilst obtaining an accurate voidage measurement is relatively simple, obtaining the cell size distribution has proved challenging due to dough being opaque and its fragile structure requiring careful handling in order not to alter the cell size distribution (Bellido et al., 2006; Wagner et al., 2008a).

Literature shows that the use of density measurements is the most common method of determining bread dough aeration. This method has developed since 1946 when Baker and Mize measured the displacement of known weights of dough with oil. Since then, methods used include placing dough into liquids of known densities to find that of which the dough is neutrally buoyant (Campbell, 1991; Campbell et al., 1993), with some researchers freezing their dough beforehand for convenience and ease of handling, with the belief that dough structure is little influenced by freezing (Scanlon et al., 2008). This is unlikely, however, due to the thermal stresses induced by water as it freezes (Whitworth and Alava, 1999). Since 2001 the double cup method has been a popular method of determining dough density (Campbell et al., 2001; Chiotellis and Campbell, 2003; Chin et al., 2004; Martin et al., 2008). The double cup method was used for work in this thesis and is detailed in Section 3.4.3. It is convenient and has the advantages of being accurate, quick, simple, requiring no sample preparation and enabling measurements to be taken dynamically.
Dynamic measurements of dough aeration have also been monitored using a rheofermentometer with a gasometer unit recording carbon dioxide pressures (Shah et al., 1999) and through collection of the carbon dioxide produced (Peighambardoust et al., 2009). Every method has its pros and cons. For example, the rounding of the small samples in the method used by Peighambardoust et al. (2009) is likely to have caused loss of gas, impairing the accuracy of the results. Advantages of this method includes ease of obtaining the equipment, and the small sample size requiring only a small quantity of ingredients, which can be useful if working with rare flour varieties or scarce quantities of flour.

Change in dough volume has been monitored via digital image analysis to determine changes in dough voidage (Shehzad et al., 2010). Models have also been developed to determine dough aeration, based on experimental results. Elmehdi (2003) developed a model based on the non-invasive technique, ultrasound. Velocity and attenuation of ultrasonic waves through the dough was plotted as a function of proving time. Changes in the velocity and attenuation coefficient illustrated the potential of low frequency ultrasound in detecting changes in the dough structure. Shah et al. (1999) developed a model for proving, enabling carbon dioxide production and cell growth to be investigated. However, the cell size distribution, coalescence, yeast kinetics and dough properties were ignored, resulting in an overestimation in cell growth. This model was later extended by Campbell and Herrero-Sanchez (2001) to incorporate a range of cell size distributions. Although the model generated improved results, a number of simplifying assumptions had been made. Huang and Kokini (1999) and Martin et al. (2004a) also developed models for dough aeration. Martin et al. (2004a) used a population balance model to model bubble entrainment, disentrainment and break up over the whole duration of mixing. Understanding these parameters enables improved control of mixing, helping deliver the desired structures in breads. The authors solved their population balance model for different cell breakup scenarios: various breakup, no breakup and instantaneous breakup, although they concluded from the results that the instantaneous breakup model could not quantitatively describe the results throughout the whole mixing process. However, good agreement was found between their no breakup and various breakup model with experimental results. Their no breakup population balance model is used in Chapter 5 of this thesis to assess the effect of sugar on bread dough aeration. It uses voidage measurements from experiments to assess the degree of entrainment and disentrainment during mixing and was developed to account for the experimental voidage changes during mixing following a pressure step change. A detailed description of the model is given in Section 2.2.4.

Gas production has also been monitored throughout baking using a gas analyser equipped with an infrared detector in the oven (Zhang et al. 2007; 2008). Magnetic resonance imaging (MRI) has proved a popular technique for monitoring dynamic processes (Grenier et al., 2003; Zhang et al., 2007; Wagner et al., 2008a; Guio et al., 2009). It is based on nuclear magnetic resonance (NMR) and relies on energy absorption and emission. Researchers have distributed microcapsules throughout the dough and related changes in their positions to the structural
changes in dough. However, use of microcapsules throughout dough can interfere with cell mechanics and heat and mass exchange (Wagner et al., 2003).

An established method of determining the aeration of finished bread is through density measurements using the seed displacement method (Calderón-Domínguez et al., 2008). Although time consuming, this method is simple and inexpensive.

As mentioned previously, accurately determining cell sizes in bread dough has proved a challenging task. Over the years, the methodology for determining cell sizes in dough has advanced significantly. Higher resolutions are now obtained, using non-destructive and non-invasive techniques. Table 2.1 lists some resolutions and cell densities obtained using different imaging techniques on bread dough from some publications which are discussed in this section. Some variation is expected amongst the observed cell densities due to the different techniques used, their resolutions and differences in ingredient formulations and dough processing.

<table>
<thead>
<tr>
<th>Authors, year</th>
<th>Technique</th>
<th>Resolution (µm)</th>
<th>Cell density</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carlson and Bohlin, 1978</td>
<td>Light microscopy</td>
<td>90</td>
<td>87600 cm³</td>
</tr>
<tr>
<td>Bloksma, 1990</td>
<td>Microscopy</td>
<td>-</td>
<td>10^3-10^5 cm³</td>
</tr>
<tr>
<td>Campbell et al., 1991</td>
<td>Microscopy</td>
<td>39</td>
<td>78500 cm³</td>
</tr>
<tr>
<td>Campbell et al., 1991</td>
<td>Microscopy</td>
<td>39</td>
<td>33 100 cm³</td>
</tr>
<tr>
<td>Shimiya and Nakamura, 1997</td>
<td>Optical microscopy, light microscopy</td>
<td>3, 40</td>
<td>300 cm³</td>
</tr>
<tr>
<td>Whitworth and Alava, 1999</td>
<td>Optical microscopy</td>
<td>28</td>
<td>-</td>
</tr>
<tr>
<td>Whitworth and Alava, 1999</td>
<td>SEM</td>
<td>50-3500</td>
<td>-</td>
</tr>
<tr>
<td>Whitworth and Alava, 1999</td>
<td>X-ray CT</td>
<td>1000</td>
<td>-</td>
</tr>
<tr>
<td>Bellido et al., 2006</td>
<td>X-ray CT</td>
<td>10</td>
<td>30410 cm³</td>
</tr>
<tr>
<td>Bellido et al., 2006</td>
<td>X-ray CT</td>
<td>10</td>
<td>56540 cm³</td>
</tr>
<tr>
<td>Whitworth, 2008</td>
<td>X-ray CT</td>
<td>680</td>
<td>-</td>
</tr>
<tr>
<td>Babin et al., 2008</td>
<td>X-ray CT</td>
<td>15</td>
<td>-</td>
</tr>
<tr>
<td>Turbin-Orger et al., 2012</td>
<td>X-ray CT on synchrotron</td>
<td>5</td>
<td>-</td>
</tr>
</tbody>
</table>

Ultrasound is an example of a non-invasive technique that has been used to measure the cell size distribution in bread dough (Leroy et al., 2008; Scanlon et al., 2008). Through use of different ultrasonic frequencies, different structural features can be detected, and cell sizes can be determined. This is due to the large density difference between the dough matrix and cells, and the ease in propagation of ultrasonic waves depending on material density. Cell
size determination relies upon the dependency of characteristic resonance frequency on cell size. Although ultrasound is non-invasive, like microscopic techniques, preparation of the sample can alter its structure. Microscopy was the first technique used in literature to measure cell size distribution in bread dough. Using light microscopy, a resolution of 90 µm was obtained (Carlson and Bohlin, 1978). Cells as small as 5 µm in diameter have now been reliably detected (Turbin-Orger, 2012) and it is likely that smaller cells exist, thus the cell size distribution obtained from Carlson and Bohlin would have been inaccurate, due to a significant number of undetected cells.

There are many types of microscopy and although they can be used to visualise structures which cannot be seen by the unaided human eye, the use of 2D images are not as useful as 3D images. In 1991, Campbell et al. noted that when measuring the cell diameter from a 2D image, the diameter is likely to be underestimated, due to the likelihood of cells being sliced off centre, thus the authors extended Carlson and Bohlin's method to take this into account; the addition of a stereological technique was used to reconstruct the true cell size from the 2D images. The authors were able to measure cells as small as 39 µm in diameter, and found a mean cell diameter of 71 µm and 89 µm in food processor and Tweedy 10 dough respectively and noted the observed voidage was always lower than density measurements. This is likely due to the resolution causing both smaller and larger cells to be missed. Campbell et al., (1991) used the technique of digital imaging to obtain the cell size distribution within their dough samples. Digital imaging techniques are popular due to their speed, reduced difficulties in quantifying structural features (Falcone et al., 2006), and reduced susceptibility to human error (Sapirstein, 1999; Zghal et al., 1999, Bellido et al., 2009; Pérez-Nieto et al., 2010). Unfortunately, they have the disadvantage of thresholding deviations having a significant effect on results (Sapirstein, 1999).

Scanning electron microscopy (SEM) has also been used to obtain cell size distributions (Whitworth and Alava, 1999; Calderón-Domínguez et al., 2008; Baron and Butler, 2008). Electron microscopy focuses a beam of electrons onto the sample. Through emission and reflection of the sample surface, the topography of the dough can be obtained. Confocal laser scanning microscopy (CLSM) is another method which uses microscopes to visualise bread dough structure (Dürrenburger et al., 2001; Li et al., 2004; Upadhayay et al., 2012). The sample is uniformly illuminated and images are taken from one focal plane, which can be assembled to give the 3D structure of the dough. Thin sections of thick samples can be imaged with CLSM. However, only cells close to the surface can be visualised and it is possible that the cell size distribution here differs to the bulk of the dough (Deshlahra et al., 2009). In addition, samples need to be treated for visualisation, which although useful for highlighting certain components can alter their structure. Shimiya and Nakamura (1997) used optical microscopy and stereomicroscopy to investigate cell size distribution in bread dough, obtaining resolutions of 3 µm and 40 µm, respectively. 3 µm is the lowest 2D resolution of bread dough imaging published to date. However, their methodology sacrificed precision. Dough samples were compressed between glass slides. Preparation of their dough most likely
altered cell volumes, caused some cell rupture, and led to a number of cells being undetected, as reflected by the lower cell number they detected compared to others. This illustrates the necessity of a non-destructive method for obtaining the cell size distribution in bread dough.

In 2006, Falcone et al. published a review on techniques used to study the microstructure of food. They illustrated the wide range of techniques that have been used and showed how choice of techniques depended upon the product being investigated i.e. how fragile it is and whether it is susceptible to the technique. The authors highlighted that preparation of samples for microscopic techniques frequently result in artefacts, thus they recommended that more than one microscopic technique is used in obtaining results. With this in mind and the generation of 2D images, micrographs cannot accurately represent the cell size distributions in bread dough, as reflected by the increasing popularity of X-ray imaging seen in the increase in number and development of synchrotron facilities worldwide. X-ray CT is based on the contrast in X-ray images, arising from differences in density within samples. X-rays are passed through an object from different angles, producing images that show the density differences at different points throughout the sample. These images, known as orthoslices, can be stacked up using a technique called X-ray CT to generate a 3D distribution of material density within the object. Currently, sub-micron resolution is attainable in the laboratory. However, high resolutions are at the expense of a longer acquisition time and smaller sample sizes, and must be achieved without compromising the field of view. X-ray CT is used in many applications including medicine, material science, geology, biology, chemical engineering and also food, mainly to study the microstructure of aerated food products and its relation to the properties of these products. Some foods investigated include ice cream, dairy samples and dried vegetables (Dalen, 2012); chocolate (Frisullo, 2010), processed meat (Frisullo, 2009), and bread (Whitworth an Alava, 1999; Falcone, 2005; Bellido, 2006; Babin, 2006; 2008, Whitworth, 2008; Turbin-Orger, 2012). Lim and Barigou (2004) listed some of the advantages of X-ray CT over microscopy. These include (1) samples can be visualised in their natural state at atmospheric pressure, preventing sample preparation artefacts, (2) high spatial resolution, and (3) 3D details of the inner microstructure as opposed to surface structure can be captured. Synchrotron light consists of beams of X-rays, infrared and ultraviolet light, combined into an intense light. This intense light is generated by synchrotron beamlines, through use of particle accelerators, which cause the electrons to change direction. Synchrotron X-rays have all the benefits of X-rays over microscopic techniques, as well as the advantage of being able to image a larger range of materials and sample thicknesses than conventional sources, and produce images of higher resolution and contrast. Through using coherence and phase contrast branches of synchrotron beamlines, it is possible to obtain resolutions beyond the limitations of the detector and X-ray optics, and obtain better contrast, enabling features of similar density to be distinguished.

Researchers investigating bread dough aeration have used X-rays to obtain detailed information on the cellular structure of bread dough during proving (Turbin-Orger et al.,
2012), and both proving and baking (Whitworth and Alava, 1999; Babin et al., 2006; 2008, Whitworth, 2008), on dough generally (Whitworth and Alava, 1999) and finished bread (Falcone et al., 2005). Babin et al. (2006) observed little change in the cell structure and cell wall structure during baking and illustrated the significance of proving on the cell structure of bread.

In recent years, the synchrotron has been used to probe into the dynamic structure of dough (Babin et al., 2006; 2008; Turbin-Orger et al., 2012). Although researchers using this technique have published the cell size distributions of dough samples, they have not utilised the synchrotron to its full capacity and provided cell size in terms of mean diameter, as opposed to volume. Cell volume is more representative of cell size than cell diameter, as images of cells show cells to range from spherical to ellipsoid in shape (Babin et al., 2006; 2008; Bellido et al., 2006).

The use of conventional X-ray CT to visualise the microstructure of dough has cost and availability advantages over synchrotron light. Like the synchrotron, conventional X-ray scans are a non-destructive and non-invasive method of determining the microstructure of dough. Conventional X-ray sources can also produce high resolution and contrast images, enabling the dough voidage, the number of cells and their volume and the distance between cells to be determined, and visualisation of cell interaction and dynamic processes through fast X-ray CT (Whitworth and Alava, 1999; Whitworth, 2008). The authors obtained a resolution of 1 mm and 0.7 mm respectively - much lower than the resolution X-ray CT is capable of obtaining, illustrating the importance of the instrument used and the sample set up. One problem with using X-ray CT to visualise dough microstructure is the micrometre scale diameters of the cells means detection of the smallest cells requires the dough sample to be on the millimetre scale size. Therefore in order to obtain a more representative insight into the microstructure, either experimental repeats, or the scanning of a range of sizes of dough samples is necessary.

Attempts to quantify the microstructure of dough throughout mixing using X-ray CT were previously made by a research group at the University of Manchester. The group looked at cell size distribution and voidage distribution in both single pressure doughs and doughs subjected to pressure-vacuum mixing. Their results were not published due to the large disparity between the density measurements obtained via the double cup method and X-ray CT. This is illustrated in Figure 2.2, where some of their results are presented. The research group experimented with their sample set up and X-ray settings, but felt the root of their problem was the sample handling. They felt the sample was compressed a lot to get it into the sample holder, resulting in a non-representative dough sample. This illustrates the challenge encountered when handling the fragile dough samples, and highlights that quantitative information obtained from published X-ray CT work, like other methods relies upon a suitable method of sample preparation.
Figure 2.2 Unpublished voidage measurements obtained from X-ray CT. The results show the voidage measured from X-ray CT and the double cup method, illustrating the large disparity between the measurements.

As well as X-ray CT, the degree of aeration in finished bread can be analysed through digital imaging software such as the C-Cell imaging system which investigates the bread crumb structure (Sroan and MacRitchie, 2008; Martin et al., 2008b; Whitworth, 2008). The C-Cell imaging system was developed by the Campden and Chorleywood Food Research Association for evaluation of bread. It is quick and easy to use and produces consistent results. It was used for work conducted within this thesis and is detailed in Section 3.3.5.

2.2.4 Quantifying aeration using a population balance model

The population balance model published by Martin et al. (2004a) was the first model that enabled prediction of the voidage response to pressure step changes and thus enable design of pressure-vacuum mixing procedures. The model was also the first to provide a reliable measurement of the gas entrainment rate into the dough and therefore the oxygen available for dough chemistry. However, a limitation of the model is that only a small number of standard estimated cell sizes were used in the model. Section 6.3 in this thesis applies the cell sizes obtained from X-ray tomography to assess the validity of this model. This section describes the model. The scenarios to which the model can be applied, some equations in the model, how parameters within it relate to the experiments and the assumptions made on application of the model are also discussed.

One of three scenarios can be made to which the model can be applied: the two extremes: instantaneous cell breakup and no cell breakup, and the option in between, variable cell breakup, which is the most realistic situation. Results published by Martin et al. (2004a) showed the instantaneous breakup model did not representatively model the aeration process throughout the whole mixing process. The other two scenarios showed a good fit to experimental results, with the no breakup model simplifying the equations involved, thus this option was used to model aeration in the three different sugar content doughs.
The no breakup model is based on the assumption that no cell breakup occurs over the mixing session and therefore the number of cells is constant over the step change.

It is important to model the ex-situ dough properties as these are the dough conditions for the next stage of the breadmaking process. For ex-situ measurements the dough is at atmospheric pressure and therefore density and voidage measurements are taken at atmospheric pressure. Dough voidage and density are related using the gas free dough density, as seen in Equation (5).

\[ \Phi_{\text{outside}} = 1 - \frac{\rho_{\text{dough}}}{\rho_{\text{gf}}} \]  

(5)

where \( \Phi_{\text{outside}} \) is the dough voidage outside the mixer, and \( \rho_{\text{dough}} \) (g cm\(^{-3}\)) and \( \rho_{\text{gf}} \) (g cm\(^{-3}\)) are the dough density and dough voidage respectively.

However, if the dough was mixed at a different pressure, the dough experiences an almost instantaneous change in external pressure on removal from the mixer to atmospheric pressure.

Cells will respond to a change in external pressure. The pressure of a bubble of radius \( r \) with a dough height of \( z \) above it, is the sum of four components: the external pressure, \( P \), the hydrostatic pressure, \( \rho gz \), the effect of surface tension, \( \sigma, P_s = 2\sigma/r \), and the extra pressure due to the stresses generated by mixing, \( P_t \). The hydrostatic pressure is negligible given the size of the dough piece. Campbell et al., (1998) estimate the surface tension pressure to be 0.016 bar, and the extra mixing pressure to be 2 bar throughout mixing and zero as soon as mixing comes to an end. The extra mixing pressure only occurs in dough regions experiencing large rates of deformation, compressing cells in these regions until the deforming regions move on and the region returns to ambient pressure. During pressure vacuum mixing, the headspace pressure is changed from one pressure \( P_1 \) to another pressure, \( P_2 \), and on removal of the dough to atmospheric pressure, \( P_{\text{atm}} \). This occurs within a couple of seconds (Martin et al., 2004a). The proportion of cells compressed is assumed to be insufficient to affect the mean dough voidage. Equation (6) takes into account this step change and relates the voidages before and after the step change using the ideal gas law:

\[ \frac{\Phi_{\text{outside}}}{\Phi_{\text{inside}}} = \frac{P_2}{P_{\text{atm}}} \left[ 1 + \left( \frac{P_2}{P_{\text{atm}}} - 1 \right) \Phi_{\text{inside}} \right] \]  

(6)

where \( \Phi_{\text{inside}} \) is the dough voidage inside the mixer, \( P_{\text{atm}} \) is the atmospheric pressure (bara), \( P_2 \) is the second mixing pressure (bara) and \( \Phi_{\text{outside}} \) is the dough voidage outside the mixer.

The model involves a mass balance of dough aeration, where the net birth rate of cells depends upon (a) incorporation of cells through entrainment; (b) loss of cells through disentrainment; (c) loss of cells through breakup; (d) incorporation of cells through larger cells breaking up into smaller ones. Gas is entrained when two dough surfaces come into
contact, trapping gas between them. The model assumes this is independent of other conditions and therefore the number of cells entrained per volume per revolution, \( N_{\text{in}} \), is constant. There are three stages of cell break up as proposed by Campbell et al. (1998) based on the experimental finding that the number of cells per volume of dough, is approximately proportional to the mixing pressure: (a) entrained cells have the same pressure as the headspace and a volume independent of the headspace; (b) all cells are compressed to the same volume by any change in mixing pressure; (c) compressed cells break up into cells of constant volume and the number of cells depends upon the compressed volume. Equation (7) expresses the ratio of the compressed cell volume at pressure \( P + P_\tau \) to the uncompressed volume at pressure \( P \):

\[
\frac{V_t}{V} = \frac{1}{1 + \frac{P_\tau}{P}}
\]

If \( P_\tau \) is large and constant compared with \( P \), then the number of cells per unit volume is approximately proportional to \( P \).

It is assumed here that only small regions of the dough are stressed and break up only occurs in these regions. The release of stress from these regions immediately results in the cells returning to their original pressure and expanding.

It was hypothesised for design of the model that the probability of cell breakup and disentrainment phenomena is proportional to cell volume, with disentrainment being more likely in larger cells. The model makes a series of additional assumptions for simplicity and its functioning: (1) cells are evenly distributed throughout the dough during mixing, (2) cell growth does not occur, (3) mixing is rate-independent, (4) the only significant property of a cell is its volume, (5) the pressure change occurs instantaneously (6). All cells have the same volume, \( V_{\text{in}} \), regardless of headspace pressure, and (7) coalescence is negligible because of the high viscosity of the continuous phase and the relatively low voidage.

The model is fitted to the results through and using Solver in Microsoft Excel to minimise the root of the mean of the squares of absolute errors (RMSE) by changing the parameters: initial voidage, entrainment rate and disentrainment coefficient.

The model fitting for the no breakup model following a step change is based on Equation (8):

\[
\Phi = \frac{N_{\text{in}}V_{\text{in}}}{V_{\text{in}}w_{\text{d}}C_{\text{d}}} \left( 1 - e^{-V_{\text{in}}w_{\text{d}}C_{\text{d}}(\zeta - \zeta_0)} \right) + \Phi_{\text{step}} + e^{-(P_1/P_2)}V_{\text{in}}w_{\text{d}}C_{\text{d}}(\zeta - \zeta_0)
\]

where the first term on the right hand side of the equation models the voidage contribution of cells entrained after the step change and the second term the voidage contribution prior to the step change. The nomenclature for Equation (8) is as follows:

\( \Phi \) dough voidage (volume of gas per unit volume of dough)
$N_{in}$ number of cells entrained per unit volume per revolution
$V_{in}$ standard entrained cell volume (mm$^3$)
$\omega_d$ disentainment frequency per unit volume
$C_d$ cell disentainment probability constant of proportionality
$\zeta$ number of mixer revolutions
$\zeta_0$ number of mixer revolutions at initial state
$P_1$ first mixer headspace pressure
$P_2$ second mixer headspace pressure
$\Phi_{step+}$ voidage immediately after the pressure step change

2.3 Summary of bread’s history, significance and manufacture

Bread is an aerated food made from a mixture of grains and liquid, and is consumed worldwide. Its microstructure determines its characteristics and plays a significant role in its popularity amongst consumers. Different bread products are purchased for their qualities which are dependent upon their cell size distributions. Ciabatta is enjoyed for the large cells which can hold pools of olive oil, and sandwich bread is enjoyed for being soft and uniform, a result of the many small cells that make up the product. The aerated structure of bread begins during mixing and changes over the breadmaking process. It is important to determine the cell size distributions following different processing parameters and ingredients to understand the links between the processing variations, cell size distribution and quality of the final bread produced. A large number of techniques have been used over the years to measure dough voidage and investigate its microstructure, both statically and dynamically. Recent years have seen an advance in the study of the fragile material through use of non-invasive and non-destructive methods, at increasingly high resolutions. In 2012, a publication detailed the observation of cells as small as 5 µm in diameter using X-ray CT via a synchrotron. This is the highest resolution of 3D bread dough imaging to date and although the resolution was not high enough to conclude that cells smaller than this exist, this and other benefits of the technique highlights the superiority of X-ray imaging via a synchrotron beamline over other imaging techniques.
Chapter 3 - Experimental methodologies

3.1 Outline

Chapter 3 is the first experimental chapter in this thesis and is present to ease reading of the upcoming experimental chapters through presenting material that would otherwise be repeated throughout this thesis. This chapter is divided into four main sections: Section 3.2 and Section 3.3 list the materials and equipment used, respectively. Section 3.4 describes the analytical techniques used throughout this thesis. Section 3.5 characterises the flour and dough formulations used throughout this thesis.

3.2 Materials used

The materials used for this thesis and their procurement sites are presented in Table 3.1. Due to experiments having been performed on different formulation doughs, their quantities for each experiment are detailed in the relevant experiment.

<table>
<thead>
<tr>
<th>Material</th>
<th>Procurement site</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flour a, 10.1% protein</td>
<td>3663, High Wycombe, UK</td>
</tr>
<tr>
<td>Flour b, 10.4% protein</td>
<td>3663, High Wycombe, UK</td>
</tr>
<tr>
<td>Flour c, 11% protein</td>
<td>3663, High Wycombe, UK</td>
</tr>
<tr>
<td>Flour d, 11.5% protein</td>
<td>3663, High Wycombe, UK</td>
</tr>
<tr>
<td>Flour e, 15% protein</td>
<td>Waitrose, Berkshire, UK</td>
</tr>
<tr>
<td>Water</td>
<td>University of Manchester, Manchester, UK</td>
</tr>
<tr>
<td>Sugar</td>
<td>Tate &amp; Lyle, London, UK</td>
</tr>
<tr>
<td>Salt</td>
<td>Sainsburys, Holborn, UK</td>
</tr>
<tr>
<td>Cornflour</td>
<td>Sainsburys, Holborn, UK</td>
</tr>
<tr>
<td>Fresh yeast</td>
<td>Sainsburys, Holborn, UK</td>
</tr>
<tr>
<td>Quick yeast</td>
<td>Doves Farm, Berkshire, UK</td>
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<tr>
<td>Sunflower oil</td>
<td>Waitrose, Berkshire, UK</td>
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<tr>
<td>Vegetable fat</td>
<td>Trex, Liverpool, UK</td>
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<tr>
<td>Ascorbic acid</td>
<td>Holland &amp; Barretts, Nuneaton, UK</td>
</tr>
<tr>
<td>Dough improver</td>
<td>CSM, Manchester, UK</td>
</tr>
<tr>
<td>Bread loaves</td>
<td>Supermarkets, UK</td>
</tr>
<tr>
<td>5 cs Silicone oil</td>
<td>Basildon Chemicals, Oxon, UK</td>
</tr>
</tbody>
</table>

Five flours were used due to their differing protein contents. Their protein contents were 10.1%, 10.4%, 11%, 11.5% and 15%, according to packaging, corresponding to flour a, b, c, d and e, respectively. Technically, flours a and b are classified as weak flours, and flours c, d
and e as strong flours, due to their protein contents. Note that although five flours were used, these were not simultaneously compared on each occasion. Initially two flours were procured, flours a and c, their packaging labelled as plain flour and strong white flour. These are generally referred to as “weak” and “strong” flour respectively. Experiments were usually conducted on both flours to test that the results were not just applicable to that flour. Sufficient flour from the same batch was procured for all experiments in this study. This plan eliminated the need for multiple flour characterisations and ensured that flours with identical properties, the deterioration associated with age aside, were used throughout all experiments. However, an equipment fault led to the repeat of a series of experiments, thus more flour was procured. Flours b and d were brought to replace flours a and c. These were the plain and strong flours sold by the food wholesaler 3663 at the time. Flour e was chosen for its very high protein content, typical of a superior hard wheat grown in Canada, and used for the fast X-ray imaging of bread dough proving described in Section 6.4 to ensure representative visualisation of proving.

Fresh yeast was initially used in experiments due to it most closely matching the yeast used for industrial breadmaking, in terms of confidence levels of active yeast. However, due to fresh yeast being difficult to obtain, and its carbon dioxide producing properties not required in all experiments, it was later eliminated from the dough formulation. When yeast was required in the proving experiment described in Section 6.4, convenient quick yeast was used.

Within each formulation, although the sugar content changes, the quantities of the other ingredients remain the same, and each formulation is presented on the basis of the sugar content as a Baker’s percentage. Changing the quantity of water in proportion to the quantity of sugar was considered to ensure gluten hydration and the relatively soft dough characteristic of CBP doughs. The concern of the addition of water increasing dough stickiness and causing processing difficulties, and how much the water content should be increased to take into account the increasing sugar levels then arose. It is known that when increasing the salt concentration during industrial manufacturing of bread, water levels are increased (Hirning, 2010). However, the function of salt in a dough formulation differs to that of sugar and therefore how the water levels are adjusted may vary. In a study on the effect of bran levels during breadmaking processes, Campbell et al. (2008a) substituted weight for weight, flour with bran, as wheat bran was found to increase water absorption. Measuring the water absorption for each formulation would have been one method of determining how much water should be added to compensate for the increased sugar levels. However, access to equipment capable of this was not readily available.

3.3 Equipment used

Equipment used in the experiments described in Chapters 3-6 are listed in Table 3.2. Note not every piece of equipment is listed. Standard equipment such as stopwatches and razor blades, for example, have not been included in Table 3.2.
### Table 3.2 Experimental equipment, their makes and information on use of the equipment

<table>
<thead>
<tr>
<th>Equipment used</th>
<th>Information</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tweedy 1 Mixer</td>
<td>See Section 3.3.1</td>
</tr>
<tr>
<td>Nikon Metris 225/320kV X-ray CT</td>
<td>See Section 3.3.2</td>
</tr>
<tr>
<td>Brabender Farinograph</td>
<td>See Section 3.3.3</td>
</tr>
<tr>
<td>Newport Scientific Micro-Dough Lab</td>
<td>See Section 3.3.4</td>
</tr>
<tr>
<td>Calibre C-Cell CC200.05</td>
<td>See Section 3.3.5</td>
</tr>
<tr>
<td>Stable Micro Systems TA.XT. Plus</td>
<td></td>
</tr>
<tr>
<td>Texture Analyser</td>
<td>See Section 3.3.6</td>
</tr>
<tr>
<td>FEI Quanta 200 Environmental</td>
<td>A type of scanning electron microscope</td>
</tr>
<tr>
<td>Scanning Electron Microscope</td>
<td></td>
</tr>
<tr>
<td>Kenwood Chef Classic KM336</td>
<td>Kitchen planetary mixer</td>
</tr>
<tr>
<td>Hinari Homebaker</td>
<td>Breadmaker</td>
</tr>
<tr>
<td>Simon Rotary Test Baking Oven</td>
<td></td>
</tr>
<tr>
<td>Fischer Scientific ET100 Water Bath</td>
<td></td>
</tr>
<tr>
<td>Ohaus Explorer® Pro Mass Balance</td>
<td>Accurate to nearest 0.01 g, used for weighing of salt, ascorbic acid and dough improver</td>
</tr>
<tr>
<td>Mettler PE 6000 Mass Balance</td>
<td>Accurate to nearest 0.1 g, used for weighing materials not weighed on the Ohaus Explorer® Pro mass balance</td>
</tr>
</tbody>
</table>
3.3.1 Tweedy 1 mixer

The Tweedy 1 mixer is a scaled down version of the Tweedy mixer, the key component of the CBP. It is used to produce bread industrially in numerous countries worldwide, including the UK, New Zealand and Australia. Its name, “Tweedy 1”, is based on its capacity of 1 lb of flour. The key feature of the mixer is its fast mixing speed, capable of producing a homogeneous and developed dough in less than 5 minutes. In a typical low speed mixer, this would take approximately 10 minutes. The Tweedy 1 mixer is illustrated in Figures 3.1 to 3.3.

Figure 3.1 Inside the Tweedy mixer

Figure 3.2 Outside the Tweedy 1 mixer with mixer lid in position
The mixer blade sits in the bottom of the mixer bowl. It is attached to a centrally rotating spindle and consists of an octagonal base with two helical paddles. The shape of the blade ensures a good shearing action during mixing. There are three baffles in the mixer bowl. Their presence creates an obstruction, helping rotate the dough and ensuring thorough mixing. The top of the mixer bowl is lined with neoprene. This ensures an airtight environment when the lid shown in Figure 3.2 is fully clamped onto the mixer bowl. An airtight environment is essential in maintaining the headspace pressure. To alter the headspace pressure or composition, a gas pipeline must be attached to an attachment point. The degree the valves are open affect the rate the headspace composition and pressure are altered. The pressure gauge and gas flow monitor attached to the mixer allow the pressure and gas flow rate in the mixer to be monitored.

The Tweedy 1 mixer is linked to a data acquisition computer, illustrated in Figure 3.3. Using this set up measurements of the mixing speed, torque and time can be recorded.

The speed at which the Tweedy 1 mixer operates can be varied using the speed control box. The speed settings range from 0-50, where 50 is equivalent to approximately 77 rad s⁻¹. Unless otherwise stated, the speed setting of 45 was used, equating to a nominal speed of approximately 70 rad s⁻¹. This speed was a compromise for ensuring long term care of the mixer, whilst using a high but not maximum mixing speed.

When operating the Tweedy 1, the liquid was first put into the mixer bowl, followed by the dry ingredients due to mixing being more efficient than when loading the mixer bowl in the reverse order.
Each dough made in the Tweedy 1 mixer was based on a loading of 400 g of flour. A test series of doughs were developed according to their work input after mixing under these conditions.

Unless otherwise stated, each dough was mixed for 3 minutes and at a pressure of 1 bara. For the doughs tested, this was found to be sufficient time to develop the dough based on the work input. All pressure step changes occurred within 3 s.

Occasionally in the Tweedy mixer the dough ingredients are not mixed fully into a dough or the mixing pattern will differ to that of the norm. In a normal mixing pattern, a single dough ball is kneaded as it rotates around the mixer bowl between the mixer blade and wall, with the helical impeller mixing downwards. The dough mix will differ from the norm when the mixing pattern is such that the dough spins around with the blade at the top of the mixer, or when a proportion of the dough ingredients stick onto the sides of the mixer and are not incorporated into the dough. When these problems occurred the batch of dough was discarded and another batch made, to avoid anomalous results.

3.3.2 Nikon Metris 225/320kV X-ray CT

The Nikon Metris 225/320 kV X-ray CT (XT H 225) is housed in a customised bay with the Manchester X-ray Imaging Facility at the University of Manchester. It is illustrated in Figures 3.4 and 3.5. This X-ray equipment was used for scanning bread dough, as after mixing cell sizes are on the micrometre scale (Whitworth and Alava, 1999), the resolution the XT H 225 achieves. The resolution of the other X-ray equipment at the facility meant the XT H 225 was best suited to the scale of the features to be detected within bread dough samples. The high resolution is due to the large detector (2K x 2K Perkin Elmer 1621-16-bit amorphous silicon flat-panel detector with 200 micron pixel pitch). With the appropriate X-ray settings and sample volume, a sample resolution of 3 µm can be achieved. This was not possible with the bread dough samples used, however. In addition, the detector allows fine differences in contrast to be detected. This enables detection of a number of features which would not have been seen on the standard Nikon Metris 225 kV X-ray CT instrument.
**Figure 3.4** Nikon Metris 225/320kV X-ray CT. The source, manipulator stage and detector positions can be adjusted to optimise scan settings.

**Figure 3.5** Nikon Metris 225/320kV X-ray CT set up. This shows the equipment housed in a customised bay, whilst control of the equipment is located outside the bay for safety purposes.
The target material within the source is interchangeable to ensure the highest X-ray density for the energy range is used for the sample material. This ensures the best signal and contrast for the material. With these target materials, an energy range of 40-230 kV can be obtained, allowing the scanning of biological materials through to dense metals. For the X-ray imaging conducted within this thesis, either molybdenum or tungsten was the target material used. When imaging static dough, molybdenum was used, and when imaging dynamic dough, tungsten was used. Different target materials were used due to the differing densities of the glass and plastic sample holders.

The large walk-in bay and heavy duty sample manipulator means samples as large as 1 m in diameter and 1 kg in weight can be imaged. The material to be scanned sits upon the heavy duty rotating manipulator stage. The stage position can be adjusted using a series of joysticks. The stage also allows for attachments of specialist rigs, enabling the addition of another dimension to the scan. As the sample is scanned, the scan data is collected on an acquisition PC.

3.3.3 Farinograph
The Farinograph is a mixer commonly used worldwide to determine flour quality through dough consistency measurements on dough as it is mixed. The torque is measured on the sigma shaped paddles of the mixing blade, and graphically recorded as a function of time by a pen actuated by a system of levers. The axes on the graph paper are time and Brabender units, which is essentially the torque. From the curve produced, information such as the flour water absorption, flour hydration time, dough breakdown, stability and development time, and mixing tolerance index can be derived.

3.3.4 Micro-Dough Lab
The Micro-Dough lab is a mixer with the loading capacity of a dough made from 4 g of flour. It is used to determine the quality and processing characteristics of flour and dough from torque measurements. Its small sample volume requirement has the advantage that very small quantities of material are required to determine flour quality and processing characteristics. The Micro-Dough Lab has a sigma shaped mixing paddle, which mixes the dough at the chosen speed ranging from 0-200 rpm. It can be programmed to mimic the Farinograph method of determining water absorption and has the advantages of more precise temperature control and automated water delivery to the sample.

3.3.5 C-Cell
The C-Cell is a digital imaging system used throughout the bread industry worldwide to objectively analyse the crumb structure of bread. It is used as a quality control tool and to assess newly developed products. The C-Cell was used with software version 2.0 (Calibre Control International, Warrington, UK). Individual slices of bread were placed onto a black tray in the instrument and illuminated to contrast the cells and the cell walls. An image of the bread was then acquired and analysed by the C-Cell. Alternative images were produced from the original image, on parameters that determine bread’s quality. These details include
the brightness, shape and coarseness of the bread, and the shape, size, number, and orientation of cells within the bread. The number of cells, their shape and size, determines the structure of the bread, and therefore its eating characteristics. How these cells are then distributed affects the coarseness of bread, as well as crumb brightness, which is dependent upon cell orientation and crumb structure.

The advantages of the C-Cell are its ease and speed of use. Results can be obtained within seconds. The digital imaging system also means consistent results are obtained. The disadvantages are that the resolution of the C-Cell is no greater than that of the human eye and the cell characteristics provided are based on a 2D image.

3.3.6 Texture Analyser

The texture analyser is used throughout the food industry to measure the sensory qualities of food using a range of probes and attachments. These probes are attached to either the testing bed or the arm of the texture analyser, depending on the test being conducted. The texture analyser is illustrated in Figure 3.6.

---

Figure 3.6 Texture analyser. A Stable Microsystems instrument used within the food industry to quantify the texture and physical properties of products. A range of attachments to the arm and testing bed can be used depending on the property being measured.
The texture analyser is used to compare the characteristics of one product to another and to determine quantitatively the customer’s product perception. A number of characteristics can be quantified with the texture analyser. These include the elasticity of a material, its cohesiveness, and firmness. Being mechanical, consistency and non-subjectiveness make it advantageous over human judgements. The product to be analysed is placed on the testing bed or on an attachment on the testing bed, and the test parameters controlled from Exponent T.A. software (Stable Microsystems, Surrey, UK) on a connected computer. The texture analyser works by collecting data on the force, distance and time for an action such as lowering the probe to penetrate the product, before returning to its initial position. This data can be collected at a rate of up to 500 points a second, thus providing detailed information.

3.4 Analytical techniques used
There are several analytical techniques used throughout the course of this PhD. These techniques are described within Section 3.4 and referred to in later sections of this thesis.

3.4.1 Determining flour water absorption in the Farinograph
The flour water absorption was used to determine the optimal quantity of water required by the flour to form a dough and assess the mixing pattern of different sugar content doughs. The value of the flour water absorption obtained for each flour was used to determine the quantity of water used to make up doughs from this flour. Water absorptions of flours were determined according to AACC method 54-21.01.

3.4.2 Determining flour water absorption on the Micro-Dough Lab
As mentioned in Section 3.4.1, flour water absorption is used to determine the optimal quantity of water required by the flour to form a dough. The Micro-Dough Lab was used in addition to the Farinograph to obtain another measurement of water absorption from different equipment. This allowed the mean of the two values to be taken, providing a more accurate value.

The method used to determine the water absorption of the flour using the Micro-Dough Lab was as follows: 4 g of flour was loaded into the Micro-Dough Lab mixing bowl. An estimate of the flour’s water absorption value was input into the Micro-Dough Lab software. The Micro-Dough Lab heated up and added a quantity of water, based on the estimate, to the flour through a syringe. The flour and water were mixed, as the Micro-Dough Lab software recorded the torque, and utilised the values to derive the flour’s water absorption. This was repeated until the estimated water absorption value matched that of the quantity of water required.

3.4.3 Measuring dough density
Dough density depends upon the density of the dough matrix and the quantity of gas in the cells. The double cup system described below was used to measure the dough density, due to it being a cheap, convenient and established method, having been used by numerous
researchers for more than a decade (Campbell et al., 2001; Martin et al., 2004a; Chin and Campbell, 2005a; Ktenioudaki et al., 2009).

As the cell volume distribution varies throughout the dough, to obtain a representative density value, six samples were taken from each dough mix. The dough pieces, each approximately 10 g in weight were carefully cut from different regions of the dough ball and the density of each piece individually determined using the double cup system shown in Figure 3.7(a). The mean of the six values was then taken as the density of the dough.

The double cup system consists of the double cup in Figure 3.7(b), held up on a stand, so the top cup is exposed to air and the bottom cup immersed in silicone oil in a jacketed beaker. Note the slightly larger and gently rounded anti-float cup over the bottom cup. This allows the weight of materials less dense than silicone oil to be taken and avoids exerting strain on the sample. The weight was read from the mass balance the beaker is sat on.

Figure 3.7 (a) Double cup system, used to measure the dough density through calculations involving weight measurements of dough in a fluid and air (b) Double cup

The beaker was connected to a water bath and filled with silicone oil. Silicone oil was chosen as it is safe, non-wetting and non-dissolving of the dough. The water bath was set at 32.5°C. This ensured that by the time the water reached the jacketed beaker, its temperature was 30°C. 30°C is the temperature of industrially made bread after mixing, and also the temperature of the dough (±2°C) following mixing. This desired temperature was achieved through adjusting the temperature of the water used in dough formulations using ice.

The dough samples were weighed on the top cup in air and then on the bottom cup in silicone oil and its weight in both recorded. Using the weight of the dough in air, in silicone oil and the density of silicone oil at 30°C, the density of the dough was determined using Equation (9),

\[ \rho_{dough} = \frac{W_{\text{air}} \times W_{\text{oil}}}{W_{\text{air}} + W_{\text{oil}}} \quad (9) \]
where $\rho_{\text{dough}}$ (g cm$^{-3}$) and $\rho_{\text{oil}}$ (g cm$^{-3}$) are the density of the dough and oil respectively and $w_{\text{air}}$ (g) and $w_{\text{oil}}$ (g) are the weight of the dough in air and oil respectively. This method is referred to throughout this thesis as the double cup method.

3.4.4 Determining the gas free dough density

At the macroscopic level bread dough consists of two components: the gas which makes up the cells and the dough matrix. The gas free dough density ($\rho_{gf}$) is the density of the dough matrix. To determine the gas free dough density, six doughs were made in the Tweedy 1 mixer, through mixing at a range of pressures for 3 minutes, and their densities determined using the double cup method. The pressures used were: 0.25 bara, 0.5 bara, 1 bara, 1.5 bara, 2 bara and 2.5 bara. Mixing at these pressures was conducted in a random order to minimise run order artefacts. Compressed air was used to achieve the desired pressures and a flow rate of approximately 10-15 L min$^{-1}$ was maintained throughout mixing. Prior to beginning each mixing session the headspace atmosphere was first obtained and held for 30 s by flushing with compressed air or a vacuum line, depending on the mixing pressure.

When the density of the mixed dough at the different pressures was obtained, a graph was produced with density plotted against pressure. The gas free dough density was obtained by extrapolating to a pressure of zero bara.

3.4.5 Determining dough voidage

The dough voidage is the quantity of gas present within the dough. When mixing under pressure or vacuum the dough voidage will differ if the dough is inside the mixer, and on removal of the dough from the mixer, due to differences in pressure; dough is subjected to a pressure step change on removal from the mixer to atmospheric pressure. The voidage measurement outside the mixer is generally used, as further breadmaking processes occur at atmospheric pressure. The voidage outside the mixer is determined from the dough’s density measured outside the mixer and the gas free dough density. The gas free dough density was obtained using the methodology in Section 3.4.4. These values were then input into Equation (5) in Section 2.2.4.

When dough undergoes a sudden pressure change, both its voidage at the time inside and outside the mixer are used to quantify the voidage change caused by the pressure change. When pressure changes occur inside the mixer, this affects aeration of the dough; a sudden pressure change causes a sudden change in dough voidage. To quantify the change, the voidage inside the mixer is taken as the new voidage, and for voidage measurements after this time, the voidage inside the mixer is used. In this thesis, all pressure change experimental doughs begin from atmospheric pressure mixing. From beginning mixing at atmospheric pressure, the relationship between the voidage inside and outside of the mixer is given in Equation (6) in Section 2.2.4.
3.4.6 Specific volume and density of bread
The specific volume is the ratio of the volume of a material to its mass. To determine the specific volume and density of bread AACC Method 10-05.01 was used.

These were determined in triplicate using the seed displacement method and Equations (10) and (11).

\[
\text{Specific Volume} = \frac{V}{w}
\] (10)

The density of bread was calculated through Equation (4),

\[
\rho = \frac{W}{V}
\] (11)

where \( \rho \) is the density (g cm\(^{-3}\)) and \( V \) (cm\(^3\)) and \( w \) (g) are the volume and mass of the bread respectively.

3.5 Flour and dough characterisation
Flour characterisation is carried out to compare the two main flours used throughout this thesis, to ensure different flours have been chosen, thus allowing generalisation of results. In this section, flours \( a \) and \( c \) are characterised in terms of their water absorption and gas free dough density. The gas free dough density was also measured in doughs made from flours \( b \) and \( d \). These results are presented in Section 4.3, as the doughs were made containing different quantities of sugar for the purpose of comparing the gas free dough density of different sugar content doughs.

3.5.1 Flour water absorption
Ideally the optimal quantity of water required to form a dough would have been determined for all five flours used. However, due to no access to the required equipment, the water absorption values were only obtained for flours \( a \) and \( c \). For flours \( b \) and \( d \), identical values to flours \( a \) and \( c \) were used, and for flour \( e \), the recommended water content according to the packaging was used.

The water absorption of flour depends upon its protein content and the amount and type of grinding performed during milling of the wheat (Campbell et al., 2012), which dictates the starch damage. Damaged starch absorbs twice its own weight of water, in comparison to undamaged starch, which absorbs approximately 40% its weight in water (Cauvain and Young, 2004).

High protein content flours generally absorb more water. The protein content and water absorption values of 3 of the flours used and 75 flours derived from lists of experimentally assessed flours in six publications (Zounis and Quail, 1997; Räsänen et al., 1997; Chin and Campbell, 2005; Bellido et al., 2006; Dobraszczyk and Salmanowicz, 2008; Sroan et al., 2009;) are presented in Figure 3.8. A range of water absorption values for flours with different protein contents is illustrated. It does not show a correlation between protein
content and water absorption, suggesting that starch damage is a larger factor in influencing water absorption. Different protein content flours were used for experiments within this thesis regardless, as flours as sold on the basis of their protein content. For the three flours used, a positive correlation can be seen between protein content and water absorption.

With a large range of water absorption for flours containing different amounts of protein, and no trend seen in Figure 3.8, this is why with no equipment available for measuring water absorption, the same water absorption values were used for flours b and d, to the plain and strong flours, a and c, rather than estimating based on protein content.

![Figure 3.8 Protein content vs. water absorption values of flours. 78 values flours are illustrated, including 75 flours in literature and 3 flours used throughout this thesis.](image)

The water absorption values for flours a and c obtained using the Farinograph and Micro-Dough Lab, and the water absorption values consequently used for experiments, are displayed in Table 3.3. The methodology is detailed in Sections 3.4.1 and 3.4.2.

**Table 3.3 Flours and their corresponding water absorption values. These were determined from the Micro-Dough Lab, Farinograph and packaging. The final column gives the water absorption value used in experiments within this thesis, based on these measurements**

<table>
<thead>
<tr>
<th>Flour (protein content)</th>
<th>Farinograph (%)</th>
<th>Micro-Dough Lab (%)</th>
<th>Water absorption value used (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flour a (10.1%)</td>
<td>55±0.5</td>
<td>57.5±0.3</td>
<td>56.25</td>
</tr>
<tr>
<td>Flour b (10.4%)</td>
<td>-</td>
<td>-</td>
<td>56.25</td>
</tr>
<tr>
<td>Flour c (11%)</td>
<td>58±0.5</td>
<td>58.0±0.3</td>
<td>58.00</td>
</tr>
<tr>
<td>Flour d (11.5%)</td>
<td>-</td>
<td>-</td>
<td>58.00</td>
</tr>
<tr>
<td>Flour e (15%)</td>
<td>-</td>
<td>-</td>
<td>66.60</td>
</tr>
</tbody>
</table>

Both methods give a lower water absorption in flour a than c, where in flour c a water absorption value of 58% was found via both methods. When taking into account the error,
however, the values given by the Micro-Dough Lab suggest the same water absorption between the two flours.

3.5.2 Mixing at constant pressure: gas free dough density
The methodology described in Section 3.4.4, was used to determine the gas free dough density. The gas free dough density was obtained for flours a and c, and a number of doughs throughout this thesis. This section only details the gas free dough density of flours a and c, as the gas free dough densities of flours b, d and e were not needed. The gas free dough density of the other doughs investigated are detailed in the appropriate chapter. Tables 3.4 and 3.5 list the formulation used to determine the gas free dough density of flours a and c respectively.

Table 3.4 Formulation used to determine the gas free dough density of a dough made from flour a

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>Quantity (g)</th>
<th>Quantity (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flour a (10.1%)</td>
<td>400</td>
<td>64.0</td>
</tr>
<tr>
<td>Water</td>
<td>225</td>
<td>36.0</td>
</tr>
</tbody>
</table>

Table 3.5 Formulation used to determine the gas free dough density of a dough made from flour c

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>Quantity (g)</th>
<th>Quantity (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flour c (11%)</td>
<td>400</td>
<td>63.3</td>
</tr>
<tr>
<td>Water</td>
<td>232</td>
<td>36.7</td>
</tr>
</tbody>
</table>

Figure 3.9 shows the dough density of the two doughs over a range of pressures. It shows the same gas free dough density to five significant figures, 1.2603 g cm$^{-3}$, for doughs made using only flour and water.

Figure 3.9 Density of doughs made from flour a and c, following mixing at a range of headspace pressures for 180 s. The data points are the mean of six measurements from an identical batch of dough. Error bars have been omitted as they are smaller than the markers used. Linear regression trend lines are shown.
Figure 3.10 compares the gas free dough density of the two flours to those obtained from publications where they have been experimentally derived using the same technique (Chin et al., 2004; Martin et al., 2004a; Martin et al., 2004b). For the four flours from literature, as the protein content increases, the gas free dough density decreases. Although, the flours used do not lie on the line of best fit, considering the small number of literature values used, the gas free dough density obtained are reasonable for the protein content of the flours.

Figure 3.10 Gas free dough density of flours from literature and flours a and c.

3.5.3 Dough voidage
The methodology used in Section 3.4.5, was used to determine the voidage of a number of doughs throughout this thesis, including those of flours a and c, formulated in Tables 3.4 and 3.5 respectively. Table 3.6 shows the values used to calculate the dough voidage, and the dough voidage for both flours.

Table 3.6 Characterisation of flours a and c

<table>
<thead>
<tr>
<th></th>
<th>$\rho_{\text{dough}}$ (g cm$^{-3}$)</th>
<th>$\rho_{\text{gf}}$ (g cm$^{-3}$)</th>
<th>$\Phi$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flour a (10.1%)</td>
<td>1.1697</td>
<td>1.2603</td>
<td>0.0675</td>
</tr>
<tr>
<td>Flour c (11%)</td>
<td>1.1752</td>
<td>1.2603</td>
<td>0.0719</td>
</tr>
</tbody>
</table>

The dough produced from flour c has a greater voidage than the dough produced from flour a. These dough voidages have not been compared to literature values, as there are a number of variables such as the mixer blade, speed, pressure and mixing time, which affect the dough voidage, thus preventing direct comparisons from being made.

3.5.4 Characterisation summary
Characterisation of flours a and c found both flours to have the same gas free dough density. Flour a had a lower water absorption value and a lower voidage. This voidage difference shows these flours to behave differently and therefore deemed them suitable for drawing generalisations for wheat flour doughs.
3.6 Additional methodologies

In Chapter 4 of this thesis, bread is made using both a breadmaker and a Tweedy mixer to illustrate the effect of sugar on different quality parameters in baked bread. The two breadmaking methods used described in this Section, with details of the methodology used for X-ray CT in Chapter 6.

3.6.1 Breadmaking

This section describes the breadmaking techniques used in Section 4.11 to make breadmaker loaves and Tweedy loaves.

For loaves made in the breadmaker, water at 35±2°C was first poured into the breadmaker tin. The yeast was then crumbled up and the vegetable fat broken into small pieces and these alongside the remaining ingredients put into the breadmaker baking tin, and the ingredients levelled off for more efficient mixing. The bread was made using the medium setting for basic bread. As soon as the bread was cooked, it was removed from the breadmaker and put onto a cooling rack. Three loaves of each recipe formulation were made.

For bread made in the Tweedy 1 mixer, the ingredients were mixed for three minutes at a speed of 77 rad s⁻¹, with the water at 35±2°C. Following mixing, the dough was put into a Pyrex bowl. All exposed surfaces of the dough were greased with vegetable fat and the bowl covered with a damp tea towel to prevent the dough from drying out. It was proved for 60 minutes at 39°C in Gallenkamp oven 300 plus series (Weiss Technik UK, Leicestershire, UK) over a bowl of water, which was present to create a humid atmosphere. The dough was then knocked back (kneaded briefly) and 500±1 g of the dough was shaped and put into a greased 1 lb baking tin, covered with a damp tea towel and proved again for 25 minutes over a bowl of water. Finally, the dough was baked in a Simon Rotary Test Baking Oven (Henry Simon LTD, Stockport, UK) for 25 minutes at 450°F. Immediately following baking, the bread was turned out onto a cooling rack. Three loaves of each recipe formulation were made.

3.6.2 X-ray imaging

X-ray CT is used in Chapter 6, for three separate experiments. Bread dough samples were imaged following mixing for a specific length of time. In each of the experiments, there is some variation amongst the X-ray settings and also the processing of the data, in order to obtain the appropriate data at the optimum settings for each experiment.

In a test to assess if the dough voidage was altered in preparing it for X-ray CT, a dough was made up and its density measured from six samples using the double cup method. Another six samples from the dough were then put into the tubing as done for the X-ray imaging experiments and their densities measured using the double cup method. By subtracting the density of the tubing from the measured densities, the density of the prepared dough could then be calculated. Assessment of sample preparation altering the dough voidage found the sample preparation method to be non-destructive of the dough, giving accurate density measurements.
For the investigations in Section 6.2 and 6.3, following mixing, a dough piece approximately 10 g in weight was cut from the dough and its voidage determined from the double cup method. The voidage was later compared to the voidage obtained via X-ray CT to determine if the dough preparation procedures involved in X-ray CT altered its voidage.

Dough pieces approximately 1.3 cm$^3$ in volume was then cut from the batch of the dough and imaged in tubing. A sufficient dough quantity was inserted in the tube to ensure it would not slump during imaging. Following preparation, the dough samples were placed on the Nikon Metris 225/320 kV CT system rotation stage and imaged in a customised bay. Full details of the sample set-up are given in the corresponding sections of this thesis.

Following X-ray imaging, the images were reconstructed using a reconstruction algorithm on the software Nikon Metris CTPro 3D (Metris XT 2.2, version 2.2.4365.28608). The algorithm was devised by staff at the Manchester X-ray Imaging Facility at the University of Manchester. The Manchester X-ray imaging facility is a world renowned UK leading X-ray imaging facility which collaborates with the UK synchrotron facility, Diamond Light Source in Oxfordshire.

The reconstructed images were then analysed and processed in Avizo Fire. Sections 6.2 and 6.3 were processed in Avizo Fire 6.3.1 and Section 6.4 in Avizo Fire 7.1 (Visualization Sciences Group, Bordeaux, France). For the doughs presented in Sections 6.2 and 6.3, a volume was cropped from the reconstructed data, resulting in a sample size of approximately 1.2 cm$^3$. The dough towards the bottom of the tube was not analysed due to the possibility of compression altering the cellular structure. The edges of the dough sample also were not used, to avoid edge artefacts. The dough matrix and cells were segmented based on greyscale values. Once segmented, material statistics was run to quantify the volume of each material present and 3D volume in quantification was used to obtain the number and volumes of cells within the samples. The cell volumes were processed in Microsoft Office Excel and a 5 voxel cell diameter size, equating to a sphere size of 65 cubic voxels used as a cut-off point to ensure cells were identified with high confidence and false identifications (from noise and artefacts) were not made. This cut-off size is based on cell sizes in orthoslices and ESEM images in Section 4.9. Note the smallest cells at this stage of breadmaking are the least important in the breadmaking process due to their small voidage contribution and their high internal pressure limiting growth during proving and baking (Shah et al., 1998). The cell volume distribution, cell number density, mean cell volume, cumulative cell percentage and cumulative voidage per unit volume were then calculated for the doughs. Cell number density is used to take into account the differences in sample sizes. The distribution of cell sizes is also presented to enable any cells that skew the data to be identified.

The procedure used for the 3D scan in Section 6.4 differ more to those of the 2D scans in Section 6.2 and 6.3 and are explained in Section 6.4.1 for ease of the reader.
Chapter 4 - Assessment of the effect of sugar on dough properties during breadmaking processes

4.1 Introduction

The addition of sugar to bakery products increases their appeal by increasing the indulgence factor of the product. Currently the widely available sweetened breads have their sweetness boosted by the addition of glazes, fillings and dried fruit. It is thought that one reason the addition of pure sugar as a sweetener is limited is it creates a sticky product, which causes processing difficulties in industry. Another is pressure from the Food Standards Agency to reduce cases of coronary heart disease, obesity and dental decay resulting from high fat and sugar diets (Sheehy et al., 2008). This Chapter uses ten short experimental sections to investigate the effects of sugar during the breadmaking process to better understand how sugar affects the phenomena that contribute to the creation of bread structure at different stages. Section 4.2 uses a simplified dough comprising starch and water, with the addition of different quantities of sugar to assess changes in the mixture’s properties. In Section 4.3 different sugar content doughs are characterised in terms of their gas free dough density and voidage. Section 4.4 assesses how the properties of the dough change during mixing through torque measurements in a Farinograph. Section 4.5 continues from Section 4.4 in assessing how the properties of the dough change during mixing through torque measurements in a Tweedy 1 mixer. In Section 4.6 the effect of mixing speed on aeration of different sugar content doughs is assessed. Section 4.7 assesses the stickiness of different sugar content doughs using a texture analyser. As the expansion capacity of the dough is key to loaf volume, the extensibility of different sugar content doughs is assessed in Section 4.8, again using a texture analyser. In Section 4.9, dough microstructures from 0% and 15% sugar dough are compared using micrographs obtained via environmental scanning electron microscopy (ESEM). The effect of sugar during proving is assessed via dynamic density measurements of yeasted doughs of varying sugar contents in Section 4.10. Finally, in Section 4.11, the effect of sugar on baked bread is assessed, through a number of tests on quality parameters. The findings from these ten sections are then summarised in Section 4.12. Further work is also carried out within this thesis on the effect of sugar during the breadmaking process. This work includes an extensive investigation into the aeration of sugared doughs via a population balance model in Chapter 5, and X-ray CT of sugared bread doughs in Section 6.2. These investigations have not been located in this chapter as they are more extensive than these preliminary studies.

4.2 Interaction of sugar and starch-water mixture

The main component of flour is starch and therefore the interaction of sugar with starch-water mixture was investigated as a simplified dough system to determine if sugar has an effect on the behaviour of bread doughs. This was done initially to avoid wasting time on the design and execution of more elaborate experiments if this experiment found sugar not to
have an effect. It was expected that sugar would affect the simplified dough system and that the observed effects would have been greater with more sugar, due to its hygroscopic nature. Higher viscosity doughs were also expected with larger quantities of sugar due to the increased weight of solids to liquids.

The source of the starch used in the simplified dough system was cornflour, as its protein content is negligible, and therefore any differences observed amongst the different sugar content mixtures are likely to be reflected in wheat flour doughs. With starch being the main component of flour, the effect of sugar on protein was not investigated.

4.2.1 Method: Interaction of sugar and starch-water mixture
To simplify the effect of sugar in bread dough, investigations were carried out in a simplified dough system, comprising cornflour and water. 130 g of water was added to 180 g of cornflour and between 0 to 27 g of sugar in a bowl. This equated to between 0% and 8% sugar in proportion to all the ingredients. This water content was chosen through trial and error, and was the quantity of water in the absence of sugar that enabled display of the typical behaviour associated with starch slurries. Four doughs were made, each of different sugar concentrations. These sugar concentrations were 0%, 5%, 10% and 15% of the flour weight, all of which were completely soluble in water. 15% sugar was used as the extreme as it is more than the quantities commonly used for the industrial production of sweetened bread. 0% sugar was used as a control, and 5% and 10% were used as in-between values. By using these in-between values it was possible to determine if any effects observed with sugar were gradual or sudden with the addition of a specific quantity of sugar.

Following production of each batch, the mix was prodded, stirred at different speeds and the bowl tilted, as observations of the mixtures response were noted.

4.2.1 Results and discussion: Interaction of sugar and starch-water mixture
As the sugar content of the dough increased from 0% to 5% to 10% to 15%, the properties of the cornflour mixture changed in proportion to this. At 0% sugar, the mixture was thixotropic. On application of sudden force to the mixture, it resembled a solid by resisting flow and displaying an elastic response. It was not possible to stir the mixture and following stirring attempts, none of the mixture stuck to the stirrer. However, application of more gradual force to the fluid enabled penetration of the stirrer and resulted in a light coating on it. In addition, on tilting the bowl, it flowed as a viscous fluid.

At the other extreme, the mixture containing 15% sugar flowed readily as a low viscosity fluid without obvious thixotropic or yield stress properties. The mixture flowed when tilting the bowl, could be stirred and did not display an elastic response under application of sudden or gradual force. Following application of force with the stirrer, it became covered in the mixture.

The properties of the mixture at 5% and 10% sugar lay between these two extremes. This suggests that the influence of sugar on dough is in proportion with the quantity of sugar.
present, rather than suddenly at the presence of a set amount of sugar in the dough. The behaviour of the cornflour and sugar mixtures varied between that of solids and liquids. In solids the molecules are closely packed and held in a fixed position thus they cannot move between each other. On the other hand, the molecules in liquids are less tightly packed, enabling some movement between each other. Cornflour is also known as corn starch. Starch consists of two components: amylose and amylopectin. These are long chains of sugar, which adhere strongly to another and cannot move past another, thus starch is a solid. When water is added to cornflour, it lubricates the starch chains and enables them to slide past another, behaving like a liquid. On applying force to the mixture, water is squeezed out from the chains, and they revert to a solid (Crane, 2012). When the force is released, water seeps between the chains, enabling them to move again.

Observations of the mixtures showed that starch does not dissolve in water at ambient temperatures. Sucrose, in the quantities added, was fully soluble at room temperatures and therefore as well as acting as a lubricant, increased the volume, though not the quantity of fluid present. Sugar would have also lowered the water activity of the mixture (Pennington and Baker, 1990). The water activity of a wheat flour bread dough is approximately 0.95 and decreases significantly on addition of sugar. For example, a high sugar product such as jam has a water activity of 0.75-0.8 (Pennington and Baker, 1990). The largest component of wheat flour is starch, and although the proportions of the two glucans from which the starch is comprised will vary between corn starch and wheat starch, it is expected that the water activity of the cornflour mixture is similar to that of bread dough. It is expected therefore that on addition of sugar, the mixture would become less fluid, as opposed to more fluid, as observed. It is known that when mixing the water and starch, a proportion of the starch granules absorb water and swell (Belitz et al., 2004). The addition of sugar interferes with the absorbance of water by starch, therefore the starch molecules in the sugar containing mixtures will be smaller. The most likely explanation of the observed behaviour is that the water activity of the starch mixture is reduced on addition of sugar resulting in the reduced swelling of the suspended starch chains, which increases flow in the mixture. Increased flow is generally associated with lower viscosity solutions. Kim and Walker (1992) found the larger the starch particles the higher the viscosity of the solution.

Due to the similarities between a starch-water mixture and wheat flour dough, and the observations made in this section, it is expected that the physical properties of a wheat flour dough will change on addition of sugar. The results suggest that despite the increase in the ratio of weight of solids to weight of liquids, the viscosity of wheat flour dough will be reduced on addition of sugar due to less swelling of starch particles. As dough viscosity is known to be linked to dough rheology and aeration, sugar is expected to affect these parameters.
4.3 Gas free dough density and voidage of different sugar content doughs

Section 2.2 discussed the insight offered by measuring dough voidage throughout the breadmaking process. However, calculation of this requires both knowledge of the dough density and gas free dough density. The double cup system has been extensively used for accurate and precise dough density measurements, and by extrapolating density pressure plots to zero pressure the dough gas free density can be obtained (Campbell et al., 1993). This section presents data from a study of dough sugar content and dough density over controlled mixer headspace pressures. Voidage and gas free dough density measurements are common in literature investigating the aeration of bread dough, and simple and cheap methods of characterising the dough. By varying the sugar content of the doughs in the following experiment and in incrementing quantities, the overall effect of sugar on bread dough aeration could be observed and how different quantities of sugar affect bread dough aeration could also be determined. It was expected that doughs would become less aerated on increasing the sugar content due to a greater proportion of solids to liquids meaning formation of a more viscous dough, making entrainment of gas through enveloping pockets of gas more difficult. The hygroscopic nature of sugar would also mean there is less water available for protein hydration and therefore dough development, required for retaining cells within the dough.

4.3.1 Method: Gas free dough density and voidage of different sugar content doughs

The dough density and voidage of different sugar content doughs were determined and compared. Doughs were made containing between 0% and 15% sugar and either strong or weak flour. Their formulations are provided in Tables 4.1 and 4.2. Their gas free dough density and voidage were determined according to the methods in Section 3.4.4 and 3.4.5 respectively.

Table 4.1 Dough formulations used to assess the voidage and gas free dough density of different sugar content strong flour doughs.

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>0% sugar</th>
<th>1.5% sugar</th>
<th>5% sugar</th>
<th>10% sugar</th>
<th>15% sugar</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>232.0 / 35.7</td>
<td>232.0 / 35.4</td>
<td>232.0 / 34.7</td>
<td>232.0 / 33.7</td>
<td>232.0 / 32.7</td>
</tr>
<tr>
<td>Sugar</td>
<td>0.0 / 0.00</td>
<td>6.0 / 0.916</td>
<td>20.0 / 2.99</td>
<td>40.0 / 5.80</td>
<td>60.0 / 8.46</td>
</tr>
<tr>
<td>Salt</td>
<td>7.2 / 1.10</td>
<td>7.2 / 1.10</td>
<td>7.2 / 1.08</td>
<td>7.2 / 1.04</td>
<td>7.2 / 1.02</td>
</tr>
<tr>
<td>Flour c (11%)</td>
<td>400.0 / 61.6</td>
<td>400.0 / 61.1</td>
<td>400.0 / 59.8</td>
<td>400.0 / 58.0</td>
<td>400.0 / 56.4</td>
</tr>
<tr>
<td>Fresh yeast</td>
<td>10.0 / 1.54</td>
<td>10.0 / 1.53</td>
<td>10.0 / 1.49</td>
<td>10.0 / 1.45</td>
<td>10.0 / 1.41</td>
</tr>
</tbody>
</table>
Table 4.2 Dough formulations used to assess the voidage and gas free dough density of different sugar content weak flour doughs

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>0% sugar</th>
<th>1.5% sugar</th>
<th>5% sugar</th>
<th>10% sugar</th>
<th>15% sugar</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>225.0 / 35.0</td>
<td>225.0 / 34.7</td>
<td>225.0 / 34.0</td>
<td>225.0 / 33.0</td>
<td>225.0 / 32.0</td>
</tr>
<tr>
<td>Sugar</td>
<td>0.0 / 0.00</td>
<td>6.0 / 0.926</td>
<td>20.0 / 3.02</td>
<td>40.0 / 5.86</td>
<td>60.0 / 8.54</td>
</tr>
<tr>
<td>Salt</td>
<td>7.2 / 1.12</td>
<td>7.2 / 1.11</td>
<td>7.2 / 1.09</td>
<td>7.2 / 1.06</td>
<td>7.2 / 1.03</td>
</tr>
<tr>
<td><em>Flour a</em> (10.1%)</td>
<td>400.0 / 62.2</td>
<td>400.0 / 61.7</td>
<td>400.0 / 60.4</td>
<td>400.0 / 58.6</td>
<td>400.0 / 57.0</td>
</tr>
<tr>
<td>Fresh yeast</td>
<td>10.0 / 1.56</td>
<td>10.0 / 1.54</td>
<td>10.0 / 1.51</td>
<td>10.0 / 1.47</td>
<td>10.0 / 1.42</td>
</tr>
</tbody>
</table>

4.3.2 Results: Gas free dough density and voidage of different sugar content doughs

Figures 4.1 and 4.2 illustrate the effect of sugar on the gas free dough density in strong and weak flour doughs, respectively. Each point is the mean of six measurements from the same dough; the standard deviations are typically smaller than the symbols so are not shown. The density of doughs (once the doughs have been removed from the mixer) made from both flours appears to vary linearly with headspace pressure.

The linear trends were extrapolated back to zero pressure by linear regression to find the gas free dough densities for each sugar content dough. The results show an increase in the gas free dough density as the sugar content increases amongst doughs produced from both flour types. This is most likely a result of the greater proportion of solids in the doughs containing more sugar. In addition, the gradient of the linear regression lines decreases as the dough sugar contents increases. This corresponds to denser doughs with lower voidage, as the sugar content increases.
Figure 4.1 Density of different sugar content strong flour doughs following 180 s of mixing at different mixer headspace pressures. The data points illustrated are the mean of six measurements. Error bars have been omitted as they are smaller than the symbols used. Linear regression lines are shown.

Figure 4.2 Density of different sugar content weak flour doughs following 180 s of mixing at different mixer headspace pressures. The data points illustrated are the mean of six measurements. Error bars have been omitted as they are smaller than the symbols used. Linear regression lines are shown.
Figures 4.3 and 4.4 illustrate the change in voidage in different sugar content doughs over mixer headspace pressures ranging from 0.25 bara to 2.5 bara. Dough voidage at different sugar contents is similar between the two flours at positive mixing pressures. When mixing under partial vacuum however, the strong flour doughs had a lower voidage. In Figure 4.3 voidage appears to be linearly proportional to the sugar content, with a decrease in voidage as sugar content increases at higher pressures and less of a relationship between the two at lower pressures, as expected with there being less gas available for entrainment during mixing. For the weak flour dough in Figure 4.4 dough voidage increases as the sugar content increases from 0% to 5%, and decreases at increasing sugar contents. These results suggest that sugar affects dough aeration during mixing and it is possible that a small quantity of sugar produces doughs of a lower voidage. Entrainment and disentrainment of gas during mixing is investigated in Chapter 5. In addition, the results show that the higher the mixer headspace pressure, the greater the voidage across doughs made from both flours and of all sugar content investigated. This is due to more air entering the mixer headspace and therefore the dough when mixing at higher pressures.

![Figure 4.3 Voidage of different sugar content strong flour doughs, following 180 s of mixing at mixer headspace pressures ranging from 0.25 bara to 2.5 bara.](image-url)
The results show that dough density increases on increasing the sugar content, due to both a decrease in voidage and an increase in the gas free dough density. The increased sugar concentration in the free water phase, and therefore increased solution density, will lead to the higher gas free dough density. However, the decrease in voidage with sugar content must be due to the aeration processes during mixing. It remains to be seen later in the thesis how this decrease in initial dough voidage arises in terms of the relative rates of entrainment and disentrainment of gas during mixing, and how it relates to sweetened bakery products.

4.4 Mixing different sugar content doughs in the Farinograph

The Farinograph is a specialised bakery tool used worldwide to measure flour properties and processing characteristics from torque measurements. In this section, the Farinograph curves (Farinograms) produced from four different sugar content doughs are used to compare their mixing behaviour and development. The torque at different times and significant stages during mixing are investigated to determine dough strength, and the times to reach significant stages during mixing to indicate the required mixing time. It was expected that increasing the sugar content would increase the quantity of solids to liquids present and therefore form a mass quicker, but require more time to reach development due to the lower availability of water for gluten hydration. It was expected that the torque at the end of mixing would increase with sugar, due to more resistance from the larger quantities of solids present.

4.4.1 Method: Mixing different sugar content doughs in the Farinograph

The mixing characteristics of dough containing different sugar quantities were determined through mixing different sugar content doughs in the Farinograph following the standard AACC method (54-21). In Figure 4.5, a typical Farinogram with key areas of interest highlighted is presented. The Farinogram was created by an analogue torque measurement using a pivoted marker pen on a moving reel of graph paper for a standard mixing time. The paper was
aligned such that mixing starts at time zero on the recording. The y-axis is curved due to the pivoted recorder and is in Brabender Units (B.U.) which is proportional to the mixer drive shaft torque.

![Figure 4.5 A typical Farinogram with significant features annotated](image)

The dough development time is the time from the start of mixing, at 0 minutes, to the point on the curve immediately prior to the first signs of weakening. If two peaks are observed, then the second peak is used to determine the development time, as the first peak is usually caused by flour hydration. The Farinograph determines the dough development time accurate to 30 s. This is not very accurate considering the length of time required for dough development, but was adequate for determining if a difference exists between the different sugar content doughs investigated.

The arrival time indicates the point of flour hydration, and the departure time is the point at which the dough begins to break down and become overdeveloped. The difference between the arrival and departure time is termed stability time. It is the time that the dough maintains its maximum consistency and is indicative of dough strength. The Farinograph determines stability time accurate to 60 s.

Eight doughs were mixed in the Farinograph and Farinograms obtained for each. Their formulations are presented in Tables 4.3 and 4.4.
Table 4.3  Strong flour dough formulations used in the Farinograph

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>0% sugar</th>
<th>5% sugar</th>
<th>10% sugar</th>
<th>15% sugar</th>
</tr>
</thead>
<tbody>
<tr>
<td><em>Flour c</em> (11%)</td>
<td>300.0 / 5.93</td>
<td>300.0 / 57.6</td>
<td>300.0 / 56.0</td>
<td>300.0 / 54.5</td>
</tr>
<tr>
<td>Vegetable fat</td>
<td>15.0 / 2.97</td>
<td>15.0 / 2.88</td>
<td>15.0 / 2.80</td>
<td>15.0 / 2.72</td>
</tr>
<tr>
<td>Salt</td>
<td>4.5 / 0.890</td>
<td>4.5 / 0.865</td>
<td>4.5 / 0.840</td>
<td>4.5 / 0.817</td>
</tr>
<tr>
<td>Fresh yeast</td>
<td>6.0 / 1.19</td>
<td>6.0 / 1.15</td>
<td>6.0 / 1.12</td>
<td>6.0 / 1.09</td>
</tr>
<tr>
<td>Ascorbic acid</td>
<td>6.0 / 1.19</td>
<td>6.0 / 1.15</td>
<td>6.0 / 1.12</td>
<td>6.0 / 1.10</td>
</tr>
<tr>
<td>Water</td>
<td>174.0 / 34.4</td>
<td>174.0 / 33.4</td>
<td>174.0 / 32.5</td>
<td>174.0 / 31.6</td>
</tr>
<tr>
<td>Sugar</td>
<td>0.0 / 0.00</td>
<td>15.0 / 2.88</td>
<td>30.0 / 5.60</td>
<td>45.0 / 8.17</td>
</tr>
</tbody>
</table>

Table 4.4  Weak flour dough formulations used in the Farinograph

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>0% sugar</th>
<th>5% sugar</th>
<th>10% sugar</th>
<th>15% sugar</th>
</tr>
</thead>
<tbody>
<tr>
<td><em>Flour a</em> (10.1%)</td>
<td>300.0 / 59.9</td>
<td>300.0 / 58.2</td>
<td>300.0 / 56.6</td>
<td>300.0 / 55.0</td>
</tr>
<tr>
<td>Vegetable fat</td>
<td>15.0 / 3.00</td>
<td>15.0 / 2.91</td>
<td>15.0 / 2.83</td>
<td>15.0 / 2.75</td>
</tr>
<tr>
<td>Salt</td>
<td>4.5 / 0.899</td>
<td>4.5 / 0.873</td>
<td>4.5 / 0.848</td>
<td>4.5 / 0.825</td>
</tr>
<tr>
<td>Fresh yeast</td>
<td>6.0 / 1.20</td>
<td>6.0 / 1.16</td>
<td>6.0 / 1.13</td>
<td>6.0 / 1.10</td>
</tr>
<tr>
<td>Ascorbic acid</td>
<td>6.0 / 1.20</td>
<td>6.0 / 1.16</td>
<td>6.0 / 1.13</td>
<td>6.0 / 1.10</td>
</tr>
<tr>
<td>Water</td>
<td>169.0 / 33.8</td>
<td>169.0 / 32.8</td>
<td>169.0 / 31.9</td>
<td>169.0 / 31.0</td>
</tr>
<tr>
<td>Sugar</td>
<td>0.0 / 0.00</td>
<td>15.0 / 2.91</td>
<td>30.0 / 5.66</td>
<td>45.0 / 8.25</td>
</tr>
</tbody>
</table>

For the formulations in Tables 4.3 and 4.4, the water content was determined using the Farinograph and Micro-Dough Lab. The quantity and proportions of the other ingredients, with the exception of sugar, were those recommended by the Farinograph guide. For this reason, the proportions differ to those used in other formulations within this thesis, but all formulations are within the normal proportion ranges used in industrial bread dough formulations.

4.4.2 Results and discussion: Mixing different sugar content doughs in the Farinograph

In Figures 4.6-4.9, the Farinograms for the strong and weak flour doughs containing 0%, 5%, 10% and 15% sugar are presented respectively. Each experiment was conducted in triplicate and peaks were found to be repeatable to within ±20 Brabender units and ±30 s. Therefore, only single representative Farinograms are presented for each formulation.

In the sugar contents investigated, typical features of a Farinogram were not observed beyond a sugar content of 5%. The Farinograms also differ in their shape, in terms of the number of peaks and the dough consistency amongst the eight doughs investigated. Dough formulations containing 0% and 5% sugar produced one peak and those containing 10% and 15% sugar produced two peaks during mixing in the Farinogram. The times these peaks occurred are presented in Figure 4.10. When taking into account the error of the Farinogram, there are no differences between the times that the peaks occurred in the strong and weak flour
doughs. There is strong correlation between the shape of the curve of the strong and weak flour doughs of the same sugar content, suggesting a similar effect of sugar on doughs produced from both flour types. All doughs drop in torque towards the end of the mixing session and the weak flour doughs generally had a lower torque throughout mixing suggesting they were less viscous than the strong flour doughs. This is illustrated in figure 4.11. Figure 4.11 also illustrates that the doughs containing more sugar had a greater torque during mixing. This is likely to be due to the greater quantity of solids compared to liquids in the dough. The change in dough consistency with time over the mixing session is a result of changes occurring to the dough, through the mixing action of the Farinograph and the increase in dough temperature caused by mixing.

Figure 4.6 illustrates the Farinograms of the two non-sugar containing doughs. The peak representing the dough development time occurs within 0.5 minutes at 520 B.U. There is then a clear dip in the curve, as the dough consistency is reduced. As mixing of this dough continues, the dough consistency continues to drop. The dough consistency drops to 410 B.U. in the strong flour dough and 430 B.U. in the weak flour dough.

Figure 4.7 illustrates the Farinograms of the two doughs containing 5% sugar. The dough development time represented by the peak occurs in the first minute of mixing. In both doughs the torque then decreases for the next 2 minutes to 440 B.U., before increasing again. In the strong flour dough it appears as though the dough consistency drops after 10 minutes of mixing, although this is not clear from the curve. In the weak flour dough, the dough consistency appears to drop after 9 minutes of mixing. Note the difference in peaks between Figures 4.6 and 4.7-4.9, with Figures 4.7-4.9, the dough containing more sugar, having a less defined peak and also a higher torque at the end of the mixing time. This lack of peak and increase in torque with mixing time becomes more defined as the sugar content of the dough increases. These differences amongst the Farinograms illustrate that the presence of sugar affects the mixing of dough and the dough properties. However, the presence of sugar in the dough produces curves where the interpretations of the peaks are unclear because they do not follow convention. The change in torque with sugar is likely to be due to sugar slowing down or limiting the breakdown and hydration of flour components by the mixer blade.

The addition of sugar does not appear to have affected the dough formation time. All doughs, regardless of sugar content, appear to have formed within 1 minute. Although the solid to liquid ratio increases on increasing the sugar content, it is likely that on increasing the sugar content, the mix becomes more sticky, through formation of a syrup, enabling a dough of higher solids content to form within a similar time frame.

Two peaks are exhibited in some of the results. Calderón-Domínguez et al. (2003; 2008) also found two peaks were expressed during the mixing of sweet yeasted doughs in the Farinograph, and believed the second peak, as highlighted in Figure 4.8 (a), to be the one indicative of an optimum developed dough. The authors also found significant differences in the Farinograms of sweet yeasted doughs and non-sugar yeasted doughs. In addition, the
authors measured dough development time on the Farinograph in terms of the time taken to reach a certain dough consistency, and found a longer mixing time for development of sugared doughs. In the doughs investigated in this section, 500 B.U. units was not always reached. It is possible that through significant extension of the mixing time, as found by Calderón-Domínguez et al. (2003) that peak torque would be reached. As the curve shape following extended mixing times varies from that of a typical Farinogram, however, the peak torque in this case may not represent the dough development time.

During mixing of bread dough in industrial bread production, the mixer action will differ from that in the Farinograph, and mixing will most likely occur in a Tweedy mixer. Although the mixing characteristics will differ, the Farinograms produced show differences in behaviour in the different sugar content doughs, which are likely to be reflected across different mixers. Section 4.5 uses torque measurements to assess the effect of different quantities of sugar during mixing in the Tweedy 1 mixer.
**Figure 4.6** (a) 0% sugar strong dough Farinogram (b), 0% sugar weak dough Farinogram

**Figure 4.7** (a) 5% sugar strong dough Farinogram (b), 5% sugar weak flour dough Farinogram

**Figure 4.8** (a) 10% sugar strong dough Farinogram (b), 10% sugar weak dough Farinogram

**Figure 4.9** (a) 15% sugar strong dough Farinogram (b), 15% sugar weak dough Farinogram
Figure 4.10 Mixing time to Farinogram peaks in different sugar content doughs

Figure 4.11 Dough consistency at significant times indicated by the Farinogram

4.5 The effect of sugar on torque during mixing

Section 4.4 in this chapter used torque measurements from doughs mixed in a Farinograph to assess how sugar affects their processing characteristics. The torque can also be measured during mixing in the Tweedy 1 mixer. The Farinograph uses torque measurements to determine dough development time, a key processing characteristic. However, the dough development time differs from one mixer to another, in particular when comparing a standard mixer to a high speed mixer such as the Tweedy, which can mix dough to development to a third to half of the time required in a standard mixer.

The Tweedy 1 has a unique mixing action. Due to the large quantity of air space in the mixer bowl, its rotor action does not reflect changes in dough consistency like the Farinograph. Initially the ingredients are mixed at the bottom of the mixer bowl. As the dough develops, it becomes elastic and moves to the top of the bowl. Here, it interacts with the mixer blade differently and the mixing action changes (Chin and Campbell, 2005).

In this section, the torque is used to assess significant points during mixing in the Tweedy 1 mixer. Particular attention is paid to the peak and end torque, two parameters which can
easily be derived following mixing in the Tweedy 1 mixer. It is expected that torque measurements in the Tweedy mixer will show when a solid mass occurs, and this will be quicker in doughs containing more sugar due to a larger quantity of solids to liquids, and also due to sugar dissolving into the water and creating a sticky syrup that helps bind the ingredients together.

4.5.1 Method: The effect of sugar on torque during mixing

Measuring the torque in the Tweedy 1 is advantageous to measuring it in the Farinograph due to the Tweedy 1 being a scaled down version of the high speed CBP mixers. This makes the results obtained from the Tweedy 1 more relevant to the CBP. The main differences between mixing in the Tweedy 1 and the Farinograph are the faster mixing speed and the large air space in the Tweedy mixer.

The formulations used to assess torque during mixing of different sugar content doughs are given in Table 4.5. Following work on the Farinograph, Calderón-Domínguez (2003) suggested an increase in sugar increased the dough development time. Experimental results in Section 4.3.2 suggest this may not be the case as results did not follow convention and thus were difficult to interpret. Taking this into account, the doughs were mixed for an extended time of 4 minutes, as opposed to the 3 minutes that the majority of doughs throughout this thesis were mixed for, and the torque in the Tweedy 1 monitored over the mixing session.
Table 4.5 Dough formulations used for torque measurements during mixing in the Tweedy 1 mixer

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>0% sugar</th>
<th>2% sugar</th>
<th>4% sugar</th>
<th>6% sugar</th>
<th>8% sugar</th>
<th>10% sugar</th>
<th>12% sugar</th>
<th>14% sugar</th>
<th>16% sugar</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flour d (11.5%)</td>
<td>400.00</td>
<td>400.00</td>
<td>400.00</td>
<td>400.00</td>
<td>400.00</td>
<td>400.00</td>
<td>400.00</td>
<td>400.00</td>
<td>400.00</td>
</tr>
<tr>
<td></td>
<td>/ 60.7</td>
<td>/ 59.9</td>
<td>/ 59.2</td>
<td>/ 58.5</td>
<td>/ 57.9</td>
<td>/ 56.6</td>
<td>/ 55.9</td>
<td>/ 55.3</td>
<td>/ 55.3</td>
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<td>7.20 /</td>
<td>7.20 /</td>
<td>7.20 /</td>
<td>7.20 /</td>
<td>7.20 /</td>
<td>7.20 /</td>
<td>7.20 /</td>
<td>7.20 /</td>
</tr>
<tr>
<td></td>
<td>1.09</td>
<td>1.08</td>
<td>1.07</td>
<td>1.05</td>
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<td>1.03</td>
<td>1.02</td>
<td>1.01</td>
<td>1.00</td>
</tr>
<tr>
<td>Sugar</td>
<td>0.00 /</td>
<td>8.00 /</td>
<td>16.00 /</td>
<td>24.00 /</td>
<td>32.00 /</td>
<td>40.00 /</td>
<td>48.00 /</td>
<td>56.00 /</td>
<td>64.00 /</td>
</tr>
<tr>
<td></td>
<td>0.00</td>
<td>1.19</td>
<td>2.37</td>
<td>3.51</td>
<td>4.63</td>
<td>5.72</td>
<td>6.79</td>
<td>7.83</td>
<td>8.85</td>
</tr>
<tr>
<td>Vegetable fat</td>
<td>20.00 /</td>
<td>20.00 /</td>
<td>20.00 /</td>
<td>20.00 /</td>
<td>20.00 /</td>
<td>20.00 /</td>
<td>20.00 /</td>
<td>20.00 /</td>
<td>20.00 /</td>
</tr>
<tr>
<td></td>
<td>3.03</td>
<td>3.00</td>
<td>2.96</td>
<td>2.93</td>
<td>2.89</td>
<td>2.86</td>
<td>2.83</td>
<td>2.80</td>
<td>2.77</td>
</tr>
<tr>
<td>Ascorbic acid</td>
<td>0.03 /</td>
<td>0.03 /</td>
<td>0.03 /</td>
<td>0.03 /</td>
<td>0.03 /</td>
<td>0.03 /</td>
<td>0.03 /</td>
<td>0.03 /</td>
<td>0.03 /</td>
</tr>
<tr>
<td></td>
<td>0.00455</td>
<td>0.00450</td>
<td>0.00444</td>
<td>0.00439</td>
<td>0.00434</td>
<td>0.00429</td>
<td>0.00424</td>
<td>0.00419</td>
<td>0.00415</td>
</tr>
<tr>
<td>Water</td>
<td>232.00</td>
<td>232.00</td>
<td>232.00</td>
<td>232.00</td>
<td>232.00</td>
<td>232.00</td>
<td>232.00</td>
<td>232.00</td>
<td>232.00</td>
</tr>
<tr>
<td></td>
<td>/ 35.2</td>
<td>/ 34.8</td>
<td>/ 34.4</td>
<td>/ 34.0</td>
<td>/ 33.6</td>
<td>/ 33.2</td>
<td>/ 32.8</td>
<td>/ 32.4</td>
<td>/ 32.1</td>
</tr>
</tbody>
</table>

Each experiment was conducted in triplicate and anomalous readings that were a result of electrical spikes removed. The mean values for each time was calculated and a moving mean of 19 s was taken for the torque measurements throughout mixing. (The time on the software was out by 3 s per minute, with 57 s on the software being equivalent to 60 s in real time.) This moving mean was chosen as it divided into the mixing time to give a whole number, and is not too long that it would obscure the data, or too short that the data would not be smoothed out.

The peak torque and torque at the end of mixing (end torque) are considered key parameters during mixing. These points can be identified using the Tweedy mixer and data acquisition computer, allowing comparison in the different sugar content doughs.

In non-mechanical dough development processes, the peak torque can be interpreted as the point of optimum dough development. However, the point of optimum dough development is disputed amongst researchers, and in a mechanical dough development process such as the CBP, energy input is a common measure of dough development.

The parameters which were assessed included: peak torque, end torque, work done after 240 s, and mean mixing speed. The peak torque is the highest torque prior to its decrease during mixing. The end torque is the torque at the end of the mixing session. The mixing speed for each sugar content was calculated as the mean of the mixing speed over the whole duration of the three mixing sessions. Blade revolutions were not calculated, as it is a function of time.
and mixing speed, and thus the mixing speed is indicative of the blade revolutions over the 240 s mixing session.

4.5.2 Results and discussion: The effect of sugar on torque during mixing

In the Tweedy mixer, torque is indicative of how the dough interacts with the mixer blade. In Figure 4.12 the torque traces of different sugar content doughs are presented. The shape of the curve is similar amongst all sugar contents. The torque increases for the first 80-120 s, and slowly declines thereafter. This increase in torque corresponds with the formation of a solid mass, the dough, with the peak torque being when the dough has formed. The results show less time is required to form a dough on increasing the sugar content. These times are illustrated in Figure 4.13. The decrease in time with increasing sugar is likely to be due to sugared doughs forming a solid mass quicker due to their adhesive properties. In addition, the peak and end torque appear to decrease as the sugar content increases. These are examined more closely later in this section.

![Figure 4.12](image_url) Torque of different sugar content doughs over mixing time whilst mixing at atmospheric pressure. The torques presented are based on a moving mean of 19 s, taken from the mean of three experiments.

Figure 4.13 shows the mixing time required to reach peak torque in the different sugar content doughs. It illustrates a gradual decline from 142 s in the non-sugar containing dough to 44 s in the 16% sugar containing dough. This is as expected due to the sugar dissolving into the water to create a sticky liquid, which helps bind the ingredients together in a shorter length of time.
Figure 4.13 Time to reach peak torque in different sugar content doughs. The data points illustrated are the mean of three measurements.

Figure 4.14 shows the peak torque during mixing of different sugar content doughs. The results show an increase in torque up to a sugar content of 1.5% sugar, a drop in torque to 3.05 Nm at 4% sugar, before the torque increases again to 3.04 Nm at 6% sugar and decreases as the sugar content increases beyond this. These results suggest that the peak torque, the torque of the dough on its formation, decreases on increasing the sugar content beyond 5%. The end torque presented in Figure 4.15 follows a similar trend. This reflects the results seen in Section 4.5.

Figure 4.14 Peak torque in different sugar content doughs whilst mixing at atmospheric pressure for 240 s. The data points illustrated are the mean of three measurements. The error bars presented are one standard deviation on either side of the mean.

Figure 4.15 End torque in different sugar content doughs, following mixing at atmospheric pressure for 240 s. The data points illustrated are the mean of three measurements. The error bars presented are one standard deviation on either side of the mean.
In Figure 4.16 the work done over the mixing session in different sugar content doughs is presented. The results do not show a trend. Section 4.4 suggesting sugar containing doughs have a higher consistency led to the expectation that more work would be done in a given time in doughs containing more sugar. It may be that a trend was not observed due to the small number of measurements and the 4°C end temperature range of the dough, which can affect work.

![Graph of work done in different sugar content doughs](image)

**Figure 4.16** Work done at end torque in different sugar content doughs, following mixing at atmospheric pressure for 240 s. The data points illustrated are the mean of three measurements. The error bars presented are one standard deviation either side of the mean.

The speed with which mixing in the Tweedy mixer occurs depends upon the mixing speed set on the system and the resistance exhibited by the dough mass in the mixer. The mixer speed was set to the same value for each dough. In Figure 4.17 the mixing speed during mixing of different sugar content doughs are illustrated. The results show a range of mixing speeds for each sugar content dough and no change in the mixing speed with sugar content, despite the lower torque with increased sugar. This finding illustrates that the torque in the Tweedy mixer differs to conventional torque measurements. Although the dough exhibited greater resistance to mixing in Section 4.4 on addition of sugar, the torque measurement in the Tweedy mixer does not correspond to this.

![Graph of mixing speed in different sugar content doughs](image)

**Figure 4.17** Mean mixing speed during 4 minute mixing sessions of different sugar content doughs. Each data point is an average of 684 data points from three batches of dough. The error bars presented are one standard deviation either side of the mean.

The results in this section show a quicker formation of a solid mass with an increase in dough sugar content and a difference during the mixing of the different sugar content doughs, with torque decreasing as the sugar content increases beyond 5%, a reflection of the differences in dough rheology.
4.6 The effect of mixing speed on aeration of sugared doughs

Section 2.2.2 discussed the importance of mixing speed on dough development. Chin and Campbell (2005) found that dough aeration is greater at faster mixing speeds. This section tests if this also applies to sugar containing doughs. It is important to know this, as mixing at a higher speed for less time is more efficient and therefore will save time and money. It was expected that mixing at a higher speed would be more efficient in sugared doughs, also.

4.6.1 Method: The effect of mixing speed on aeration of sugared doughs

Figure 4.17 of Section 4.5 showed that on changing the dough’s sugar content between 0% and 15%, the set mixing speed was not affected. It was hypothesized that over a set mixing time, increasing the mixing speed increases dough aeration, due to the occlusion of more gas. Doughs containing 0% and 15% sugar were mixed in the Tweedy 1 at different speeds for 180 s and their density determined using the double cup method. The formulation of the doughs and the mixing speeds used are presented in Table 4.6.

Table 4.6 Dough formulations and the mixing speeds used to assess aeration of different sugar content doughs at different mixing speeds

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>0% sugar dough formulation</th>
<th>15% sugar dough formulation</th>
<th>Mixing speed (rad s⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flour c (11%)</td>
<td>400.00 / 59.8</td>
<td>400.00 / 54.9</td>
<td>40, 47, 54, 62, 70, 77</td>
</tr>
<tr>
<td>Water</td>
<td>232.00 / 34.7</td>
<td>232.00 / 31.8</td>
<td></td>
</tr>
<tr>
<td>Sugar</td>
<td>0.00 / 0.00</td>
<td>60.00 / 8.23</td>
<td></td>
</tr>
<tr>
<td>Salt</td>
<td>7.20 / 1.08</td>
<td>7.20 / 0.99</td>
<td></td>
</tr>
<tr>
<td>Ascorbic acid</td>
<td>0.03 / 0.00448</td>
<td>0.03 / 0.00411</td>
<td></td>
</tr>
<tr>
<td>Vegetable fat</td>
<td>20.00 / 2.99</td>
<td>20.00 / 2.74</td>
<td></td>
</tr>
<tr>
<td>Fresh yeast</td>
<td>10.00 / 1.49</td>
<td>10.00 / 1.37</td>
<td></td>
</tr>
</tbody>
</table>

4.6.2 Results and discussion: The effect of mixing speed on aeration of sugared doughs

Figure 4.18 shows the density of the 0% and 15% sugar dough mixed at different speeds. Consistent with previous results in this thesis, the 15% sugar dough is denser at all mixing speeds. The results show a decrease in density following mixing at higher speeds over the set mixing time in both doughs. This is likely to be due more mixer blade revolutions in the mixing time and thus more gas is entrained in the dough. These results illustrate that higher mixing speeds are more efficient. It was also noted in Chapter 2 that it was necessary to mix above a critical mixing speed to develop the dough. The data points for each sugar content dough also lie on lines of matching gradient, suggesting that dough density is proportional to the mixing speed.
4.7 The effect of sugar on dough stickiness

Dough stickiness depends upon both its cohesive and adhesive properties. Increased quantities of sugar are likely to increase the stickiness of dough and earlier results in this chapter suggest they may help doughs form in less time. Increased cohesion may make portioning of the dough during production difficult and increased adhesion may cause a number of processing difficulties, such as yield loss and factory downtime from dough sticking to equipment, which increases production costs. This section investigates how dough adhesiveness changes with sugar and as a result, the quantity of sugar that can be processed in a Tweedy 1 mixer, based on the basic dough ingredients used.

4.7.1 Method: The effect of sugar on dough stickiness

Doughs with sugar contents ranging from 0-20% were mixed in the Tweedy 1, and prepared for analysis in the texture analyser. Table 4.7 details the formulation of these doughs.

Table 4.7 Strong flour dough formulations used to assess the effect of sugar on dough adhesiveness

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>0% sugar</th>
<th>1.5% sugar</th>
<th>5% sugar</th>
<th>10% sugar</th>
<th>15% sugar</th>
<th>16% sugar</th>
<th>17% sugar</th>
<th>18% sugar</th>
<th>19% sugar</th>
<th>20% sugar</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flour c (11%)</td>
<td>400.00 / 60.7</td>
<td>400.00 / 60.1</td>
<td>400.00 / 58.9</td>
<td>400.00 / 57.2</td>
<td>400.00 / 55.6</td>
<td>400.00 / 55.3</td>
<td>400.00 / 55.0</td>
<td>400.00 / 54.7</td>
<td>400.00 / 54.5</td>
<td>400.00 / 54.1</td>
</tr>
<tr>
<td>Vegetable fat</td>
<td>20.00 / 3.03</td>
<td>20.00 / 3.00</td>
<td>20.00 / 2.94</td>
<td>20.00 / 2.85</td>
<td>20.00 / 2.78</td>
<td>20.00 / 2.57</td>
<td>20.00 / 2.75</td>
<td>20.00 / 2.74</td>
<td>20.00 / 2.72</td>
<td>20.00 / 2.71</td>
</tr>
<tr>
<td>Salt</td>
<td>7.20 / 1.09</td>
<td>7.20 / 1.08</td>
<td>7.20 / 1.06</td>
<td>7.20 / 1.03</td>
<td>7.20 / 1.00</td>
<td>7.20 / 0.985</td>
<td>7.20 / 0.980</td>
<td>7.20 / 0.980</td>
<td>7.20 / 0.974</td>
<td></td>
</tr>
<tr>
<td>Ascorbic acid</td>
<td>0.03 / 0.00455</td>
<td>0.03 / 0.00451</td>
<td>0.03 / 0.00442</td>
<td>0.03 / 0.00448</td>
<td>0.03 / 0.00417</td>
<td>0.03 / 0.00415</td>
<td>0.03 / 0.00413</td>
<td>0.03 / 0.00410</td>
<td>0.03 / 0.00408</td>
<td>0.03 / 0.00406</td>
</tr>
<tr>
<td>Sugar</td>
<td>0.00 / 0.00</td>
<td>6.00 / 0.902</td>
<td>20.00 / 2.94</td>
<td>40.00 / 5.72</td>
<td>60.00 / 8.34</td>
<td>64.00 / 8.85</td>
<td>68.00 / 9.35</td>
<td>72.00 / 9.85</td>
<td>76.00 / 10.3</td>
<td>80.00 / 10.8</td>
</tr>
<tr>
<td>Water</td>
<td>232.00 / 35.2</td>
<td>232.00 / 34.9</td>
<td>232.00 / 34.2</td>
<td>232.00 / 33.2</td>
<td>232.00 / 32.3</td>
<td>232.00 / 32.1</td>
<td>232.00 / 31.9</td>
<td>232.00 / 31.7</td>
<td>232.00 / 31.6</td>
<td>232.00 / 31.4</td>
</tr>
</tbody>
</table>
Following mixing under atmospheric pressure for three minutes, the dough was rolled to a thickness of 11 mm. This is illustrated in Figure 4.19. This thickness was used to ensure a sufficient depth of dough was present for application of force with the texture analyser probe.

![Figure 4.19 Set up for rolling of the dough to obtain the desired thickness](image)

A circular cutter of 50 mm in diameter was then used to obtain three samples from each dough formulation. The base of the texture analyser was lightly dusted with flour to prevent the dough from sticking to it. The three dough samples were individually placed onto the base and a flat ended cylindrical probe penetrated the dough, under the adhesive test programme on the texture analyser. This is illustrated in Figure 4.20 and the settings used are presented in Table 4.8.

![Figure 4.20 Texture analyser probing dough sample to assess adhesiveness](image)

**Table 4.8 Texture analyser settings used for dough adhesiveness test**

<table>
<thead>
<tr>
<th>Texture Analyser Setting</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Pre-test speed</td>
<td>1 mm s⁻¹</td>
</tr>
<tr>
<td>Test speed</td>
<td>1 mm s⁻¹</td>
</tr>
<tr>
<td>Post-test speed</td>
<td>5 mm s⁻¹</td>
</tr>
</tbody>
</table>
4.7.2 Results and discussion: The effect of sugar on dough stickiness

The results of the investigation of sugar on dough stickiness are presented in Figures 4.21 to 4.23. Although investigations were carried out for doughs containing up to 20% sugar, experimental results were not obtained for the 20% sugar dough as adhesion of the dough to the surface and rolling pin prevented it from being rolled out.

Figure 4.21 shows the time taken to apply 0.98 N of force via a probe into the dough, removal of the probe from the dough and its return to starting position. The y-axis for force has negative and positive values, depending upon the direction the probe was moving and the force applied. Downwards movements towards the dough are expressed in negative units, and the converse applies for the upwards movement of the probe. Figure 4.21 shows the application of the 0.98 N force, followed by some multidirectional movement in force as it penetrates the dough, and then a sudden decrease in the force as the probe returns to its starting position. Another instability in force is then observed. It is the two instabilities that are relevant to the dough’s adhesive properties. The first is due to the resistance to removal resulting from the dough’s adhesive properties. The second is also due to dough stuck on the end of the probe, and any force seen to act on the dough at the end of the experimental time is due to dough stuck at the end of the probe. Following removal of the probe and at the end of the experimental time there appears to be some residual force on the probe for the experiment of the 19% sugar after 22 s.

For all doughs, the time required for removal of the probe from the dough is approximately 2 s. It was expected that this would increase as the sugar content increased. Figure 4.21 shows an increase in time taken to apply 0.98 N of force to the dough with an increase in sugar. This is most likely due to a more deformable and therefore softer dough. Figure 4.22 shows the distance the probe travelled to apply 0.98 N of force with time in the different sugar content doughs. Figure 4.22 shows a penetration depth ranging from 8-14 mm and times ranging from 11 to 21 s. It shows that the distance the probe travels to apply a force of 0.98 N increases with sugar again suggesting the dough is more deformable with sugar. This distance is better illustrated in Figure 4.23. Note the dough was only rolled to a depth of 11 mm. The greater dough penetration depth observed is due to the dough relaxing and suggests the dough should have been rested after mixing. Although the possible penetration depth differed, this should not have affected tests for dough stickiness, as sufficient depth was present for application of 0.98 N.

Observations found increased stickiness during handling and Figure 4.21 found doughs with increasing sugar contents were more likely to stick to the probe at the end of the experiment than doughs containing lesser quantities of sugar. The results also indicated a decrease in
dough firmness as sugar content increased. These results are in agreement with the observations made in Section 4.2.

![Graph showing force and time for different sugar contents](image1)

**Figure 4.21** Time and force required for application of 0.98 N of force with a probe to different sugar content doughs and then to return the probe to starting position. The data points illustrated are the mean of measurements from three samples from one dough batch.

![Graph showing distance and time for different sugar contents](image2)

**Figure 4.22** Penetration depth of a probe when applying 0.98 N of force to different sugar content doughs and distance travelled by the probe on returning to the starting position. The data points illustrated are the mean of measurements from three samples from one dough batch.

![Graph showing distance vs. sugar content](image3)

**Figure 4.23** Distance travelled by a probe to apply 0.98 N of force to different sugar content doughs. Each data point illustrated is the mean of three measurements from one dough batch.
4.8 The effect of sugar on dough uniaxial extension

The limit of expansion is directly related to rupture of cells and loss of gas (Dobraszczyk et al., 2001). Cells in dough expand during proving and baking. This occurs biaxially, thus a representative method of determining dough extensibility is to inflate the dough. Sheets of dough were inflated using a texture analyser and the pressure required to inflate the dough measured. However, it was found that the texture analyser frequently could not generate sufficient pressure to rupture the cell, or during inflation the dough would slip out from the plates that were holding it down. Thus a uniaxial extension method adapted from that used by Charalambides et al. (2006) was utilised. The force and distance the dough could be extended before snapping was measured, with expectations that sugar containing doughs, having received less gluten hydration and therefore not being as viscoelastic, would snap at less distance and force.

4.8.1 Method: The effect of sugar on dough uniaxial extension

This section looks into the rheology of bread dough using stress and strain during dough extension. Dough extensibility is an indicator of its elasticity. Measurements of dough extensibility are useful for estimating proving and baking performance. Measurements of force, distance and time were taken during a tension test on the dough using the texture analyser. The extensibility of four sugar contents strong flour doughs was investigated. Their formulations are given in Table 4.9.

| Table 4.9 Strong flour dough formulations used for uniaxial extension of dough |
|-------------------------------|---------------|-------------|--------------|
| Ingredient                      | 0% sugar      | 7.5% sugar  | 15% sugar    |
|                                | Quantity (g / %) |             |              |
| **Flour c (11%)**              | 400.00 / 60.7 | 400.00 / 58.0 | 400.00 / 55.6 | 400.00 / 55.0 |
| Vegetable fat                  | 20.00 / 3.03  | 20.00 / 2.90 | 20.00 / 2.78 | 20.00 / 2.75  |
| Salt                           | 7.20 / 1.09   | 7.20 / 1.04  | 7.20 / 1.00  | 7.20 / 0.990  |
| Ascorbic acid                  | 0.03 / 0.00455| 0.03 / 0.00435| 0.03 / 0.00417| 0.03 / 0.00413|
| Water                          | 232.00 / 35.2 | 232.00 / 33.66| 232.00 / 32.3 | 232.00 / 31.9 |
| Sugar                          | 0.00 / 0.00   | 30.00 / 4.35 | 60.00 / 8.34 | 68.00 / 9.35  |

Two batches of dough were made from each formulation and three 8 g samples cut from each dough, resulting in six measurements on each dough formulation. The dough samples were hand rolled into a cylindrical shape and inserted into a cylindrical mould with the internal length of 25 mm and width of 10 mm. The cylindrical mould is illustrated in Figure 4.20. It is a two piece hollowed out cylinder, allowing the dough sample to sit along its centre. The dough sample size was chosen as it would fill a greater space than that in the mould. Therefore, once sat in the mould there were no obvious air gaps, and all samples were consistent in dimension.
Figure 4.24 Two piece cylindrical mould used for shaping dough for uniaxial dough extension experiments

The excess dough in the mould extruded from the ends and between the two halves of the mould. The extruded ends were each flattened with a 200 g weight, which sat on the dough ends for 30 minutes as the dough rested. This formed handles from which the dough could be gripped. To ensure the flattened ends were equal in length and width, after 30 minutes, a rectangular stencil was placed over the ends, and the excess dough removed using a sharp knife. Hot air from a hairdryer was blasted at each dough end for 4 minutes to dry it out, preventing stretching during extension. This comprised of 2 minutes on each side of the dough. As drying out the dough can weaken it, a thin line of cyanoacrylate was squeezed along the edges of where the dough would be gripped, as highlighted in Figure 4.21. A rectangular stencil with 1 mm gaps every 1.5 mm and red liquid food colouring was used to mark the dough so uniformity of the extension could be observed. The colouring was then left to dry for 5 minutes.

Figure 4.25 Dough sample prepared for uniaxial extension system. Due to the similarity in colour of the dough and background, the dough has been marked out in the image. The areas where cyanoacrylate was applied is highlighted in yellow.

Using the rectangular dough ends, the dough was clamped onto the uniaxial extension device on the texture analyser and a tension test run. The dough was extended by upwards movement of the top clamp. The experimental set up is illustrated in Figures 4.22 and 4.23 and the settings are presented in Table 4.10.

Once the results were obtained, the shear stress and engineering strain on the dough when it snapped was calculated. Shear stress is experienced when a force acts parallel to the sample surface. The stress was calculated by dividing the force required to snap the dough by the
size of the area being extended. Engineering strain is experienced when a stress acts perpendicular to the sample surface. The strain was calculated by dividing the change in length of the dough sample by its length prior to extension.

**Table 4.10 Texture analyser settings used for dough uniaxial extension tests**

<table>
<thead>
<tr>
<th>Texture analyser parameter</th>
<th>Texture analyser setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>Test mode</td>
<td>Tension</td>
</tr>
<tr>
<td>Pre-test speed</td>
<td>6 mm s⁻¹</td>
</tr>
<tr>
<td>Test speed</td>
<td>6 mm s⁻¹</td>
</tr>
<tr>
<td>Post-test speed</td>
<td>6 mm s⁻¹</td>
</tr>
<tr>
<td>Target mode</td>
<td>Distance</td>
</tr>
<tr>
<td>Distance</td>
<td>400 mm</td>
</tr>
<tr>
<td>Trigger type</td>
<td>Auto (force)</td>
</tr>
<tr>
<td>Trigger Force</td>
<td>59 mN</td>
</tr>
<tr>
<td>Break mode</td>
<td>Level</td>
</tr>
<tr>
<td>Break sensitivity</td>
<td>29 mN</td>
</tr>
<tr>
<td>Break detect</td>
<td>Stop</td>
</tr>
<tr>
<td>Stop plot at</td>
<td>Start position</td>
</tr>
<tr>
<td>Tare mode</td>
<td>Auto</td>
</tr>
</tbody>
</table>

*Figure 4.26 Uniaxial extension system*

*Figure 4.27 Uniaxial extension system in action*
A weakness in the methodology was noted. It was not possible to measure the degree of clamping and hence the dough pieces were clamped to different extents. If clamped too loosely, the dough sample would fall out or be pulled out from the clamp during the test and if clamped too tightly, the sample would snap around the area of clamping. The different degrees of clamping affect the stress placed on the dough and can affect the force required to snap the dough. This limits reliability of the results and illustrates that the methodology requires further development. The learning points can be utilised in developing a new method. To improve the methodology, the dough could be extended horizontally with the bulk of the dough resting on a surface to prevent stress on the dough. Horizontal extension rather than vertical would ensure less force was placed on the lower parts of the dough, and less dough present through slumping on the lower half of the dough. Pulling at both ends of the dough would also provide a more uniform force throughout the dough, as would clamping dough ends to the same degree. An alternative method approved by the AACC of determining dough extensibility is through use of the Brabender Extensograph. This involves holding a strip of dough horizontally at both ends, and measuring the resistance on pulling the dough downwards with a hook.

4.8.2 Results and discussion: The effect of sugar on dough uniaxial extension

The methodology used to obtain these results requires further development to conclude. In addition trends were not expressed in the results presented in Figures 4.24-4.28.

It is known that during cell expansion, local thinning can occur, therefore it was expected that weaker areas of the dough exist and extension of the dough strips would not be uniform. It was expected that on increasing the sugar content of the dough, the gluten would not develop sufficiently and thus reduce the viscoelastic properties of the dough. Previous results in this chapter show sugar containing doughs to have a lower viscosity. This would result in the doughs being able to withstand less force and extension prior to snapping.

Observation of the marked lines on the dough strips showed a lack of uniform extension in the dough samples. Due to the requirement of further development in the methodology, measurements of the uniformity during extension were not assessed.

Figure 4.24 shows the force applied to each dough to snap it. As the sugar content increased, less force was required to snap the dough. This is most likely due to sugar weakening the dough structure, through preventing sufficient hydration of the proteins in the flour. Insufficient hydration causes less viscoelasticity, resulting in less elasticity reflected resistance during extension. The overlap in the error bars presented within the results and other results within this section reflect the difficulty in repeatability in the method and illustrate a need to further refine the method.
Figure 4.28 Force involved with time during uniaxial extension of a dough piece at speeds of 6 mms\(^{-1}\). Each point illustrated is a moving average of 60 points.

Figure 4.29 Distances involved with time during the extension of a dough piece at speeds of 6mms\(^{-1}\). Each point illustrated is a moving average of 60 points.
Figure 4.30 Force required to snap different sugar content dough strips during uniaxial extension of the dough at a speed of 6 mm s\(^{-1}\). The data points illustrated are the mean of six measurements. The error bars shown are one standard deviation either side of the mean.

Figure 4.25 shows the distance each sugar content dough is stretched before snapping. The results do not show a trend between the distance stretched before snapping and sugar content. It was hypothesized that the doughs containing more sugar would be less extensible. Again this is due to sugar interfering with gluten hydration and therefore development, and resulting in reduced viscoelastic properties.

Figure 4.31 Distance different sugar content dough strips were extended during uniaxial extension at a speed of 6 mm s\(^{-1}\) before snapping. The data points illustrated are the mean of six measurements. The error bars shown are one standard deviation either side of the mean.

Figure 4.26 illustrates the relationship between the force applied to different sugar content doughs to snap them and the distance these doughs will stretch before they snap. The results do not show a trend amongst distance travelled before snapping and force applied to snap the dough, although it is difficult to tell with just four points and scatter amongst the results. It was expected that as the force applied to the dough increased, the distance the dough could be stretched before it snapped would also increase. This was expected as a weaker dough should snap under less force and therefore be stretched less before snapping than stronger doughs. It was expected that sugar containing doughs would be weaker than non-sugar containing doughs.
Figure 4.32 The relationship between the force applied to a dough strip to cause it to snap and the distance the dough strip is extended before snapping, during uniaxial dough extension tests in different sugar content doughs.

Figure 4.27 shows the engineering strain on different sugar content doughs during the test. The results do not show a trend between engineering strain and sugar content. As discussed for Figure 4.25, it was expected that doughs containing more sugar would not be stretched as much before snapping. Therefore it was expected that the strain would decrease as the sugar content increased.

Figure 4.33 The engineering strain at snapping point on different sugar content doughs during uniaxial extension tests. The data points illustrated are the mean of six measurements. The error bars shown are one standard deviation either side of the mean.

Figure 4.28 shows the tensile stress on different sugar content doughs as they snap. The results show a decrease in the mean stress as the sugar content increases. However, the scatter in the data reduces confidence levels in the results seen. It was expected that the stress would decrease due to less force being required to snap the dough as the dough’s sugar content increased.
Figure 4.34 The stress at snapping point on different sugar content dough strips as they snap during uniaxial extension tests. The data points illustrated are the mean of six measurements. The error bars shown are one standard deviation either side of the mean.

4.9 ESEM of different sugar content doughs

Microscopy is commonly used to view the microstructure of bread dough. There are many types of microscopy. Environmental Scanning Electron Microscopy (ESEM) is a type of SEM. Using a focused beam of electrons, detailed micrographs were obtained of doughs containing 0% and 15% sugar. ESEM was used instead of SEM, as SEM requires samples to be electrically conductive and completely dry, therefore the dough would first have to be dried, altering its structure. ESEM employs modest gas pressures, allowing the dough to be scanned in its natural state, and does not require the dough to be coated in a conductive material which may conceal surface features. The microstructures of sugar and non-sugar containing doughs were compared using a FEI Quanta 200 (Hillsboro, Oregon, USA). This was carried out to help understand how the differences in structure affect the differences observed in sugar containing and non-sugar containing doughs throughout the thesis.

4.9.1 Method: ESEM of different sugar content doughs

For this experiment, a gas pressure of 0.88 Torr was created in the sample chamber using water vapour. The dough formulations used are given in Table 4.11.

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>0% sugar</th>
<th>15% sugar</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flour c (11%)</td>
<td>400.00 / 59.7</td>
<td>400.00 / 54.9</td>
</tr>
<tr>
<td>Water</td>
<td>232.00 / 34.7</td>
<td>232.00 / 31.8</td>
</tr>
<tr>
<td>Salt</td>
<td>7.20 / 1.08</td>
<td>7.20 / 0.99</td>
</tr>
<tr>
<td>Ascorbic acid</td>
<td>0.03 / 0.00448</td>
<td>0.03 / 0.00411</td>
</tr>
<tr>
<td>Sugar</td>
<td>0.00 / 0.00</td>
<td>60.00 / 8.23</td>
</tr>
<tr>
<td>Vegetable fat</td>
<td>20.00 / 2.99</td>
<td>20.00 / 2.74</td>
</tr>
<tr>
<td>Yeast</td>
<td>10.00 / 1.49</td>
<td>10.00 / 1.37</td>
</tr>
</tbody>
</table>
These doughs were subjected to a pressure step decrease from 1 bara to 0.5 bara to mimic CBP doughs and match the doughs presented in this thesis. The step change occurred after 110 s of mixing, and mixing continued for 230 s.

Following mixing, a dough sample approximately 10 g in mass was cut off with a razor blade and frozen in liquid nitrogen. A hammer was used to fracture the sample and a fragment of dough was placed into the ESEM and a scan run.

4.9.2 Results and discussion: ESEM of different sugar content doughs
Figures 4.35 and 4.36 are ESEM micrographs of fractured frozen dough samples containing 0% and 15% sugar, following 230 s of mixing with a pressure step decrease at 110 s from 1 bara to 0.5 bara. These are shown at two magnifications: 600 and 1200 times. At this level of magnification, different features in the dough can be identified. These include the gluten network, starch granules and gas cells. The gluten network is the wispy material scattered throughout the dough. Two types of starch granules are apparent, one which is ellipsoid in shape and the other spherical in shape. The gas cells are the sunken ellipsoid holes scattered throughout the dough and can be seen to range in diameter from 9-54 µm. The gas cells are similar in size to the starch granules, and it may be the case that some of the cells are recesses left by starch granules during fracturing.

When comparing the micrographs from the two dough formulations, differences can be seen in the gluten. In Figure 4.35 showing the micrographs for the non-sugar containing dough, the gluten strands are consistently distributed across the micrograph. However, in Figure 4.36, the micrographs for the dough containing 15% sugar, although the gluten has formed a connected network, it is intermittent throughout the dough matrix. A poorly formed gluten veil exists along the left hand side of Figure 4.36(b), and gluten is scattered elsewhere amongst the dough matrix, with areas existing with no significant quantities of gluten. This suggests the ingredients were not uniformly mixed and the dough was not developed. The connected network is a result of disulphide bonds between glutenin and gliadin and a sign of dough development. It is likely that the presence of sugar meant insufficient water was present to hydrate all the ingredients and distribute them uniformly throughout the dough mass. Sugars are smaller in size than starch. Sugars have a large surface area to size ratio, enabling them to dissolve fast during mixing, and prevent hydration of proteins and thus formation of gluten.
Figure 4.35 ESEM micrograph of 0% sugar dough mixed for 230 s with a step change from 1 bara to 0.5 bara at 110 s (a) at 600 x magnification (b) at 1200 x magnification
Figure 4.36 ESEM micrograph of 15% sugar dough mixed for 230 s with a step change from 1 bara to 0.5 bara at 110 s (a) at 600 x magnification (b) at 1200 x magnification
4.10 The effect of sugar on the dynamic dough density during proving

In Section 2.2.2, the double cup method of measuring density was introduced. The technique was later explained in Section 3.4.3 and has so far been used in this thesis for static measurements of dough density. This technique can also be used to monitor the dynamic dough density (Mousia et al., 1997; Chiotellis and Campbell, 2003), as done in this section to assess how sugar affects proving rates and the expansion capacity of bread dough. The proving rate is important, as a longer proving time costs more money. The expansion capacity is important as it is a measure of the dough’s viscoelastic properties, and the final volume of the bread. It was expected that the presence of some sugar would increase proving rates, due to the immediate supply of substrate available for yeast metabolism, and larger quantities of sugar would induce osmotic stress on the yeast, slowing down metabolism and therefore rates of bread dough proving. Expansion capacity of bread is expected to decrease with more sugar, due to less of the flour proteins becoming hydrated and therefore the doughs having lower viscoelastic properties.

4.10.1 Method: The effect of sugar on the dynamic dough density during proving

The effect of sugar on proving time and dough expansion capacity was determined through dynamic dough density measurements via the double cup method. The minimum dough density and time taken to reach minimum density during proving, were compared for strong and weak flour doughs containing different quantities of sugar. The dough formulations used are given in Tables 4.12 and 4.13.

**Table 4.12 Strong flour dough formulations used to observe the dynamic dough density during proving at 38°C**

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>0% sugar (g / %)</th>
<th>1.5% sugar (g / %)</th>
<th>5% sugar (g / %)</th>
<th>10% sugar (g / %)</th>
<th>15% sugar (g / %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flour c (11%)</td>
<td>400.0 / 61.4</td>
<td>400.0 / 60.9</td>
<td>400.0 / 59.6</td>
<td>400.0 / 57.9</td>
<td>400.0 / 56.2</td>
</tr>
<tr>
<td>Salt</td>
<td>7.2 / 1.11</td>
<td>7.2 / 1.10</td>
<td>7.2 / 1.07</td>
<td>7.2 / 1.04</td>
<td>7.2 / 1.01</td>
</tr>
<tr>
<td>Dough improver</td>
<td>2.0 / 0.307</td>
<td>2.0 / 0.304</td>
<td>2.0 / 0.298</td>
<td>2.0 / 0.289</td>
<td>2.0 / 0.281</td>
</tr>
<tr>
<td>Water</td>
<td>232.0 / 35.6</td>
<td>232.0 / 35.3</td>
<td>232.0 / 34.6</td>
<td>232.0 / 33.6</td>
<td>232.0 / 32.6</td>
</tr>
<tr>
<td>Fresh yeast</td>
<td>10.0 / 1.54</td>
<td>10.0 / 1.52</td>
<td>10.0 / 1.49</td>
<td>10.0 / 1.45</td>
<td>10.0 / 1.41</td>
</tr>
<tr>
<td>Sugar</td>
<td>0.0 / 0.00</td>
<td>6.0 / 0.913</td>
<td>20.0 / 2.98</td>
<td>40.0 / 5.79</td>
<td>60.0 / 8.44</td>
</tr>
</tbody>
</table>
Table 4.13 Weak flour dough formulations used to observe the dynamic dough density during proving at 38°C

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>0% sugar</th>
<th>1.5% sugar</th>
<th>5% sugar</th>
<th>10% sugar</th>
<th>15% sugar</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flour a (10.1%)</td>
<td>400.0 / 62.1</td>
<td>400.0 / 61.5</td>
<td>400.0 / 60.2</td>
<td>400.0 / 58.5</td>
<td>400.0 / 56.8</td>
</tr>
<tr>
<td>Salt</td>
<td>7.2 / 1.12</td>
<td>7.2 / 1.11</td>
<td>7.2 / 108.4</td>
<td>7.2 / 1.05</td>
<td>7.2 / 1.02</td>
</tr>
<tr>
<td>Dough improver</td>
<td>2.0 / 0.310</td>
<td>2.0 / 0.308</td>
<td>2.0 / 0.301</td>
<td>2.0 / 0.292</td>
<td>2.0 / 0.284</td>
</tr>
<tr>
<td>Water</td>
<td>225.0 / 34.9</td>
<td>225.0 / 34.6</td>
<td>225.0 / 33.9</td>
<td>225.0 / 32.9</td>
<td>225.0 / 32.0</td>
</tr>
<tr>
<td>Fresh yeast</td>
<td>10.0 / 1.55</td>
<td>10.0 / 1.54</td>
<td>10.0 / 1.51</td>
<td>10.0 / 1.46</td>
<td>10.0 / 1.42</td>
</tr>
<tr>
<td>Sugar</td>
<td>0.0 / 0.00</td>
<td>6.0 / 0.923</td>
<td>20.0 / 3.01</td>
<td>40.0 / 5.85</td>
<td>60.0 / 8.52</td>
</tr>
</tbody>
</table>

To prepare the dough, following mixing it was rolled out to a thickness of 11 mm and a cutter used to obtain two 23 mm in circular diameter samples. Each sample was swivelled for 30 s in a lightly floured conical flask to smooth out any damage on the outer dough edges to avoid unrepresentative loss of gas. The dough’s weight in air was taken, before it underwent proving in the double cup system. Proving took place at 38°C to mimic proving temperatures. The double cup system recorded the dough’s weight in silicone oil every 10 s. Recording took place digitally using Labview 7 Express (National Instruments, Newbury, Berkshire, UK) until the dough’s weight began to increase, and thus loss of gas was greater than that entrained in the cells through yeast carbon dioxide production. Four sets of measurements were carried out on each dough formulation, using two batches of dough. The mean of the four measurements was used to provide the results below.

4.10.2 Results and discussion: The effect of sugar on the dynamic dough density during proving

In Figure 4.37 the change in density over proving time for strong and weak flour doughs of different sugar contents are presented. For all doughs, the density decreases slowly initially before the rate picks up and slows down as it approaches a minimum and then increases. The changes in density observed agree with voidage measurements via X-ray CT during proving in Section 6.4.2.

There are three reasons why the initial drop in density is slower. The first is the cells in the dough are smaller and therefore more difficult to inflate. Secondly, the carbon dioxide generated by the yeast dissolves in the liquid phase of the dough before entering the bubbles; it takes time for the carbon dioxide concentration to build up sufficiently for rapid bubble growth. Finally, it is also slower initially as the dough temperature is lower and therefore yeast activity is slower. The dough leaves the mixer at 30°C, whereas the temperature in the experimental set up is 38°C. With the small sample size used, the temperature of the dough quickly increases with time, until it reaches 38°C. It is known that the initial rate is not slower
due to yeast activation rates, as fresh yeast was used. Unlike dried yeast, fresh yeast does not require hydration for activation.

The rate the density drops decreases as the density approaches the minimum due to the presence of larger cells (a result of cell growth and coalescence, as seen in Section 6.4.2) rupturing and causing loss of gas from the dough. As this occurs, cells continue to grow from yeast carbon dioxide production. After the accelerated decrease in dough density, the rate the density falls slows down as it approaches minimum density. The minimum density is reached when the viscoelastic properties of the dough have reached its limit for the total quantity of gas which can be retained. From this point, although the yeast continue to produce carbon dioxide, a greater quantity of gas than that which diffuses into the cells escapes from the dough as the cells breakup, resulting in an increase in dough density. The density of the dough continues to increase as proving continues, slowing down as the dough’s gas free density is approached. With the large surface area of the dough pieces, loss of gas from the dough samples is quicker than would occur in standard loaf sizes. Comparison to the results in Section 6.4.2, also show a higher proving rate in these samples, despite the larger dough samples used in the dynamic dough density measurements via the double cup. This is likely to be a result of higher proving temperatures increasing yeast carbon dioxide rates.

Figure 4.38 shows the time taken to reach minimum density during proving in different sugar content strong and weak flour doughs. An increase in proving time with increased sugar quantities is seen in agreement with Williams and Pullen (2007). The doughs containing 1.5% sugar are an exception to this, and reach minimum density in less time than the other doughs. The quickest proving rate was expected in the 1.5% sugar dough. This proportion of sugar is regularly used in industrial breadmaking to accelerate proving. Yeast use sugar as a substrate, so the addition of a small quantity of sugar to the dough accelerates carbon dioxide production rates. This is because there is an immediate supply available for yeast metabolism. When sugar is not added, there is some sugar present from starches within the flour, although the majority of these starches must first be broken down through enzymic activity. This effect is only true when small proportions of sugar are added. Lallemand (1996b) recommend 20% of the yeast weight of sugar is added to the dough formulation to avoid the lag phase. In the case of the dough formulation used here, this would be equivalent to 2% sugar. This is reflected in the overlap of lines between the 0%, 1.5% and 5% sugar doughs in Figure 4.37 around when the minimum density is reached. As seen by the addition of 10% and 15% sugar, increased quantities of sugar slow down yeast carbon dioxide production and therefore the proving rate of bread dough. This is likely to be true also in higher quantities of sugar than those investigated.

The observed reduction in proving rate with sugar could be due to a number of reasons: 1. Cells are smaller and therefore more difficult to inflate, as suggested in Section 6.2.2. 2. The increased ethanol production as a result of increased quantities of sugar creates a toxic environment (Carlson et al., 1991). It is known that the rate of carbon dioxide production is reduced by approximately 20% at ethanol concentrations of 4%, and 1 g of sugar yields 0.5 g
ethanol (Anon, 1996b). 3. The proportion of yeast present in the dough decreases as the sugar content increases, resulting in less carbon dioxide production in relation to the quantity of dough present. 4. Yeast became stressed due to the increased osmotic pressure on them, a result of the reduced water activity in the dough. It is known that sugar is one of the main characteristics that affects yeast performance and different strains are used worldwide to enable production of typical bread varieties of the respective country (Anon, 1996a). In addition, the presence of more sugar does not increase the rate at which yeast can digest this sugar. Thus once sufficient quantities of sugar are provided for yeast to begin metabolising immediately, more sugar is surplus. These results suggest that more appropriate strains of yeast are available for producing sweetened breads via the CBP; the slow carbon dioxide rate associated with the yeast strain used would incur increased production costs in sweetened doughs.

The results in Figure 4.38 also show that the minimum density was reached quicker in the weak flour doughs than the strong. This was as expected, as the lower gluten content of weak flour doughs means their viscoelastic properties and hence gas retaining capacity is lower.

Figure 4.37 Change in density of different sugar content (a) strong doughs (b) weak doughs, during proving at 38ºC. The results presented are the mean of four data measurements taken from two batches of dough. The error bars have been omitted for clarity.

Figure 4.38 Proving time required for different sugar content strong and weak flour doughs, to reach their minimum density. The data points are the mean of four measurements, taken from two batches of dough. Error bars are one standard deviation either side of the mean.
Figure 4.39 shows the minimum density reached during proving of the different sugar content strong and weak flour doughs respectively. The results do not show a trend between dough minimum density and sugar content, and doughs from the two flours illustrate a different result. The strong flour dough shows a decrease in the minimum density up to a sugar content of 10%. An increase in sugar to 15% sees the dough minimum density increase to a value greater than all of the other sugar contents. In the weak flour dough, the minimum density increases from a sugar content of 0% to 1.5% before decreasing to a sugar content of 10% and increasing as the dough sugar content increases to 15%. In the weak flour dough there is a greater difference in the minimum density between the different sugar content doughs than the strong flour doughs. This is likely to be due to the weak flour doughs being less stable. It was expected that the minimum density of the doughs during proving would be greater (more dense) in the doughs containing more sugar, due to their higher gas free dough densities, lower voidage and the expectation that a proportionally lower gluten content and most likely insufficient dough development, would result in lower viscoelastic properties and gas retaining capacity of the dough. The results show that the quantity of sugar present in the dough affects proving through extension of the proving time, which adds to the cost of bread production. The minimum density of doughs reached upon proving was not affected by the sugar content.

![Graph showing minimum density reached by different sugar content strong and weak flour doughs during proving at 38°C.](image)

**Figure 4.39** Minimum density reached by different sugar content (a) strong doughs (b) weak doughs when proving at 38°C. The data points are the mean of four measurements, taken from two batches of dough. Error bars have been omitted for clarity, as they were smaller than the markers used.
The effect of sugar on finished bread

Baking is the third and final stage of the three main breadmaking stages. The heat transferred leads to gas expansion and liquid vaporisation which causes further expansion of the dough volume and changes the dough into an edible and appealing product. This section investigates the effect of sugar on the baking of bread and on the characteristics of the finished bread. Bread made using a Tweedy mixer and a breadmaker were assessed. Industrially made bread is typically made in a Tweedy mixer, but by carrying out investigations on both pieces of equipment, the investigation becomes more interesting and the effect of sugar can be observed. Characteristics such as loss of water during baking, loaf firmness, loaf volume and the cell size distributions within the cells were investigated. These are expected to differ in different sugar content doughs, and it is expected that the different processing will lead to differences in results between the breadmaker and Tweedy loaves.

4.11.1 Method: The effect of sugar on finished bread

Table 4.14 gives the formulations for the assessed laboratory made loaves.

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>0% sugar</th>
<th>1.5% sugar</th>
<th>5% sugar</th>
<th>10% sugar</th>
<th>15% sugar</th>
<th>17% sugar</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Flour a</strong></td>
<td>400.00 /</td>
<td>400.00 /</td>
<td>400.00</td>
<td>400.00</td>
<td>400.00</td>
<td>400.00</td>
</tr>
<tr>
<td>(10.1%)</td>
<td>59.8</td>
<td>59.2</td>
<td>58.0</td>
<td>56.4</td>
<td>54.9</td>
<td>54.1</td>
</tr>
<tr>
<td><strong>Vegetable</strong></td>
<td>20.00 /</td>
<td>20.00 /</td>
<td>20.00</td>
<td>20.00</td>
<td>20.00</td>
<td>20.00</td>
</tr>
<tr>
<td>fat</td>
<td>2.99</td>
<td>2.96</td>
<td>2.90</td>
<td>2.82</td>
<td>2.74</td>
<td>2.71</td>
</tr>
<tr>
<td><strong>Salt</strong></td>
<td>7.20 / 1.08</td>
<td>7.20 / 1.07</td>
<td>7.20 / 1.04</td>
<td>7.20 / 1.02</td>
<td>7.20 / 0.987</td>
<td>7.20 / 0.975</td>
</tr>
<tr>
<td><strong>Ascorbic</strong></td>
<td>0.03 /</td>
<td>0.03 /</td>
<td>0.03</td>
<td>0.03</td>
<td>0.03</td>
<td>0.03</td>
</tr>
<tr>
<td>acid</td>
<td>0.00448</td>
<td>0.00444</td>
<td>0.00435</td>
<td>0.00423</td>
<td>0.00411</td>
<td>0.00406</td>
</tr>
<tr>
<td><strong>Water</strong></td>
<td>232.00 / 34.7</td>
<td>232.00 / 34.4</td>
<td>232.00 / 33.7</td>
<td>232.00 / 32.7</td>
<td>232.00 / 31.8</td>
<td>232.00 / 31.4</td>
</tr>
<tr>
<td><strong>Sugar</strong></td>
<td>0.00 / 0.00</td>
<td>6.00 / 0.889</td>
<td>20.00 / 2.90</td>
<td>40.00 / 5.64</td>
<td>60.00 / 8.23</td>
<td>69.00 / 9.34</td>
</tr>
<tr>
<td><strong>Fresh yeast</strong></td>
<td>10.00 / 1.49</td>
<td>10.00 / 1.48</td>
<td>10.00 / 1.45</td>
<td>10.00 / 1.41</td>
<td>10.00 / 1.37</td>
<td>10.00 / 1.35</td>
</tr>
</tbody>
</table>

Loaves of bread were made using either the breadmaker, or mixed in the Tweedy 1 and proved and baked, as detailed in Section 3.6.1. Two methods were used to determine if the effects of sugar were evident in both methods and how they varied between the methods.

In addition, three separate batches of the dough were made in the breadmaker for assessment of dough density immediately after mixing via the double cup system.
Once the loaves were cool, they were weighed and the difference in weight before and after baking was calculated as a percentage.

One parameter which can affect the final bread quality is the proving time. As seen in Section 4.10, proving time should be extended when increasing the sugar content of dough. In these experiments proving time was kept constant; each dough was proved for an hour, regardless of the sugar content. Thus it is likely that the sugar containing doughs were underproved, and the degree to which each dough was proved decreased as the sugar content increased. Proving each dough for a different length of time in accordance to the dough’s sugar content would have solved that problem. The results in Section 4.10 differ in the experimental conditions but could be used as a guideline to the proving time. However, varying the proving time creates another variable.

The specific volume of the loaves was determined using the methodology in Section 3.4.6. Following this each loaf was sliced transversely to a thickness of 15 mm using a serrated knife to ensure an even surface. Testing of the crumb structure and firmness was conducted on the three most central slices of the loaf.

The crumb structure was assessed using the C-Cell imaging system (Calibre Control International, Warrington, UK). Raw images were generated of the three most central slices from the different loaves and analysed images, focusing on the cell sizes. The C-Cell cell image is coloured in blue, with smaller cells being dark blue in colour and larger cells being lighter blue in colour. Those highlighted in red are holes, where holes are three times the standard deviation of the mean cell size. The cell number and size were the parameters of interest. Two parameters were output as measurements of cell size. These were cell volume and cell diameter. The C-Cell calculates the cell volume from 2D measurements of cell area and the estimated depth from the shadow cast into each cell. The volume of each cell is estimated through backwards calculations of the cell area. Cell diameter was used in preference to cell volume as an indication of cell size, as this is more accurate than using values obtained through estimation. A test was carried out to compare the difference in the given cell diameter and using backwards calculations of the cell volume to determine the diameter. The cell diameter was found to vary by less than 5%. This suggests the accuracy of the given C-Cell diameter is within 5%.

When presenting the number of cells, these are presented both as the number of cells of different sizes alone, and also the number of cells per square millimetre. The former is to present the full cell size distribution and enable any cells that skew the data to be identified and the latter the same, but should be more representative, taking into account the dimensions of the bread slices used.

The firmness was determined using the texture analyser. A flat ended cylindrical probe was pushed into the bread, and the force required to compress the bread by 8 mm whilst penetrating the dough at 2 mm s⁻¹ was measured. Single factor ANOVA tests at a 95% significance level were carried out on all test results.
4.11.2 Results and discussion: The effect of sugar on finished bread

In this section measurements of a range of parameters from laboratory made bread and purchased baked goods are presented respectively.

The weight of a bread product before and after baking changes as a result of moisture loss. The high temperatures cause the water in the product to evaporate and diffuse into the atmosphere. As the product cools down, it continues to lose moisture as steam. The proportion of water lost during breadmaking is important as it determines the final product weight. Bread is sold by weight, so a significant loss of moisture decreases profit margins. An investigation was carried out to determine whether the addition of sugar to a bread dough formulation would affect the quantity of water lost from the product. It was hypothesised that the addition of sugar would result in less moisture loss from the dough, due to 1. A crust forming earlier and slowing down moisture loss, and 2. Sugar increasing the temperature at which water evaporates. As the cooking time of the loaf was only 25 minutes, the window for which a crust would form was small, thus only a small difference in moisture loss is expected.

Figure 4.40 shows the relationship between the sugar content of the dough and the grams of liquid lost per gram of solid from baking each loaf. The proportion of weight loss in doughs produced from both methodologies increases up to a sugar content of 1.5% and then decreases up to a sugar content of 10%. The proportion of weight loss in the loaves then increases, although the size of the increase is twice as large in the breadmaker loaves. From a sugar content of 15% to 17% the weight loss in the breadmaker loaves increases, and that in the Tweedy loaves decreases. The results do not show a relationship between sugar content and weight loss during baking, but differences are exhibited in the breadmaking methods. There are a number of reasons why this may be the case: loaf size and in particular their surface area and the baking conditions, such as temperature, how heat is distributed and transferred to the dough, are factors that affect evaporation from the product surface.

The results do not agree with the hypothesis. It is likely that the consistent proving time meant sugar containing loaves had a lower volume and surface area, which countered the effect of reduced water loss expected with earlier crust formation and elevated evaporation temperatures.

The results show moisture loss is also consistently higher in the breadmaker loaves. This is most likely due to differences in surface area, the heat exposure time, the baking temperature, and the heat transfer method. For both cooking methods the product is cooked via conduction and convection, although convection plays a bigger role in the production of the Tweedy loaves, due to a larger quantity of hot air surrounding the loaves. This accelerates the cooking time of the loaf and explains why the length of the baking operation is greater in the breadmaker. Water evaporates at 100°C, and this temperature increases on addition of sugar. The longer the length of time bread is exposed to these temperatures, the more water that is lost from the dough. The dough was also present in the breadmaker for longer as the temperature in the breadmaker increased from the proving to baking.
temperature, whereas for the Tweedy loaves, the dough was put into a pre-heated oven. These results do not show a relationship between weight loss during baking and sugar content.

**Figure 4.40** The percentage weight loss of different sugar content loaves as a result of baking. Each data point is the mean of three measurements from separate loaves. Error bars are one standard deviation either side of the mean.

The specific volume of different sugar content loaves produced using both methods was determined. Due to the increased dough density following mixing (Section 3.4) and requirement for longer proving times (Section 4.10) it was hypothesized that the specific volume of the baked bread would decrease as the sugar content increased. Figure 4.41 shows the specific volume of different sugar content loaves made using the two methodologies. The Tweedy and breadmaker loaves show opposing trends. The Tweedy loaves show a general decrease in the specific volume as the sugar content increases, and the breadmaker loaves show an increase. However, there is also a lot of scatter in the results, in particular those of the breadmaker, making it difficult to identify with confidence if a trend exists. In Tweedy loaves an increase in the specific volume was observed as the sugar content increased from 0% to 1.5%. From a sugar content of 1.5% to 17%, the specific volume decreased from approximately 3.7 cm$^3$ g$^{-1}$ to 2.7 cm$^3$ g$^{-1}$. For the breadmaker loaves, a decrease of approximately 0.1 cm$^3$ g$^{-1}$ in the specific volume was seen as the sugar content increased from 0% to 1.5%. The specific volume continued to increase up to a sugar content of 10%. At 15% sugar, the specific volume dropped by approximately 0.3 cm$^3$ g$^{-1}$ and increased as the sugar content increased to 17%. ANOVA tests on the loaves from both methodologies found a significant difference between the specific volume of different sugar content loaves.

The results exhibited in the Tweedy loaves were in line with the hypothesis, showing a reduction in the specific volume of bread as the sugar content increased. An additional reason why sugar may reduce the specific volume of bread is dough development is likely to be
reduced, thus reducing the viscoelastic properties of the dough and making cell rupture more likely. It remains unclear why the specific volume of the breadmaker loaves increased with sugar, but is most likely related to the methodology. However, it is expected and was found that the increased dough density and increased proving time found in the Tweedy mixer would also apply to loaves produced in the breadmaker, regardless of the differences in methodology. These results are illustrated in Figures 4.42 and 4.43, respectively. Figure 4.42 shows the dough density outside the mixer following mixing, and Figure 4.43 shows the proving time of the dough mixed in the breadmaker. As with the Tweedy loaves, regardless of the dough’s sugar content and weight, the same proving time and conditions were used in all of the breadmaker loaves. The proving time and conditions used for breadmaking were according to the breadmaker programme set for a basic loaf. ANOVA tests at 95% significance level and the contrasting trends expressed by loaves of both methodologies suggest that sugar affects the specific volume, and the opposing trends are a result of the methodology. Industrial bread is made in a scaled up version of the Tweedy 1 mixer used. Upscaling increases dough aeration during mixing (Martin et al., 2004b). It is assumed this is consistent with the sugar content, thus it is likely that the trend expressed in the experimental results from the Tweedy 1 would resemble those in the CBP.

Although bread is sold by weight, consumers use the loaf’s volume as an indication of its value for money. This is a reason why the specific volume of the bread is important. The specific volume also impacts on the eating characteristics of the bread. Unless a denser bread is desired, increasing the sugar content has a negative effect on the specific volume of bread.

**Figure 4.41** The specific volume of different sugar content loaves. Each data point is the mean of three measurements from separate loaves. The error bars are one standard deviation either side of the mean.
Figure 4.42 Density of different sugar content doughs mixed in the breadmaker. Each data point is the mean of six measurements from separate loaves. The error bars are one standard deviation either side of the mean.

Figure 4.43 Change in density during proving at 38°C of different sugar content strong flour doughs mixed in a breadmaker. The results presented are the mean of four data measurements taken from two batches of dough. The error bars have been omitted for clarity.

Specific volume and density are related parameters quantifying the weight and space occupied by the dough. Figure 4.44 illustrates the relationship between dough density following mixing and proving. Figure 4.44 shows the Tweedy doughs to be less dense than the breadmaker doughs, implying greater aeration in the Tweedy mixer. However, no relationship can be seen between the density of the dough immediately following mixing and the minimum density of the dough obtained during proving. This finding reflects the well known point of the cell size distribution and gas holding capacity being responsible for the voidage of the final bread. The relationship between specific volume and the cell sizes and number density in the bread loaves made are explored later in this section.
Figure 4.44 The relationship between the density of different sugar content doughs immediately after mixing and their minimum density obtained during proving in breadmaker and Tweedy loaves.

The relationship between dough density following mixing and the density of the breadmaker and Tweedy loaves are illustrated in Figure 4.45. The results show opposing trends for breadmaker and Tweedy loaves. In Tweedy loaves, as the dough density following mixing increases, the density of the bread increases. In breadmaker loaves, as the density following mixing increases, the density of the bread increases. Although it is easy to assume that a denser dough will lead to a denser bread, there is no reason for this expectation, as it is the size and number of cells and manipulation of them throughout the breadmaking process which indicates the bread’s final density. However, the trends observed highlight that trends can exist within a set methodology and suggest the differences observed between the breadmaker and Tweedy loaves are due to differences in the methodology. The factor most likely to have caused the observed differences is the mixing. Tweedy mixers operate at a high speed, and entrain a greater volume of air in a finer distribution than doughs mixed in a conventional mixer for the same time. The opposing trends are likely to be a result of mixing affecting the cell distribution. It is known that in the breadmaker dough, doughs following mixing are denser with more sugar. The differences in distribution will have an effect at later stages of breadmaking and on the final bread.
Figure 4.45 The relationship between the density of different sugar content doughs immediately after mixing and their density as finished bread in breadmaker and Tweedy loaves. Linear regression lines are shown.

Figure 4.46 shows the relationship between minimum dough density obtained during proving and the density of the finished bread. The results do not show a correlation between the two parameters. It was expected that doughs with less sugar would have greater viscoelastic properties, which would be reflected in a lower density during proving and following baking. It is likely that the inadequate proving time for the sugar containing doughs made into bread affect the trend that would have been observed.

Consumers view firm bread as undesirable and stale. The force required to compress slices of bread is a measurement of its firmness. Two factors suspected to affect the firmness of bread are the moisture content of the crumb and the bread’s specific volume. It is hypothesised that drier crumb in bread of a greater density (higher gas free density and lower voidage) would be firmest. Although increasing the sugar content is known to decrease the water activity of the dough, the addition of sugar should help a crust form sooner during baking and increase the temperature at which the water in the dough evaporates, reducing moisture loss in the dough, therefore countering this effect. (This was not observed in Figure 4.40, (This was not observed in Figure 4.40, \( R^2 = 0.8558 \) and \( R^2 = 0.8525 \)).
In this thesis, the moisture content of bread was not determined, but density was presented previously in this section, and the relationship between loaf density and firmness is investigated later in this section.

Figure 4.47 shows the force required to compress different sugar content loaves by 8 mm. As with other results in this section, opposite trends are found in the loaves produced from both methodologies. As the sugar content increases, an increase was observed in the firmness in Tweedy loaves and a decrease in the breadmaker loaves. ANOVA tests found a significant difference between the firmness of the different sugar content loaves. These results show that production of sweetened bread with mixing in the Tweedy mixer can produce firmer bread, which consumer’s may perceive as unattractive.

![Figure 4.47](image_url)  
*Figure 4.47 The force required to compress different sugar content loaves by 8 mm. Each data point is the mean of twelve measurements from three separate loaves. The error bars are one standard deviation either side of the mean.*

In Figure 4.48, the relationship between bread density and the firmness of the crumb is presented. The results show an increase in firmness as the density of the loaves increased in both the Tweedy and breadmaker loaves. More resistance to force is expected in denser loaves due to both the presence of fewer gas cells and a denser crumb.

![Figure 4.48](image_url)  
*Figure 4.48 The relationship between the force required to compress bread 8 mm and the density of the bread in breadmaker and Tweedy loaves. Linear regression lines are shown.*
The distribution of air in bread is determined by the volume and number of cells (these are interconnected in bread). This is important in determining the eating characteristics of the bread. Different distributions are desirable depending on the eating characteristics sought. For example, the Mexican tortilla contains extremely small cells distributed amongst the flat bread, allowing it to be wrapped around different fillings without altering the bread’s texture. The gas cells in Italian ciabatta are comprised mainly of large cells. These give ciabatta a light and airy texture, as opposed to dense rye bread which contains few cells, all of which are considerably smaller in size.

In Figure 4.49, the relationship between sugar content and cell number density of the different sugar content loaves are illustrated. Figure 4.49 shows an increase from 0.6 cells mm\(^{-2}\) in non-sugar containing Tweedy mixer made bread to 0.64 cells mm\(^{-2}\) in bread containing 1.5% sugar. An increase to 5% sugar sees the cell density drop to 0.52 cells mm\(^{-2}\) before gradually increasing to 0.56 cells mm\(^{-2}\), as the sugar content increases to 17%. In the breadmaker loaves the cell density decreases from 0.62 cells mm\(^{-2}\) to 0.58 cells mm\(^{-2}\) as the sugar content increases from 0% to 10%. On increasing the sugar content to 15% and then 17% the cell density increases to 0.64 cells mm\(^{-2}\) and 0.67 cells mm\(^{-2}\), respectively. The results and ANOVA tests show differences between cell number density in different sugar content loaves, with a suggestion when considering both sets of data that as the sugar content increases, the number of gas cells decreases and then increases. These differences are expected to have a significant effect on an individual’s perception of the loaf. With differences having been observed in loaf specific volume (Figure 4.41) and force to compress the bread (Figure 4.47) it was expected that differences in the cell number density would have been observed. The observed differences found previously may also be due to differences in cell size. Cell size distribution is investigated later in this chapter.

Figure 4.49 shows the mean cell diameter of the different sugar content breadmaker and Tweedy loaves. Both figures show an initial increase in mean cell diameter and then decrease as the sugar content increases. For the breadmaker loaves, in Figure 4.50(a) from a sugar content
content of 0% to 1.5% the mean cell diameter increases by approximately 0.3 mm. As the sugar content increases to 17%, the mean cell diameter gradually decreases to 2 mm. In addition, the error bars are large and ANOVA found no significant difference in the mean cell diameter in breadmaker loaves. In the Tweedy loaves, Figure 4.50(b) shows a small increase in the mean cell diameter from 2.2 mm to 2.3 mm, as the sugar content increased from 0 to 10%. As the sugar content increased to 17%, the mean cell diameter dropped to 1.9 mm. ANOVA tests found a significant difference between the mean cell diameter of different sugar content Tweedy loaves, but no trend is expressed in the results, and the results show a lot of scatter.

**Figure 4.50** The mean cell diameter present in different sugar content (a) breadmaker loaves (b) Tweedy loaves. Each data point is the mean of nine measurements from three separate loaves. The error bars presented are one standard deviation either side of the mean.

Figures 4.51 and 4.52 show raw and analysed C-cell images of breadmaker and Tweedy loaves, respectively. The analysed C-cell images show the cell sizes by colour with smaller cells being darker in colour than larger cells, and holes being red in colour. The initial increase and then decrease in mean cell diameter seen in Figure 4.50 are illustrated in these two figures.
Figure 4.51 Raw and analysed C-cell cell intensity images of slices of breadmaker produced bread of different sugar contents.
Figure 4.52 Raw and analysed C-cell cell intensity images of slices of Tweedy produced bread of different sugar contents.
The specific volume of a loaf is largely dependent upon its cell size distribution. Figure 4.53 illustrates the relationship between specific volume and cell number density. There is scatter in the results but they show a negative correlation between the cell density and specific volume in both breadmaker and Tweedy loaves. A positive correlation was expected. The observed negative correlation is most likely a result of differences in cell size. The relationship between specific volume and mean cell diameter is illustrated in Figure 4.54. Figure 4.54 shows no trend in the relationship between the mean cell diameter in the breadmaker loaves and specific volume. A positive correlation is seen between the two parameters in the Tweedy loaves. However, there is a lot of scatter in the results. Together these results show that in the breadmaker loaves, cell number had a larger effect on the specific volume than mean cell diameter, which was found not to have an effect, and the specific volume in the Tweedy loaves was affected by both parameters. Another factor known to affect the specific volume of these loaves is the gas free dough density, which was found to increase as sugar content increased.

Figure 4.53 The relationship between the cell number density and specific volume in breadmaker and Tweedy loaves. Linear regression lines are shown.

Figure 4.54 The relationship between mean cell diameter and the specific volume of breadmaker and Tweedy loaves. Linear regression lines are shown.
Figures 4.55 and 4.56 illustrate the cell size distribution in breadmaker loaves. In Figure 4.55 this is presented as the number of cells per mm$^2$, and in Figure 4.56 as the mean number of cells per slice, thus not taking into account the differences in the slice areas, but allowing closer inspection to the raw data. The results show that as the cell size increases the cell density decreases. In addition, the results show little difference between the different sugar content doughs up to a mean cell diameter of 3 mm, but following this, as the cells increase in size, a difference in the cell density can be seen. Although a difference exists between the cell size distribution in the different sugar content doughs, a relationship between sugar content and cell size distribution is not seen in the results.

![Graph showing cell density vs mean cell diameter]

**Figure 4.55** *The cell size distribution in different sugar content finished breadmaker loaves. Each data point is measured from nine slices of bread from three separate mixes.*

![Graph showing mean number of cells per slice vs cell size]

**Figure 4.56** *The number of cells of different sizes per slice in breadmaker bread. Each data point is the mean of nine slices of bread from three separate mixes.*
Figures 4.57 and 4.58 illustrate the cell size distribution for different sugar content Tweedy loaves. In Figure 4.57, these are presented as the cell density of different diameter cells and in Figure 4.58, as the average number of different diameter cells within each slice of bread. As in the breadmaker loaves, as the cell size increases, the cell density decreases and the cell size distribution is similar up to a mean cell diameter of 3 mm. However, in the Tweedy loaves it can be seen that up to a mean cell diameter of 3 mm there are more cells in the dough containing 17% sugar, followed by the dough containing 15% sugar. As the cell size increases from 3 mm to the maximum mean cell diameter detected of 17 mm, differences in the cell size distribution can then be seen amongst the different sugar content loaves. The results show a weak trend between cell size distribution and sugar content, although the scatter in the data makes it difficult to conclude if the observations are noise or trends. Throughout the data it can be seen that as the sugar content increases the cell density decreases. Comparison of the cell size distribution in the 0% and 15% sugar Tweedy loaves (constant pressure mixed) to the cell size distribution in the 0% and 15% sugar pressure-vacuum mixed doughs obtained in the dough via X-ray CT in Section 6.2 are in agreement. In both cases they show the 15% sugar dough having a larger number of smaller cells and fewer bigger cells. As the effect of sugar on the cell size distribution are in agreement despite different mixing processes and additional breadmaking stages, the results suggest that in the quantities studied, sugar’s effect on the dough is larger than the effect of the other processes used to make this bread. However, as the X-ray dough comparison of sugared versus non-sugared doughs was only conducted on one dough sample, a conclusion cannot be drawn.

**Figure 4.57** The cell size distribution in different sugar content finished Tweedy loaves. Each data point is measured from nine slices of bread from three separate mixes.
Figure 4.58 The number of cells of different sizes per slice in Tweedy bread. Each data point is the average of nine slices of bread from three separate mixes.

The work on laboratory made loaves shows that sugar affects the quality parameters of finished bread loaves, and that there is a correlation between some of these parameters. However, the opposite results obtained through using two different methodologies emphasises the importance of the methodology in achieving the desired result. The results show that a balance must be achieved between the ingredients, methodology and the numerous characteristics which are indicative of quality.
4.12 Summary

This Chapter assessed the effect of sugar throughout the breadmaking process using a range of tests on both strong and weak flour doughs. Observations of sugar’s effect on starch water mixture showed that sugar affects its interaction, and justified studying the effect of sugar throughout the breadmaking process. The experiment also found that the addition of sugar meant doughs were less elastic and less visco. It is suspected that the former is due to reduced gluten formation due to the reduction in proportion of ingredients other than sugar which were present, and in particular water which is needed for hydration of gluten to create its viscoelastic properties, and the latter is due to reduced swelling of the starch granules. The reduced viscosity of the mixture will affect the rheology of the dough and therefore its aeration. Reduced aeration was found in sugared doughs, in terms of both the dough voidage and gas free dough density. This is undesirable, as denser bread means less value for money and is generally firmer in texture, which is perceived as stale and unfresh. The change in dough rheology that caused the changes in its aeration were reflected in the differences in torque during mixing in both the Farinograph and the Tweedy mixer. These mixers demonstrated that doughs formed in less time on increasing the sugar content and the Farinograph showed doughs to have a higher consistency with more sugar, suggesting they were stiffer. Formation of doughs in less time is likely to be due to the increased adhesive properties of the dough observed during investigation of the adhesiveness of different sugar content doughs. The formation of a dough in less time is beneficial as it cuts down on production time and therefore costs. Given the results seen in this chapter, a small quantity of sugar (less than 5%) added to the dough would decrease processing time and therefore costs, as well as adding to the flavour of bread.

Micrographs of the dough revealed that the gluten veil had not properly formed in the sugar containing dough, most likely a result of insufficient mixing and development of the dough, due to reduction in the proportions of ingredients other than sugar and insufficient quantities of water and a to disperse and hydrate the ingredients. It is important that the gluten veil is developed as it enables cells within the dough to retain more gas and therefore helps produce a loaf of greater volume.

The proving time of the dough was found to increase on increasing the sugar content of the dough in both breadmaker and Tweedy mixed produced dough, except with the addition of 1.5% sugar where the proving time was found to decrease. However, the minimum density reached during proving was not affected in the dough formulations used under the conditions assessed. It is likely that increasing the water content to compensate for the lower water activity would have improved the viscoelastic property of the dough and resulted in a difference in the proving capacity of the dough.

Different parameters were assessed in finished bread made using a Tweedy mixer and breadmaker, and contrasting trends were found in a number of parameters in both methods, showing that sugar does affect the quality parameters investigated and the methodology uses
plays a key role in determining the outcome. Industrially made loaves are typically made in a Tweedy mixer. Results in Section 4.11 can be used as a guideline to the effect of sugar on Tweedy mixer produced loaves.

This chapter also saw on a number of occasions that on addition of low quantities of sugar such as those containing 1.5% and 5%, the trends expressed in the doughs differed to those containing larger proportions of sugar. For example, up to 5% sugar, Tweedy loaves were found to increase in specific volume, and beyond 5% sugar, the specific volume of the loaves decreased. These results suggest that the effect of sugar on the quality parameters of bread dough and bread are not linear and a change in behaviour and response occurs in doughs containing somewhere between 1.5% and 5% sugar. It was also found that small quantities of sugar can be beneficial to breadmaking by formation of a dough in less time, sometimes forming less dense doughs, as well as producing softer loaves of higher volume.

This chapter illustrated that sugar affects the quality parameters within bread and different proportions of sugar and the way in which the product is made affect these qualities therefore highlighting the requirement to pay attention to every variable within breadmaking.
Chapter 5 - Aeration in bread dough with and without sugar

5.1 Introduction

Pressure-vacuum mixing is frequently practised in the CBP to produce bread of a fine cell distribution, a desirable quality in bread. Sugar containing CBP doughs made in the Tweedy mixer are generally mixed at atmospheric pressure to the required work input (Cauvain and Young, 2006). As pressure-vacuum mixing is known to benefit the structure of non-sugar containing doughs, it would be interesting to determine if pressure-vacuum mixing benefits sugar containing doughs. This can be done through assessing the dough voidage following mixing. However, aeration of bread dough during mixing is a dynamic process; gas is simultaneously entrained and disentrained from the dough, and cells within the dough breakup and coalesce. Even following completion of mixing, removal of dough from the mixer that was mixed under pressure or vacuum affects its voidage, due to the pressure step change which occurs. The population balance model of dough aeration during mixing developed by Martin et al. (2004) implements a step change, which is a generic approach of characterising dynamic systems. Through using the model, values for cell entrainment and disentrainment in bread dough during mixing can be conveniently derived from the developed and available model. Use of a population balance model also gives general insight into the aeration processes in doughs of different sugar contents which will be true across all types of mixer.

An investigation into pressure-vacuum mixing can also help determine the optimal time for which vacuum mixing should be used. Pressure-vacuum mixing for too short a time is likely to produce dough of fewer cells due to the lack of subdivision of expanded cells. On the other hand, pressure-vacuum mixing for too long may be detrimental towards the dough voidage and also has cost implications, from drawing a vacuum and the wear on the vacuum pump.

The aim of this Chapter is to assess the differences in the aeration of different sugar content doughs during mixing.

5.2 Aeration of bread dough with and without sugar methodology

Bread is not the only food that has been studied using a population balance model. Jang et al. (2005) used a population balance model to study the whipping of gelatin solutions and aqueous emulsions, using microscopy to measure cell size distributions. Casenave et al. (2012) used a population balance model to study the crystallisation of ice crystal size distribution in ice cream. This thesis uses a population balance model to study the aeration of bread dough during mixing.

The methodology used to investigate the aeration of bread dough containing varying quantities of sugar involves two elements: an experimental methodology and a pressure step change modelling methodology to which the experimental results are applied. Section 5.2.1
discusses the experimental methodology and Section 5.2.2 the pressure step change modelling methodology.

5.2.1 Experimental methodology

A series of strong and weak flour doughs containing 0%, 7.5% and 15% were characterised in terms of their gas free dough density and voidage, using the method in Section 3.5.

Strong and weak flour doughs containing 0%, 7.5% and 15% sugar were made in the Tweedy 1 mixer. 17 doughs from each sugar content and flour were made, with each being mixed to different times, as detailed below in this section, and their voidage calculated from density measurements using the double cup method. Tables 5.1 and 5.2 give the formulations of the strong and weak flour doughs respectively.

**Table 5.1 Strong flour dough formulations used to derive entrainment and disentrainment values from a population balance model on the aeration of bread dough**

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>0% sugar</th>
<th>7.5% sugar</th>
<th>15% sugar</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flour d (11.5%)</td>
<td>400.00 / 60.7</td>
<td>400.00 / 58.0</td>
<td>400.00 / 55.6</td>
</tr>
<tr>
<td>Vegetable fat</td>
<td>20.00 / 3.03</td>
<td>20.00 / 2.90</td>
<td>20.00 / 2.90</td>
</tr>
<tr>
<td>Salt</td>
<td>7.20 / 1.09</td>
<td>7.20 / 1.04</td>
<td>7.20 / 1.00</td>
</tr>
<tr>
<td>Ascorbic acid</td>
<td>0.03 / 0.00455</td>
<td>0.03 / 0.00435</td>
<td>0.03 / 0.00417</td>
</tr>
<tr>
<td>Water</td>
<td>232.00 / 35.2</td>
<td>232.00 / 33.7</td>
<td>232.00 / 32.3</td>
</tr>
<tr>
<td>Sugar</td>
<td>0.00 / 0.00</td>
<td>30.00 / 4.35</td>
<td>60.00 / 8.34</td>
</tr>
</tbody>
</table>

**Table 5.2 Weak flour dough formulations used to derive entrainment and disentrainment values from a population balance model on the aeration of bread dough**

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>0% sugar</th>
<th>7.5% sugar</th>
<th>15% sugar</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flour b (10.4%)</td>
<td>400.00 / 61.3</td>
<td>400.00 / 58.6</td>
<td>400.00 / 56.2</td>
</tr>
<tr>
<td>Vegetable fat</td>
<td>20.00 / 3.07</td>
<td>20.00 / 2.93</td>
<td>20.00 / 2.81</td>
</tr>
<tr>
<td>Salt</td>
<td>7.20 / 1.10</td>
<td>7.20 / 1.06</td>
<td>7.20 / 1.01</td>
</tr>
<tr>
<td>Ascorbic acid</td>
<td>0.03 / 0.00460</td>
<td>0.03 / 0.00440</td>
<td>0.03 / 0.00421</td>
</tr>
<tr>
<td>Water</td>
<td>225.00 / 34.5</td>
<td>225.00 / 33.0</td>
<td>225.00 / 31.6</td>
</tr>
<tr>
<td>Sugar</td>
<td>0.00 / 0.00</td>
<td>30.00 / 4.40</td>
<td>60.00 / 8.42</td>
</tr>
</tbody>
</table>

A sugar content of 15% was used, due to this exceeding the greatest quantity of sugar found in supermarket bread products, and mixing in the Tweedy mixer still being effective. It was observed that the presence of too much sugar in the dough caused it to spin around with the mixing blade, as opposed to being turned and folded onto itself. A sugar content of 0% was used. This sugar content is typical of industrial bread and reflects the results of unadulterated bread. 7.5% sugar was used as the third sugar content due to it being midway between the two extremes, thus enabling insight into whether trends observed in the 15%
sugar dough are observed with half of the sugar content, and therefore if the effect of sugar is gradual or sudden once a threshold is met.

17 separate mixes were used for each of the six dough formulations and mixing parameters. Their mixing times were 90, 100, 105, 110, 115, 120, 130, 140, 150, 160, 170, 180, 190, 200, 210, 220, and 230 s, with a pressure step change occurring at 110 s. These were conducted in a random order to minimise run artefacts. The shortest and longest mixing times were chosen as 90 s is sufficient time for the ingredients to form a dough and after 230 s the dough should have reached its steady state voidage.

A series of doughs underwent a pressure step increase or pressure step decrease during mixing to assess the effect of sugar on dough aeration dynamics. For the pressure step increase experiments the pressure was increased from 1 bara to 2 bara. For the pressure step decrease experiments, in order to quantify the differences in dough aeration over different sized pressure changes, the pressure was decreased from 1 bara to 0.5 bara and also 1 bara to 0.25 bara for the mixing sessions. These mixing conditions are presented in Table 5.3.
Table 5.3 Weak flour dough formulations used to derive entrainment and disentrainment values from a population balance model on the aeration of bread dough

<table>
<thead>
<tr>
<th>Dough</th>
<th>Mixing time (s)</th>
<th>Mixer pressure (bara)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Strong flour, 0% sugar</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Strong flour, 7.5% sugar</td>
<td>90, 100, 105, 110, 115, 120, 130, 140, 150, 160, 170, 180, 190, 200, 210, 220, 230</td>
<td>1 to 2 (Pressure step increase) $P_1/P_2 = 0.5$</td>
</tr>
<tr>
<td>Strong flour, 15% sugar</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Weak flour, 0% sugar</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Weak flour, 7.5% sugar</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Weak flour, 15% sugar</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

5.2.2 Pressure step change modelling methodology

The no break up model described in Section 2.2.4 was simultaneously fitted to each set of experimental voidages for the six dough formulations presented in Table 5.3 for the three pressure step changes for each dough formulation (varying in flour type and sugar content), producing one set of parameters for the three different step changes. The results were fitted by changing the parameters: initial voidage, entrainment rate and disentrainment coefficient. This cross-fitting reduces the problem of carrying forward experimental errors, which occurs with direct model fittings to experimental results. In direct fittings, the initial voidage, entrainment rate and disentrainment coefficient were fitted through minimising the absolute error to that set of experimental voidages alone, rather than three sets of experimental voidages at once. Figures showing direct individual model fittings to each set of experimental voidages are also presented. This ideally would have produced aeration parameters similar showing the same trend as the cross-fitted model, amongst the three different sugar content doughs. However, due to scatter amongst the experimental results, the same trend was not observed, and cross-fitting of the model was found to be more appropriate in this case.

5.3 Results and discussion

This section is split into two: Section 5.3.1 details the experimental results, their interpretation and discussion and Section 5.3.2 details the results of the modelling.
5.3.1 Experimental results

Figure 5.1 shows the characterisation of the strong and weak flour doughs of varying sugar contents used in this study. The dough voidage at 1 bara is illustrated in Figure 5.1 (a) and the gas free dough density is illustrated in Figure 5.1 (b). Figure 5.1 (a) shows a decrease in dough voidage as sugar content increases in doughs produced from both flour types, with a larger difference in voidage of different sugar content doughs in the weak flour doughs. This suggests that sugar has a greater effect on weak flour doughs than strong. Figure 5.1 (b) shows an increase in the gas free dough density as the sugar content increases in the strong flour doughs. In the weak flour doughs, as the sugar content increases from 0% to 7.5%, the gas free dough density drops from 1.2627 g cm\(^{-3}\) to 1.2590 g cm\(^{-3}\), before increasing to 1.2709 g cm\(^{-3}\) as the sugar content increases to 15%. These results are in agreement with those in Section 4.3, which showed an increase in gas free dough density and a decrease in voidage as sugar content increased, with a larger difference in dough voidage of the weak flour doughs than strong.

![Figure 5.1](image1.png)

**Figure 5.1** (a) Voidage at 1 bara of strong and weak flour doughs containing 0%, 7.5% and 15% sugar (b) Gas free dough density of strong and weak flour doughs containing 0%, 7.5% and 15% sugar

In this section, the model fitting to the experimental results is illustrated alongside the experimental results for convenience and practicality and only the experimental results are discussed. The model fitting and further results relating to the modelling are discussed in Section 5.3.2.

Figure 5.2 shows the in-situ dough voidages over mixing of the six pressure-vacuum mixed doughs with \(P_1/P_2 = 2\). (a) to (f) are the cross fittings and (g) to (l) the direct fittings. The results show the same trend among the two flour types and the three sugar concentrations. They show a sudden increase in voidage on reducing the pressure, a result of cell expansion. The dough voidage then falls over the next 20-40 s and stays fairly constant throughout the remaining duration of the mixing session, as it approaches steady state voidage. These observations are consistent with previous comparable studies reported by Martin et al. (2004a) and Campbell et al. (2008b).
Although the same trend is exhibited among the six doughs, their response to pressure-vacuum mixing differs in the extent of the immediate effect on voidage of drawing a vacuum, how quickly they change in response to the vacuum, their steady state voidages reached and the scatter among the data points.

The strong flour doughs reach steady state voidage more quickly than the weak flour doughs, have a higher steady state voidage and also show less scatter in their results after drawing a vacuum. It is likely that the scatter is a result of the weaker structure of the lower protein doughs making it less stable and thus more prone to change.

In non-sugar containing doughs, the voidage of the dough following pressure-vacuum mixing prior to the step change and at the end of the mixing session are similar. However, on addition of sugar, the results show that the higher the sugar content in the dough the quicker and larger the drop in voidage following application of a vacuum and the lower the steady state voidage. This suggests that the size to which the cells expand once a vacuum is drawn makes them more likely to be disentrained from the dough. This could be due to the cells being more likely to disentain in the sugared doughs due to the reduced viscoelastic properties of sugared doughs and the cells being larger.

Although there is some scatter in the data, the 0%, 7.5% and 15% sugar strong doughs appear to approach a steady state voidage of 0.065, 0.060 and 0.055, respectively. As mentioned previously, the weak flour results exhibit more scatter. The steady state voidage the 0%, 7.5% and 15% weak flour doughs appear to approach are 0.072, 0.058 and 0.042, respectively. These results suggest that pressure-vacuum mixing is detrimental towards the dough voidage, and therefore should not be used for the production of sugared breads. The results also suggest that mixing for less time under vacuum in the 0% sugar dough would be financially beneficial and not compromise the cell distribution, as steady state voidage may have been reached before the end of the mixing session.
Figure 5.2 Experimental and model voidages during pressure-vacuum mixing. \( P_1 = 1 \) bara, \( P_2 = 0.5 \) bara. Each data point is from a separate dough mix. (a) strong flour, 0% sugar (b) weak flour, 0% sugar (c) strong flour, 7.5% sugar (d) weak flour, 7.5% sugar (e) strong flour, 15% sugar (f) weak flour, 15% sugar
Figure 5.2 Experimental and model voidages during pressure-vacuum mixing. $P_1 = 1$ bara, $P_2 = 0.5$ bara. Each data point is from a separate dough mix. (g) strong flour, 0% sugar (h) weak flour, 0% sugar (i) strong flour, 7.5% sugar (j) weak flour, 7.5% sugar (k) strong flour, 15% sugar (l) weak flour, 15% sugar.
Figure 5.3 shows the in-situ dough voidages over mixing of the six pressure-vacuum mixed doughs with $P_1/P_2 = 4$. Figure 5.3 (a) to (f) are the cross fittings and figures 5.3 (g) to (l) are the direct fittings. The results show the same trend in the two flour types and the three sugar concentrations. They show a sudden increase in voidage on reducing the pressure, as a result of cell expansion. The dough voidage falls over the next 20-30 s and stays constant throughout the remaining duration of the mixing session, as it approaches steady state voidage.

As in the smaller pressure step down experiment, the six results exhibit the same trend in their voidage response to the pressure change. However, the extent and speed of the immediate effect on voidage of drawing a vacuum, the steady state voidages reached and the scatter amongst each set of results differ. As found in Figure 5.2, in the larger pressure step down experiment, the strong flour doughs also have a higher steady state voidage than weak flour doughs and there is less scatter amongst the voidage in the strong flour doughs.

For the doughs containing no sugar, the results show little change in voidage prior to application of a vacuum and at the end of the mixing session. The effect of vacuum mixing on the sugar containing doughs differs, causing a drop in voidage below that prior to application of a vacuum and a lower voidage at the end of the mixing session in the sugar containing doughs. Although there is some scatter in the data, the 0%, 7.5% and 15% sugar strong doughs appear to approach a steady state voidage of 0.060, 0.047 and 0.050, respectively. As mentioned previously, the weak flour results exhibit more scatter. The steady state voidage the 0%, 7.5% and 15% weak flour doughs appear to be approaching are 0.069, 0.035 and 0.026, respectively. These effects become more significant as the sugar content increases over the doughs investigated. As in Figure 5.2, these results illustrate that pressure-vacuum mixing is detrimental to the voidage in sugar containing doughs, producing doughs of lower voidage than constant pressure mixing at atmospheric pressure.

Differences were observed on comparison of Figures 5.2 and 5.3, the dough responses to the two different sized pressure step changes during pressure-vacuum mixing. To begin with, the larger pressure step change mixes in Figure 5.3 caused a greater increase in voidage on application of a vacuum. Following application of a vacuum, the approach to steady state voidage was quicker with a larger pressure step change. The voidage at the end of the mixing sessions was higher in the doughs mixed with a smaller pressure step change. A larger change in voidage was expected in the doughs subjected to a greater step change due to the effect of a larger sudden pressure change on the dough, and as the partial vacuum drawn is greater, more gas drawn out from the dough. However, as noted previously, the end voidage of pressure-vacuum mixed non-sugar dough was not affected by the step change. Section 4.4 showed that the presence of sugar in the dough formulation affects its gas free dough density and voidage. These results further illustrate that sugar affects dough aeration during mixing and show that pressure-vacuum mixing reduces the dough voidage, and the larger the step change and the sugar content, the lower the end voidage. The modelling results in Section 5.3.2 look into entrainment and disentrainment rates of gas within the different doughs, helping explain the possible causes of the observed differences in aeration.
Figure 5.3 Experimental and model voidages during pressure-vacuum mixing. $P_1 = 1$ bara, $P_2 = 0.25$ bara. Each data point is from a separate dough mix. (a) strong flour, 0% sugar (b) weak flour, 0% sugar (c) strong flour, 7.5% sugar (d) weak flour, 7.5% sugar (e) strong flour, 15% sugar (f) weak flour, 15% sugar
Figure 5.3 Experimental and model voidages during pressure-vacuum mixing. $P_1 = 1$ bara, $P_2 = 0.25$ bara. Each data point is from a separate dough mix. (g) strong flour, 0% sugar (h) weak flour 0% sugar (i) strong flour, 7.5% sugar (j) weak flour 7.5% sugar (k) strong flour, 15% sugar (l) weak flour, 15% sugar
Figure 5.4 shows the in-situ dough voidages over mixing of the six doughs mixed with a pressure step increase from 1 bara to 2 bara. (a) to (f) are the cross-fittings and (g) to (l) the direct fittings. The results show the same trend among the two flour types and the three sugar concentrations. They show a sudden decrease in voidage on reducing the pressure, a result of cell compression. The dough voidage then rises over the mixing session as it approaches steady state, and the cells relax.

The six doughs exhibit the same trend in respond to the pressure step up. Their response to the dual pressure mixing differs however, in the time taken to reach steady state voidage and the steady state voidage reached, in particular the difference in voidage at the start and end of the mixing session. On increasing the sugar content of the dough, the steady state voidage is reached sooner and is lower. The 0% sugar doughs do not appear to have reached steady state voidage over the mixing session, and there is uncertainty to whether the 7.5% sugar doughs reach steady state voidage over the mixing session. This suggests the doughs were not mixed for long enough. However, this problem is unavoidable, as longer mixing sessions increase the temperature within the mixer, producing a sticky dough, which does not follow the conventional Tweedy mixing action. Amongst the six dough formulations, the increase in voidage from prior to the step change to after the step change becomes lesser, changing from an increase in dough voidage to a decrease in dough voidage, although it is difficult to determine the extent of these voidage differences amongst the different sugar content doughs due to steady state voidage having not been reached during the mixing session. This observation may be due to an increased likelihood of cell rupture in the sugar containing doughs following a pressure step change which weakens cell stability. These results illustrate that the presence of sugar in the dough formulation affects dough aeration.

In agreement with Martin et al. (2004a) and Chin et al. (2003), the voidage following a pressure step increase changes less rapidly than the voidage following a pressure step decrease.
Figure 5.4 Experimental and model voidages during mixing with a pressure step increase from 1 bara to 2 bara. Each data point is from a separate dough mix. (a) strong flour, 0% sugar (b) weak flour, 0% sugar (c) strong flour, 7.5% sugar (d) weak flour, 7.5% sugar (e) strong flour, 15% sugar (f) weak flour, 15% sugar
Figure 5.4 Experimental and model voidages during mixing with a pressure step increase from 1 bara to 2 bara. Each data point is from a separate dough mix. (g) strong flour, 0% sugar (h) weak flour 0% sugar (i) strong flour, 7.5% sugar (j) weak flour 7.5% sugar (k) strong flour, 15% sugar (l) weak flour, 15% sugar
5.3.2 Pressure step change modelling results

This section discusses the model fitting to each of the six dough formulations, followed by the aeration parameters given by the model for the pressure step change dough mixes.

The model fittings are presented alongside the experimental results in Figures 5.2-5.4 located in Section 5.3.1 of this chapter. As expected when fitting a model to experimental results that exhibit some scatter and more so when cross-fitting a model to three sets of experimental results exhibiting some scatter, there is some deviation in the model fitting to the data sets. However, the model fittings appear to be representative of the experimental results and follow the same general trend as the experimental data. Differences in the model and experimental results include a tendency for the voidage to continue to increase at the end of the mixing session in the pressure step decrease models including on the occasions where the experimental data appears to reach a steady state. Another difference between the experimental and model results is the inaccuracy in the model's voidage prediction prior to the step change, which is largely due to the scatter in the experimental results. In Table 5.4, the RMSE for each of the model fittings are presented. The table shows that overall the model shows a good fit to the experimental data, despite differences in the dough formulations, indicating the model's applicability to the data. As expected a better fit is observed for direct fitting of the model to the results, compared to cross fitting. These can be seen on direct comparison of the figures illustrating the two different fittings of results.

### Table 5.4 RMSEs for the no breakup population balance fitted to the experimental pressure step change data

<table>
<thead>
<tr>
<th>Dough Formulations</th>
<th>Pressure Step Change</th>
<th>RMSE (cross fitted data)</th>
<th>RMSE (directly fitted data)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Strong, 0%</td>
<td>P1 = 1, P2 = 0.5</td>
<td>0.000583</td>
<td>0.000529</td>
</tr>
<tr>
<td></td>
<td>P1 = 1, P2 = 0.25</td>
<td>0.00113</td>
<td>0.00107</td>
</tr>
<tr>
<td></td>
<td>P1 = 1, P2 = 2</td>
<td>0.000253</td>
<td>0.000128</td>
</tr>
<tr>
<td>Weak, 0%</td>
<td>P1 = 1, P2 = 0.5</td>
<td>0.00106</td>
<td>0.000881</td>
</tr>
<tr>
<td></td>
<td>P1 = 1, P2 = 0.25</td>
<td>0.00158</td>
<td>0.00134</td>
</tr>
<tr>
<td></td>
<td>P1 = 1, P2 = 2</td>
<td>0.000393</td>
<td>0.00092</td>
</tr>
<tr>
<td>Strong, 7.5%</td>
<td>P1 = 1, P2 = 0.5</td>
<td>0.000252</td>
<td>0.000214</td>
</tr>
<tr>
<td></td>
<td>P1 = 1, P2 = 0.25</td>
<td>0.00116</td>
<td>0.00115</td>
</tr>
<tr>
<td></td>
<td>P1 = 1, P2 = 2</td>
<td>0.000197</td>
<td>0.000083</td>
</tr>
<tr>
<td>Weak, 7.5%</td>
<td>P1 = 1, P2 = 0.5</td>
<td>0.000767</td>
<td>0.000480</td>
</tr>
<tr>
<td></td>
<td>P1 = 1, P2 = 0.25</td>
<td>0.00296</td>
<td>0.00115</td>
</tr>
<tr>
<td></td>
<td>P1 = 1, P2 = 2</td>
<td>0.000334</td>
<td>0.000680</td>
</tr>
<tr>
<td>Strong, 15%</td>
<td>P1 = 1, P2 = 0.5</td>
<td>0.000515</td>
<td>0.000302</td>
</tr>
<tr>
<td></td>
<td>P1 = 1, P2 = 0.25</td>
<td>0.000727</td>
<td>0.000634</td>
</tr>
<tr>
<td></td>
<td>P1 = 1, P2 = 2</td>
<td>0.000152</td>
<td>0.000102</td>
</tr>
<tr>
<td>Weak, 15%</td>
<td>P1 = 1, P2 = 0.5</td>
<td>0.00192</td>
<td>0.000409</td>
</tr>
<tr>
<td></td>
<td>P1 = 1, P2 = 0.25</td>
<td>0.00324</td>
<td>0.00231</td>
</tr>
<tr>
<td></td>
<td>P1 = 1, P2 = 2</td>
<td>0.000413</td>
<td>0.000115</td>
</tr>
</tbody>
</table>
Figure 5.5 illustrates the initial voidage of the strong and weak flour doughs of different sugar contents, as predicted by the model. The results show an initial voidage ranging from 0.058 to 0.078 amongst the six dough formulations. These are reasonable and in the same magnitude as those in literature (Campbell and Shah, 1999; Whitworth and Alava, 1999; Martin et al., 2008). The results show an increase in voidage in both the strong and weak flour doughs as the sugar content increases. This differs to the voidage measurements of different sugar content doughs presented in Section 4.3 and Figure 5.1, which showed a decrease in voidage as dough sugar content increased. Inspection of Figures 5.2-5.4 explains why. The figures show a tendency to under predict initial voidages in dough containing 0% sugar, and over predict initial voidages in dough containing 15% sugar, with predictions of the voidage of the 7.5% sugar doughs being inbetween. Inspection of the figures show that this is not likely to be due to error in the model, but experimental error. The experimental results should show a consistent and steady voidage prior to the step change, rather than the decrease in voidage observed in Figure 5.2 (a) for example.

Figure 5.6 (a) and (b) show the entrainment rate and disentrainment coefficient of the six doughs, respectively. Note the difference in scale along the vertical axis between the figures for the entrainment rate and disentrainment coefficient - the disentrainment coefficient is consistently higher than the entrainment rate by a factor of 10 - 20. In Figure 5.6 (a) it can be seen that in the strong flour dough, the entrainment rate increases from 0.00114 rev⁻¹ to 0.00125 rev⁻¹ to 0.00139 rev⁻¹, as the sugar content increases from 0% to 7.5% to 15%. For the weak flour dough, as the sugar content increases from 0% to 7.5% to 15%, the entrainment rate decreases from 0.00113 rev⁻¹ to 0.000953 rev⁻¹, before increasing to 0.00128 rev⁻¹. It was expected that the same trend would have been expressed in doughs produced from the two flour types. The 7.5% weak flour dough does not fit in with the trend expressed by the other five dough formulations. Inspection of the results show a poor fit following the step change, for the pressure step increase experiment and the pressure step decrease from 1 bara to 0.5 bara. It is likely that the scatter amongst the later time in the pressure step decrease experiment has resulted in the poor model fitting of the pressure step increase experiment, which is largely responsible for giving the entrainment rate. Therefore it can be argued that the result for the entrainment rate for the 7.5% weak flour dough is not likely to be representative, and it is likely that entrainment is fairly constant, though increases a small degree as the sugar content increases.

A consistent trend can be observed in Figure 5.6 (b) which illustrates the change in disentrainment coefficient with sugar content. The disentrainment rate of gas in dough depends upon the disentrainment coefficient, cell size and quantity of gas in the dough. The figure shows an increase in the disentrainment coefficient as the sugar content increases in both the strong and weak flour doughs. This is also seen in Figures 5.2-5.4, where the model results show a steeper gradient following the step change in the sugar containing doughs. This suggests that as the sugar content of the dough increases cells are more likely to break up and become disentrained from the dough. The probability of cell break up increases when
cells are larger or when the dough structure is weaker. It is possible that either both or just the latter is the case, as it is suspected that the addition of sugar prevents the gluten in the dough from fully developing due to competition with the flour proteins for water. A more developed dough will have improved viscoelastic properties. Section 4.2 found sugar containing doughs to be less viscous, however. As the pressure step change generally causes a decrease in voidage in the sugar containing doughs, which is not observed in the non-sugar containing doughs, it is likely that the change in pressure further weakens the dough structure due to the stress applied on the dough, which would not have been observed following constant pressure mixing.

The ratio of the entrainment rate to disentrainment coefficient is the steady state voidage. This is presented in Figure 5.7 for the strong and weak flour doughs of different sugar contents. The results show a decrease in the steady state voidage as the sugar content increases amongst doughs produced from both flours, which Figure 5.6 shows is a result of the increase in disentrainment coefficient. The range of steady state voidages in the weak flour doughs is greater than the range in the strong flour doughs. These results suggest that the effect of sugar on steady state voidage is greater in weak flour doughs than strong. This can be expected due to the lower gluten content and therefore weaker structure of weak flour doughs, making retention of cells more difficult.

The steady state voidages for the no sugar dough found are of the same magnitude as the figures obtained by Martin et al. (2004) on application of the no breakup model to experimental data for the same size pressure step change in doughs of non-sugar containing doughs of differing formulation.

![Graph](image)

**Figure 5.5** Initial model voidages of strong and weak flour doughs containing 0%, 5% and 15% sugar
5.4 Summary

Chapter 5 set out to investigate the aeration of different sugar content doughs during mixing. Entrainment rates and disentrainment coefficients were calculated through application of a population balance model on different size and direction pressure changes. The investigation found that in non-sugar containing doughs pressure-vacuum mixing did not decrease the end voidage, and when mixing with a pressure step increase, the dough voidage increases over the mixing session. Therefore a negative voidage response was not observed when mixing with a pressure step change in non-sugar containing doughs. However, in the sugar containing doughs investigated, an increase in sugar content resulted in a lower steady state voidage following pressure-vacuum mixing, and when mixing with a pressure step increase, the increase in voidage observed decreased as the sugar content increased, to the extent that at 15% sugar dough voidage decreased over the mixing session. It is suspected that the compression of the cells during the step change reduces cell stability and makes cells more likely to rupture.

Figure 5.6 (a) Entrainment rate of strong and weak flour doughs containing 0%, 5% and 15% sugar (b) Disentrainment coefficient of strong and weak flour doughs containing 0%, 5% and 15% sugar

Figure 5.7 Ratio of entrainment rate to disentrainment coefficient for strong and weak flour doughs containing 0%, 7.5% and 15% sugar
The population balance model of aeration described the experimental data well, and highlighted a decrease in steady state voidage as the sugar content increased, indicating that dual pressure mixing reduces the voidage in sugar containing doughs, with a greater effect on weak flour doughs than strong. Entrainment rate was found to be fairly constant with sugar content, and the disentrainment coefficient showed that the reduction in steady state voidage was due to an increase in the disentrainment coefficient as the dough’s sugar content increased. For increased disentrainment of gas in the dough, cells must be more likely to rupture. Cell rupture involves loss of gas from the dough into the atmosphere. Disentrainment of gas occurs when cell rupture occurs at the edges of the dough. These results suggest the dough structure is weaker with increased quantities of sugar. Chapter 4 suggests this could be due to the gluten being less well developed, most probably due to the increased proportion of sugar and decreased proportion of other ingredients. Sections 4.2 and 4.8 also found sugar containing doughs to be less elastic, again suggesting that they are structurally weaker. Section 6.2 uses X-ray CT to assess the microstructure of sugared and non-sugared doughs, which may help better understand why disentrainment is more likely in sugared doughs.

This chapter concludes that pressure-vacuum mixing should not simply be extended from non-sugared to sugared doughs as these display different aeration characteristics during mixing. The chapter illustrates that the population balance model can be applied to sugared doughs as well as non-sugared doughs, thus demonstrating its suitability to a range of scenarios.
6.1 Introduction

It has been argued that mixing is the most important stage of breadmaking (Cauvain, 2000), as this is the stage when cells are initially incorporated into the dough. The cell volume distribution (number of cells and their volume) changes over the breadmaking process and the final cell volume distribution is responsible for the final bread’s voidage. This Chapter uses X-ray CT to investigate changes in dough cellular structure during mixing and proving. Understanding how variations in processing and ingredients affect the microstructure can help achieve better control of the final product structure.

Developing this technique to obtain clear and representative images of high resolution required trying out a number of X-ray sample set-ups. Tubing of different sizes and materials were investigated for use as a sample container during the scans, as well as different methods of obtaining a dough sample and inserting it into the tubing without damaging the dough piece. Published literature which has used conventional X-ray imaging to probe into the cellular structure of bread has illustrated the difficulty in obtaining a high resolution and handling the sample and setting up the scans to obtain representative samples. X-ray imaging has been used to probe into both the static (Whitworth and Alava, 1999; Bellido et al., 2006; Whitworth, 2008) and dynamic (Babin et al., 2006; 2008; Turbin-Orger et al., 2012) structure of bread dough due to its non-invasive and non-destructive nature, necessary for obtaining accurate information on a fragile material such as bread dough. X-ray studies using more reliable methods have been conducted using synchrotrons. Babin et al. (2006; 2008) investigated dough proving using synchrotron beamline BM05 at the European Synchrotron Radiation Facility (ESRF), Grenoble. Despite the use of superior imaging equipment, the voxel resolution of their scans was 15 µm$^3$. This is lower than the 10 µm$^3$ voxel resolution obtained by Bellido et al. (2006) using a medical X-ray source. Turbin-Orger et al., (2012) also investigated dough proving using synchrotron beamline BM05, obtaining a voxel resolution of 5 µm$^3$. This is the highest resolution reported for the 3D imaging of bread dough to date. Using a conventional X-ray source in the methods described below, voxel resolutions as low as 7.1 µm$^3$ were obtained on dough samples with an initial volume 2-5 times larger than those investigated using a synchrotron, therefore enabling better representation of the dough sample.

Following this introduction are three experimental sections based on the use of X-ray CT: Section 6.2 investigates the cellular structure of bread dough with and without sugar, with the aim of understanding why sugared doughs have a lower voidage. Section 6.3 investigates the transient behaviour of pressure-vacuum mixed doughs and uses cell sizes to assess the validity of a simplified population balance model. Section 6.4 investigates the changes in the
dough cellular structure during proving. The results from these three sections are then summarised in Section 6.5.

6.2 X-ray CT of non-yeasted dough with and without sugar

In this thesis an extensive investigation into the effect of sugar throughout the breadmaking process was conducted. This section continues to investigate the effect of sugar on bread dough, to help understand why the presence of sugar in the dough formulation resulted in dough of lower voidage. The cellular structure of two doughs are investigated: one containing 0% sugar and the other 15% sugar.

6.2.1 Method: X-ray CT of dough with and without sugar

The cell distribution of non-yeasted doughs containing 0% sugar and 15% sugar were compared. Table 6.1 shows the formulation of the two doughs used in this experiment. Both doughs were mixed in the Tweedy 1 for 230 s with a pressure step change from 1 bara to 0.5 bara after 110 s to mimic mixing in industrial breadmaking.

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>0% sugar</th>
<th>15% sugar</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flour c (11%)</td>
<td>400.00 / 60.7</td>
<td>400.00 / 55.6</td>
</tr>
<tr>
<td>Water</td>
<td>232.00 / 35.2</td>
<td>232.00 / 32.3</td>
</tr>
<tr>
<td>Sugar</td>
<td>0.00 / 0.00</td>
<td>60.00 / 8.34</td>
</tr>
<tr>
<td>Salt</td>
<td>7.20 / 1.09</td>
<td>7.20 / 1.00</td>
</tr>
<tr>
<td>Ascorbic acid</td>
<td>0.03 / 0.00349</td>
<td>0.03 / 0.00417</td>
</tr>
<tr>
<td>Vegetable fat</td>
<td>20.00 / 3.03</td>
<td>20.00 / 2.78</td>
</tr>
</tbody>
</table>

A dough piece was then carefully inserted into a glass tube. The tube had an internal diameter of 10 mm and a wall thickness of 0.5 mm. Cling film was stuffed into the top of the tube to minimise dehydration. The tube was wedged into some oasis foam which was held onto the X-ray sample holder using double sided sticky tape. The dough set up is illustrated in Figure 6.1.
Figure 6.1 Dough set up for X-ray imaging of static samples

Molybdenum was used as the target material and a current of 280 µA and a voltage of 50 kV used. The sample was rotated 360° and 2001 projections were obtained over approximately a 50 minute scan.

The voxel resolution of the 0% and 15% sugar dough was 9.06 µm³ and 8.83 µm³ respectively.

6.2.2 Results and discussion: X-ray CT of dough with and without sugar

In Figure 6.2 orthoslices of dough containing no sugar and 15% sugar, which were mixed for 230 s and subjected to a pressure step change from 1 bara to 0.5 bara at 110 s are presented. The voxel resolution of the scans differs by 0.23 µm³. Examination of the orthoslices shows two main differences between doughs: (1) cells in the 15% sugar dough are smaller. This lowers the contrast of the smaller cells to the dough matrix, reducing confidence in their identification, hence use of the voxel cut-off size. (2) In the 15% sugar dough, a greater quantity of wispy grey marks can be seen. These marks are not likely to be tiny folds in the dough, as folds in the dough have a different appearance and generally result in gas occlusion between the folds. It is possible that these marks are ingredients that have not been fully mixed in and broken up, as water quantities were not sufficient to mix in the solids. It is also possible that these are artefacts from having not allowed the dough to relax following mixing, or artefacts from reconstruction of the images.
The voidage derived from the double cup method and X-ray CT are presented in Figure 6.3. Both the voidage before and after the voxel cut-off size for the X-ray CT results are presented. According to all of the voidage measurements, the 0% sugar dough has a higher voidage than the 15% sugar dough. The double cup measured the voidage of the 0% sugar dough to be 3.75% and that of the 15% sugar dough to be 2.34%. This lower voidage of sugared doughs is in agreement with voidage measurements in Section 4.3 and Chapter 5. The results also show that the X-ray voidages are lower than the double cup voidages. This is likely to be a result of the contrast and resolution, although voidage variation amongst different areas of the dough is also likely to have an effect. The voidage difference before and after the cut-off vary from 6% in the 0% sugar dough to 17% in the 15% sugar dough, a reflection of the differences in the detected cell volumes in the two samples. Also, note that the X-ray voidage without the cut-off is closer to the double cup voidage than the X-ray voidage with the cut-off, suggesting that a number of the cell volumes not included in the count are from cells which are present, rather than noise. The voidage differences between the double cup method and X-ray method show that X-ray CT is capable of providing fairly accurate voidage measurements. More voidage comparisons through the two methodologies are made later in this chapter.
Figure 6.3 Dough voidage, measured by the double cup method and Avizo via X-ray CT, for dough containing 0% sugar and 15% sugar, which were each mixed for 230 s in the Tweedy 1 with a pressure step decrease from 1 bara to 0.5 bara at 110 s.

The cell number density in the two doughs above the cut-off size are presented in Figure 6.4. It is expected that use of the voxel cut-off will have a larger effect on the cell number than on dough voidage, and this can be seen on comparison of Figure 6.4 to Figure 6.3. Thus although these results have been illustrated, in reality they represent the minimum number of cells per mm$^3$ detected in the two doughs.

Figure 6.4 shows the 15% sugar dough contained 70 cells mm$^3$, which is 30% less cells mm$^3$ than the non-sugar dough, which contained 100 cells mm$^3$. This is greater than the cell number density observed by Bellido et al. (2006) via X-ray CT, who found 30-56 cells mm$^3$ in their two doughs studied and achieved a voxel resolution of 10 µm$^3$. Differences in dough formulation, mixer choice and sample preparation are expected to result in differences in cell number. The mixer used in Bellido et al.’s study operated at a constant pressure, rather than pressure-vacuum, therefore in theory should have occluded fewer cells over the mixing time. When preparing the sample, Bellido et al. (2006) squashed their 0.5 g sample between two plates and anisotropy analysis suggested that dough structure was altered. In addition it is possible that squashing of the small sample size resulted in some cell rupture.

Figure 6.4 Number of cells above cut-off per cm$^3$ for dough containing 0% and 15% sugar, which were each mixed for 230 s in the Tweedy 1 with a pressure step decrease from 1 bara to 0.5 bara at 110 s.
As mentioned previously in this thesis, the addition of sugar to bread dough decreases the water concentration of the dough. Upadhyay et al. (2012) used CLSM to investigate the mean cell diameter of doughs in a range of dough formulations and found that on decreasing the water concentration from 80% to 40%, cell diameters decreased from 22.8 µm to 16.8 µm. The addition of 15% sugar to the dough formulation would cause a 2.4% decrease in water concentration, which if in agreement, should see a decrease in mean cell size.

By using a cell cut-off size, results showing the mean cell volume in dough samples are likely to be inaccurate, due to exclusion of cells below the cut-off size. The mean cell volume for the 0% and 15% sugar doughs are presented in Figure 6.5. The results show the 0% sugar dough to have a 17% larger volume than the 15% sugar dough. The 0% sugar dough was found to have a mean cell volume of 330000 µm³ and the 15% sugar dough was found to have a mean cell volume of 280000 µm³. On backwards calculation from the volume of a sphere, cells within the 17% sugar dough have a mean diameter 2.8 µm larger than the non-sugar containing doughs. This larger cell diameter of the non-sugar containing dough is in agreement with Upadhyay et al. (2012). A lower mean cell volume may be the cause of the increased proving time seen in sugared doughs in Section 4.10. In addition, a lower cell volume suggests the cells in sugared doughs breakup more readily and thus gas is more likely to be lost from the dough. This is supported by application of the population balance model to the strong flour doughs in Chapter 5 showing an increase in the disentainment coefficient in strong flour doughs as the sugar content increased.

![Figure 6.5 Mean cell volume above cut-off per cm³ for dough containing 0% and 15% sugar, which were each mixed for 230 s in the Tweedy 1 with a pressure step decrease from 1 bara to 0.5 bara at 110 s.](image)

Figure 6.6 shows the cell size distribution in the 0% and 15% sugar dough. The cell volume distribution above the voxel cut-off size for the two doughs is illustrated in Figure 6.6(a). In Figure 6.6(b) the frequency of cells in the whole analysed sample size is illustrated. Figure 6.6 shows that as the cell volume increases, the number of cells rapidly decreases. It had been observed on numerous occasions when handling dough that fewer larger cells exist in doughs than smaller cells, so this was expected. Figure 6.6(b) illustrates that some data points are comprised from just one cell. The results also show a larger cell number density.
per unit volume in the smallest eight categories of cell volumes in the 15% sugar dough than the 0% sugar dough. Following this, there are fewer smaller cells in the 15% sugar dough.

The cell size distribution disagrees with the cell distribution Bellido et al. (2006) found through micro CT. The authors did not find a decrease in cell number as cell size increased. The authors found the cell size distribution was skewed towards the smaller cells with cell number density greatest at a cell size of approximately 70 µm in diameter. The authors reconstruction of cell sizes from individual orthoslices rather than thresholding the dough sample will have affected their measured diameters. Even with use of a voxel cut-off size in Figure 6.5, larger cell number densities were found at smaller cell sizes. It is likely that their method of sample preparation resulted in the rupture of a significant number of cells that were below this size and also rupture of larger cells.

![Graph](Figure 6.6(a)) The number density per unit volume of different cell volumes (b) The frequency of different volume cells in a 0% and 15% sugar dough following mixing for 230 s in a Tweedy 1, with a pressure step decrease from 1 bara to 0.5 bara at 110 s
In Figure 6.7 the cumulative percentage of cells above the cut-off is illustrated for the different vOLUMed cells in the dough containing 0% sugar and 15% sugar. The results show a larger proportion of smaller cells in the 15% sugar dough and a similar cell volume distribution for cells greater than $10^5 \ \mu m^3$ in the two doughs. In addition, Figure 6.6 illustrates that although cell volume ranges from $6 \times 10^4 - 10^9 \ \mu m^3$, 98% of the cells are below $10^6 \ \mu m^3$ in volume.

The results do not show a greater proportion of larger cells in the 15% sugar dough than the non-sugar containing dough, suggesting that the increase in disentainment coefficient with sugar as seen in Chapter 5 is not due to the presence of larger cells but a weaker dough structure making cell rupture more likely to occur.

![Figure 6.7 Cumulative number of cells % above cut-off in 0% and 15% sugar dough following mixing for 230 s in a Tweedy 1, with a pressure step decrease from 1 bara to 0.5 bara at 110 s](image)

Figure 6.8 shows the cumulative voidage of different volume cells in the 0% sugar and 15% sugar doughs in both cells per unit volume in Figure 6.8(a) and percentage in Figure 6.8(b). Figure 6.8 (a) shows a larger voidage in the 0% sugar dough than the 15% sugar dough, and a larger range of cell volumes in the 0% sugar dough. Figure 6.8 (b) shows the cell volume distribution as a percentage cumulative voidage is similar amongst the two doughs.

The representativeness of the larger cells can be improved upon. At the larger end of the mean cell volume sometimes just one cell of this volume existed, thus affecting the full range of cell volumes. In a larger sample size or with repeat experiments the illustrated results would be smoother.
The results in Figures 6.7-6.8 show that although the smallest cells make up the largest proportion of cells, their contribution towards the dough voidage following mixing is small, compared to larger cells. 98% of the cells are below $10^6$ µm$^3$ in volume. However, in the 0% sugar dough, these 98% of cells only make up 61% of the voidage and in the 15% sugar dough, they only make up 56% of the voidage. The effect on voidage of smaller cells also justifies using a voxel cut-off point to increase confidence levels.

6.3 X-ray CT of non-yeasted non-sugar containing bread dough throughout mixing with a pressure step change

Pressure step changes in the form of pressure-vacuum mixing are used in the bread industry to favourably manipulate the cell size distribution in the final bread. The theory is that the pressure drop expands the cells, allowing them to be subdivided. However, this theory has not beenproved. Obtaining the cell size distribution in a dynamic process is essential for understanding and improving control of opaque fluids such as bread dough. In this section, the cell size distribution throughout a pressure-vacuum mixed dough is compared to the cell size distribution in a control to test if the theory is true. The cell size distributions are also used to test the validity of the simplified no breakup population balance model, which makes a number of invalid assumptions.

6.3.1 Method: X-ray CT of bread dough without sugar throughout mixing with a pressure step change

X-ray CT was conducted on five non-yeasted pressure-vacuum mixed doughs and one constant pressure mixed dough. The dough formulations were identical amongst the six doughs, which varied in their processing. Once samples were obtained from each dough, the dough was discarded, and fresh batches made for the outstanding mixing times. For the pressure-vacuum
mixed doughs, their times under vacuum varied. Mixing was carried out in the Tweedy 1 at 1 bara for the first 110 s and then at 0.5 bara for the remainder of the mixing time, with total mixing times of 110 s, 130 s, 150 s, 180 s and 230 s, to measure transient behaviour. A dough mixed at a constant pressure of 1 bara for 230 s was made as a control. The six dough formulations and their mixing times are given in Table 6.2.

**Table 6.2 Formulations and mixing times used for comparison of pressure-vacuum and constant pressure mixed doughs via X-ray CT**

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>Quantity (g / %)</th>
<th>Mixing time (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flour c (11%)</td>
<td>400.00 / 60.7</td>
<td>110, 130, 150, 180, 230 - pressure-vacuum mix</td>
</tr>
<tr>
<td>Water</td>
<td>232.00 / 35.2</td>
<td>230 - constant pressure mix</td>
</tr>
<tr>
<td>Vegetable fat</td>
<td>20.00 / 3.03</td>
<td></td>
</tr>
<tr>
<td>Salt</td>
<td>7.20 / 1.09</td>
<td></td>
</tr>
<tr>
<td>Ascorbic acid</td>
<td>0.03 / 0.00455</td>
<td></td>
</tr>
</tbody>
</table>

The current range for the six samples was 200-285 uA and the voltage range was 50-65 kV. The sample was rotated 360° and 1801 projections were obtained over approximately a 45 minute scan. On reconstruction of the dough, the voxel resolutions and sample sizes presented in Table 6.3 were obtained. The 5 voxel in diameter cut-off equated to sample diameters of 35-54 µm in the 5 samples.

**Table 6.3 Sample volumes and resolutions for X-ray scans**

<table>
<thead>
<tr>
<th>Dough</th>
<th>Resolution (µm³)</th>
<th>Sample volume (cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>110 s</td>
<td>10.8</td>
<td>1.26</td>
</tr>
<tr>
<td>130 s</td>
<td>8.4</td>
<td>1.12</td>
</tr>
<tr>
<td>150 s</td>
<td>9.0</td>
<td>1.32</td>
</tr>
<tr>
<td>180 s</td>
<td>7.1</td>
<td>1.26</td>
</tr>
<tr>
<td>230 s</td>
<td>7.1</td>
<td>1.08</td>
</tr>
<tr>
<td>230 s, no step change</td>
<td>8.6</td>
<td>1.22</td>
</tr>
</tbody>
</table>

The voidages obtained from X-ray CT were input into the no breakup population balance model described in Chapter 5, and the model was then fitted to the experimental results to assess its applicability to the scenario given the simplifying assumptions made. The chosen entrained bubble size was selected to give model agreement to the data at time 110 s.

**6.3.2 Results and discussion: X-ray CT of bread dough without sugar throughout mixing with a pressure step change**

Figure 6.9 show orthoslices for doughs mixed at a constant pressure of 1 bara for 110 s and 230 s, and a pressure vacuum mixed dough that was mixed for a total of 230 s and underwent a pressure step decrease from 1 bara to 0.5 bara at 110 s. The cell size distribution for the 110 s and 230 s 1 bara cases look similar at first inspection. However, it appears that the
dough which experienced a step change has a significantly different cell size distribution - with fewer and possibly smaller cells.
Figure 6.9 Orthoslices of dough with highlighted region size of 60 pixels square (a) Dough mixed for 110 s at constant pressure of 1 bara. Pixel resolution is 10.8 µm. (b) Dough mixed for 230 s at constant pressure of 1 bara. Pixel resolution is 8.6 µm. (c) Dough mixed for 230 s in total: first 110 s at 1 bara, and final 120 s at 0.5 bara. Pixel resolution is 7.1 µm.
Figure 6.10 illustrates the measured differences in voidage amongst the six dough samples with and without a cut-off. With exception of the constant pressure mixed control sample where the measured voidages differed by 11%, the voidage of the other five samples with and without a cut-off were within 6% of each other. The larger voidage difference observed in the 230 s constant pressure mixed dough is due to a larger proportion of the voidage being comprised from cells smaller than the cut-off. The 110 s constant pressure mixed dough also has a larger proportion of its voidage comprised from cells smaller than the cut-off size than the pressure-vacuum mixed doughs.

Whilst the effect of using a cut-off has a small effect on voidage, parameters which depend upon the number of cells will be affected, and it must be remembered that these parameters are above the cut-off point.

![Graph](https://via.placeholder.com/150)

**Figure 6.10 X-ray measured dough voidage of pressure-vacuum mixed dough and constant pressure mixed dough**

In Figure 6.11 the cell volume distributions for the doughs are the different mixing times are presented. Figure 6.11(a) presents these are the cell number density and figure 6.11(b) as the number of cells in each scanned dough sample. The figures show a decrease in the number density of cells per unit volume as the mean cell volume increases, with some of the data points representing the mean cell volumes being comprised from just one cell. No trend in the cell volume distribution is noted prior to cells of the mean volume of $10^6 \, \mu m^3$. For cells greater than this in volume, however, it can be seen that the trend shifts to the left as the mixing time increases. As the mixing time increases, the number density of cells per unit volume decreases in the pressure-vacuum mixed doughs with time. It was hypothesized that this would occur due to cell breakup. The control dough, although containing a greater number of cells, has a similar cell volume distribution to the other constant pressure mixed dough, which was mixed for 110 s.
Figure 6.11 (a) The number density per unit volume (b) The frequency of different cell volumes within dough throughout pressure-vacuum mixing. The doughs were mixed at 1 bara for the first 110 s and 0.5 bara thereafter.

In Figures 6.12 and 6.13 the cumulative percentage of cells and voidage are presented against mean cell volume respectively, to further illustrate the cell volume distribution. Figure 6.12 shows a change in the cell volume distribution throughout mixing, with the trend shifting to the left roughly with time, and similarity in the distribution of the two constant pressure mixes. Figure 6.12 also illustrates that throughout mixing approximately half of the cells are of a volume of approximately $10^4 \, \mu m^3$ despite the maximum detected cell volume being approximately $10^{10} \, \mu m^3$. It is beneficial at this stage of the breadmaking process to have a significant proportion of cells of a smaller volume, to delay coalescence and produce bread of desirable qualities. The cell size distributions are typically sigmoidal in shape in agreement with Turbin-Orger et al. (2012).
In Figure 6.13, the higher voidage in the constant pressure mixed doughs compared to the pressure-vacuum mixed doughs is clear. The two doughs show a similar voidage distribution profile. In addition, it was noted above that half of the cells are smaller than $10^4 \, \mu m^3$ in volume. Figure 6.13 shows that despite them making up a significant proportion of the cells, their voidage contribution is less than 10% in all of the mixes. This emphasises the importance of the larger cells in the dough voidage contribution at this stage of the breadmaking process. This is in agreement with Sapirstein et al., (1999). The authors noted that the majority of cells are smaller in size than the mean cell size within the dough. Campbell et al. (1991), also noted the small contribution of smaller cells towards the dough voidage.

In Figure 6.13, some of the data points amongst the largest cell volumes are comprised of a single cell. Note that in this experiment a single large cell alone does not skew the dough voidage. This suggests that for the investigation of cell volume distribution following mixing, the sample sized used is suitable. However, the large gap in the cell volumes observed amongst the larger dough sizes suggests that the full range of larger cell sizes is not detected in this sample size, and it would be beneficial to increase the sample size or carry out repeats.

*Figure 6.12* Cumulative cell percentage for different volume cells within dough throughout mixing at 1 bara for the first 110 s and 0.5 bara thereafter, and throughout a constant pressure of 1 bara for 230 s.
Figure 6.13 Cumulative voidage for different cell volumes within dough throughout mixing at 1 bara for the first 110 s and 0.5 bara thereafter, and the control, which was mixed at a constant pressure of 1 bara.

Figure 6.14 shows the measured and modelled number of cells per volume of dough at different mixing times during constant pressure mixing and pressure-vacuum mixing. The number density ranged from 57 to 120 cells per mm$^3$; these are the same order of magnitude as those reported by Dobraszczyk et al. (2001), Martin et al. (2004a) and Bellido (2006). The results show a decrease in the cell density immediately following the pressure step-decrease, then increase again above the initial level by the end of the mix, with the cell density being lower than that for the constant pressure mixed dough at 230 s.
Figure 6.15 illustrates the model fitting to the dough voidage, and the voidage of the pressure-vacuum mixed dough in Chapter 5 (the flours used in the dough formulations differ). It is presented to illustrate how the voidage of the dough outside the mixer changes throughout pressure-vacuum mixing, as limited resources meant the voidage limited the number of points for which data collection via X-ray CT took place. It shows that on application of a vacuum, gas is drawn from the dough causing the voidage to decrease rapidly, until about 140 s when the voidage begins to level off. The voidage of the dough decreases prior to drawing a vacuum and earlier than expected in this dough. This is likely to be due to anomalous results from an unrepresentative mixing pattern session in the Tweedy mixer.

In Chapter 5, both strong and weak flour doughs were pressure-vacuum mixed, and the results showed a shared trend in the two flour types. This indicates that the effect of pressure-vacuum mixing is expressed amongst different flour types, and thus the dough from Chapter 5 can be used to represent the trend which will occur during pressure-vacuum mixing of the X-ray dough. Differences are expected amongst the voidage in the doughs and the extent of the effect of drawing a vacuum.

Figure 6.15 shows the voidage of the different doughs. It shows the voidage of the pressure-vacuum mixed X-ray dough to be lower than the voidage of the strong flour dough from Chapter 5 and the change in dough voidage between the doughs share the same trend. Note at $t = 180$ s and $t = 230$ s, the double cup and X-ray voidages measured are from different batches of dough, with a slight difference in formulation. Measured % differences between voidages of dough from an identical batch found the measurements to be within 7% of each.
other. The shared trend and closeness in voidage between the two methods gives confidence in the X-ray CT methodology. The control shows a different voidage to the pressure-vacuum mixed doughs and a higher voidage than all doughs. It was expected that the control would show a higher voidage as it was mixed at a higher pressure for longer, allowing more gas to be entrained into the dough. However, the voidage of the control dough is higher than expected. This may be due to a variety of reasons including the voidage variation from one batch of dough to another, the variation in voidage throughout the dough, the age of the dough causing it to behave differently, and the dough following a different mixing pattern in the Tweedy mixer. The trend in voidage change expressed in X-ray CT measurements reflects the voidage change which occurs throughout pressure-vacuum mixing and shows the sensitivity of the X-ray CT methodology to these changes.

Differences in voidage between the double cup voidage measurements and X-ray voidage measurements may be due to a number of reasons. It is expected that voidage measurements obtained through X-ray CT are less accurate due to the smaller sample size, use of just one sample to measure voidage as opposed to six, more handling and the effect of labelling at different thresholding levels.

In Figure 6.15, the model was fitted to the data by selecting a suitable volume for entrained cells (700516 µm$^3$, equivalent to a spherical cell of diameter of 109 µm) which meant the voidage such that the model and voidage measurements agreed at mixing time 110 s. The value chosen was the mean cell volume at 110 s. The model trends for mixing times after this point are only dependent on the parameters fitted to the dough voidage data - the model was not fitted to the number density or mean cell volume. With this in mind, the fit of the model is encouraging as it displays the same trends. In addition, the RMSNE for the model fitting to the experimental results was 0.549. Note the model under predicts the number density. This may be a result of neglecting any cell break-up.
Figure 6.15 Dough voidage outside mixer at different mixing times, with X-ray CT and double cup measurements, and fitting of the no breakup population balance model of aeration to the experimental results. Dough was either mixed at a constant pressure of 1 bara or pressure-vacuum mixed for a total of 230 s, with a pressure step decrease from 1 bara to 0.5 bara at 110 s.

Figure 6.16 shows the mean and modelled cell volume in the pressure-vacuum mixed doughs and the control dough. The measured results show a decrease in the mean cell volume from 680000 µm$^3$ at 110 s to 150000 µm$^3$ at 180 s, before levelling off. The results show the mean cell volume decreases as a result of pressure vacuum mixing, and also show a smaller mean cell volume than the constant pressure mixed dough that had a mean cell volume of $4.1 \times 10^5$µm$^3$. In addition, the results show a decrease in the measured mean volume for constant pressure mixed dough, but to a lesser extent. The results support the theory that pressure-vacuum mixing leads to a finer cell distribution. The model agreement to these trends is very encouraging - displaying the same trends and similar magnitudes. The model over predicts the mean cell volume and again this would be consistent with cell break-up occurring during mixing which has been neglected in the model.
Good agreement is shown between the dough voidage, cell number density and mean cell volume for the model, although simplifying assumptions such as a small number of discrete cell sizes were made. The model predicts the significant processing effect of vacuum mixing, showing the reduction in cell number density and volume, using just dough voidage measurements. It demonstrated that the decrease in number density is much more short lived than the decrease in size which is permanent. The model is fitted to voidage data using only three parameters, which are inexpensively obtainable from dough density measurements. Therefore the no cell breakup model has been shown to provide a practical and reasonably accurate characterisation of pressure vacuum mixing, and confirms that the simplifying assumptions made in the model are reasonable. This enables use of the model to estimate aeration rates, oxygen availability and optimal time for pressure-vacuum mixing to achieve the desired final cell size distribution in bread dough.

6.4 X-ray CT of bread dough during proving

During each stage of the breadmaking process, aeration processes occur that changes dough’s structure. Proving largely forms the cellular structure of bread dough (Dobraszczyk et al., 2001), a result of carbon dioxide produced from yeast diffusing into cells. During this dynamic process, the cell size distribution changes through their cell growth, interaction and rupture, making the process interesting to study. Observations of this process also enable improved understanding of the proving process to help obtain the desired final cellular structure. In addition, the dynamic results were to be used for development of a faster reconstruction algorithm.

Figure 6.16 Mean cell volume at different mixing times, with X-ray CT results and fitting of the no breakup population balance model of aeration to the experimental results. Dough was either mixed at a constant pressure of 1 bara or pressure-vacuum mixed for a total of 230 s, with a pressure step decrease from 1 bara to 0.5 bara at 110 s.
6.4.1 Method: X-ray CT of bread dough during proving

Dough was made up in a kitchen planetary mixer to the formulation shown in Table 6.4, and analysed through X-ray CT.

Table 6.4 Dough formulation used for X-ray CT of bread dough proving

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>Quantity (g / %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flour e (15%)</td>
<td>200.0 / 58.0</td>
</tr>
<tr>
<td>Salt</td>
<td>3.6 / 1.04</td>
</tr>
<tr>
<td>Water, 38°C</td>
<td>133.0 / 38.6</td>
</tr>
<tr>
<td>Quick yeast</td>
<td>2.0 / 0.58</td>
</tr>
<tr>
<td>Sugar</td>
<td>6.0 / 1.74</td>
</tr>
</tbody>
</table>

The dry ingredients were mixed together for 30 s on mixer setting 1 to ensure thorough distribution of the ingredients. Water was then added over a period of 30 s and the mixer speed setting increased to 2. The dough was then mixed for a further 5 minutes. A dough piece approximately 1 g in weight was carefully cut from the dough using a razor blade. It was rounded to smooth out any damaged edges and coated in sunflower oil to prevent it from drying out before being put into a plastic tube glued onto a sample holder. Cling film was used as a bung at the top of the tube to stop the sample from drying out. This set up is illustrated in Figure 6.17.

![Dough set up for X-ray imaging during proving](image)

Figure 6.17 Dough set up for X-ray imaging during proving

The sample was placed on the Nikon Metris 225/320 kV CT system rotation stage and imaged in a customised bay. The sample to source distance was 51 mm and the sample to detector distance was 1007 mm. The temperature in the custom bay was 21.5°C. This is lower than industrial proving temperatures, but had to be maintained for complete functioning of the
equipment. This temperature is also advantageous for X-ray CT of dough proving, as the changes in the dough occur over a longer duration, thus fewer changes occur over each scan time, resulting in clearer images and enabling easier reconstruction of the data set.

During analysis the sample was rotated 360° for 1.5 minutes, collecting 400 projections per scan with a voxel size of 10.2³ µm³. The experiment took place over 150 minutes, acquiring a new scan every 5 minutes. This duration was to ensure sufficient time to document changes and took into account the proving temperature. For the X-ray CT analysis an accelerating voltage of 65 kV, current of 400 µA, exposure time of 250 ms, target material of tungsten and a gain of 2 was used for each projection.

For development of a fast reconstruction algorithm, two dough samples were imaged under this set up and another two with a similar set-up, collecting 800 projections over a 3 minute scan.

The data set with 400 projections per scan was used for visualisation of proving, due to proving being a dynamic process and thus this data set should be less blurred than the data set with 800 projections per scan (which has the advantage of capturing more detail). The images were then reconstructed on Nikon Metris CTPro 3D (Metris XT 2.2, version 2.2.4365.28608) and the voxel size of 10.2 µm³ obtained. As with Section 6.1 and Section 6.2, it is difficult to identify with confidence features at the lower end of the resolution. However, as the dough is monitored over time, all of the identified cells have been included for analysis. Following reconstruction, the data was processed and analysed in Avizo Fire 7.1 (Visualization Sciences Group, Bordeaux, France). Not every scan was analysed, due to the large number of scans conducted and the requirement of sufficient time between scans to observe changes in the bread dough. The scans were analysed for dough volume and proportion of cells present at the following times: 0, 5, 10, 20, 30, 40, 50, 60, 70, 80, 90, 100, 110, 120, 130, 140, 145 minutes. More detailed information of different features of the dough’s cellular structure was then acquired at the appropriate times. This quantitative information includes cell volume distribution, cell number density, mean cell volume, cumulative cell percentage and cumulative voidage per unit volume. Cell number density is used to take into account the differences in sample sizes. The distribution of cell sizes is also presented to enable any cells that skew the data to be identified.

Two filters were applied to the data, a median filter followed by an anisotropic diffusion filter. The median filter is a noise reduction filter. It operates by sorting voxels according to their grayscale value. The anisotropic filter is a smoothing filter. It preserves strong edges of voxels and enhances their contrast. Following filtering the cells and dough matrix were segmented based on their greyscale values, and Material Statistics run on the segmented dough to quantify cell and dough matrix proportions. The quantification tool was used for calculation of cell numbers and volumes, and their Feret shape. Feret shape is defined as 1/minimum diameter divided into the maximum diameter at 90°. Feret shape and cell volumes and numbers were processed in Microsoft Office Excel, where cells were grouped.
according to volume, to give the cell volume distribution, and the voidage contribution of the groups of cell volumes calculated.

In addition, the yeast activation time was determined by mixing 2 g of the yeast with 133 g of water that was at 38°C. The time required to observe bubbling in the mix was determined as the yeast activation time.

6.4.2 Results and discussion: X-ray CT of bread dough during proving

The purpose of the work in this section was for development of a fast reconstruction algorithm for 4D scans and to conduct a comparison illustrating the benefits of the fast reconstruction algorithm compared to the standard reconstruction algorithm. Data was obtained and the algorithm was to be developed using this data by a research group within the School of Mathematics at the University of Manchester. This data has been given to the research group and is still in development. If successful, it will enable reconstructions in less time through use of every n

The yeast activation time was determined as 5 minutes. Mixing of the dough took 6 minutes. Including the set up time, the hydration time of the yeast on running the scan was 7 minutes, meaning the yeast were active on running the scan.

Figure 6.18 shows the change in dough volume as it proves. In the first 20 minutes, a slow increase in dough volume is observed. The dough volume increases from 544 mm\(^3\) to 555 mm\(^3\). The rate the dough volume increases then accelerates, before peaking and most likely levelling off at 1293 mm\(^3\) at 140 minutes, although it is difficult to say for certain as this trend is only seen from one data point. The volume increases more than 100\% over the proving time due to expansion of cells within the dough through yeast carbon dioxide production. The increase in volume is slow initially due to the requirement of larger pressures to increase the small cell volumes. As the cells increase in volume, it becomes easier for the pressure inside the cell generated through carbon dioxide production to overcome the pressure outside the cell, resulting in quicker cell expansion. As the cells grow, interaction with neighbouring cells through coalescence and disproportion occurs, resulting in fewer but larger cells. However, their stability in the dough is reduced as they increase in volume, hence the levelling off in volume observed from 140 minutes. The likely levelling off is also likely to be due to the doughs viscoelastic properties having reached its limit, therefore preventing further expansion of cells. The large cell volume, their proximity to the dough edges and the thinner cell walls seen in Figure 6.20(l), the orthoslice image at the end of the scan, support this. Had more scans been conducted and the results analysed, the dough volume most likely would have decreased soon after. The dough reaching its minimum density indicates overproving. To obtain a high volume loaf, the minimum density should be attained during baking. If the proved dough was baked, there would have been no expansion capacity left for oven spring and a large proportion of cells in the dough would rupture prior to the structure being set. This would cause significant loss of gas to the atmosphere and result in collapse of the loaf.
In Figure 6.19, the dough voidage over the proving time is illustrated. It shows a slow increase in the proportion of cells in the first 20 minutes, with the voidage increasing from 2.7% to 4.9%. The proportion of cells then increases up to 130 minutes and levels off at 66%. The results suggest the dough was over proved. At 145 minutes, 66% of the dough volume is gas, yet bread is approximately 70% gas and baking is known to be responsible for more than 4% of the gas in bread. The trend in Figure 6.19 is as expected and was previously explained in reference to Figure 6.18. The observed trends in voidage change over proving match those obtained by Babin et al. (2006) and Shehzad et al. (2010).

The trend observed in Figure 6.19 was expected to match the trend in Figure 6.18, due to changes in the proportion of cells in the dough being responsible for the change in dough volume. Two differences are expressed in the trends in Figures 6.18 and 6.19: the time the peak occurs and the smoothness of the curve. In Figure 6.18 the peak occurs at 140 minutes, whereas in Figure 6.19 it occurs at 130 minutes. There are two possible reasons for this discrepancy. The first being that between 130 and 145 minutes, cell growth and breakup occur at the same rate. The second is segmentation errors, although this is unlikely due to the voidage at these times. However, the segmentation errors are likely to be responsible for Figure 6.18 being smoother than Figure 6.19. These errors are created from the method of determining volume and voidage. Volume measurements are more accurate than voidage measurements, due to the cells being selected from the dough volume. As the voidage changes between scans, the grayscale levels within each image changes, so thresholding to the same grayscale levels was not feasible. This meant the cells were prone to over and under thresholding. In Table 6.5, the % error in voidages given by Avizo at different proving times, when using a thresholding value of +/-2 of the value used, are given. The errors were determined intermittently to illustrate its changes relative to proving times. Errors decrease as the proving time increases due to the increase in the cell to dough matrix ratio, and increase in cell volume creating a greater contrast to the dough matrix.
Figure 6.19 Change in dough voidage with proving time. The dough was mixed in a conventional plenary kitchen mixer and proved at 21.5°C.

Table 6.5 Percentage error at different thresholding levels throughout dough proving

<table>
<thead>
<tr>
<th>Time (minutes)</th>
<th>% error when using thresholding value +2</th>
<th>% error when using thresholding value -2</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>10.90</td>
<td>8.90</td>
</tr>
<tr>
<td>40</td>
<td>11.60</td>
<td>9.35</td>
</tr>
<tr>
<td>70</td>
<td>6.76</td>
<td>5.88</td>
</tr>
<tr>
<td>145</td>
<td>2.24</td>
<td>2.08</td>
</tr>
</tbody>
</table>

Non-filtered orthoslices at different stages of proving are presented in Figure 6.20. These are one dimensional images of the dough at different times during the scan. The same orthoslice number is presented in each figure to more effectively illustrate changes in the dough. These are organised chronologically from left to right and top to bottom. The volume of the dough and proportion of cells within it increases with time. A perspective of the extent of volume increase can be seen by comparison of the dough volume against the straw. Initially, approximately a sixth of the straw is in contact with the dough and the dough largely consists of dough matrix. Approximately half way through the scan, contact is made with the whole straw perimeter, and by the end of the scan, the dough largely consists of gas. The orthoslices also show an increasingly irregular cell shape with time, in agreement with Babin et al. (2006; 2008). The figures show that the cells do not have a preference for location with the centre of each dough piece. Figure 6.20(a) shows fewer cells than all the prospective times. None of the cells appear to be in direct contact with each other. From Figure 6.20(a)-6.20(d), growth, an increase in the number of cells and their proximity, can be observed. As yeast cannot generate new cells (Baker and Mize, 1941), these cells must have been present at the start of the proving, but below resolution size. The resolution of the scan was 10.2 µm. From this we know that cells smaller than 10.2 µm in diameter are entrained by the dough, and that they grow and contribute towards the final dough voidage. This also means voidage measurements are underestimated, as all the cells within the dough were not detected. However, it is likely that prior to their expansion, cells below 10.2 µm in diameter make a negligible contribution towards the dough voidage. This is in agreement with Babin et al.
who found that growth of initially detected cells has a significantly greater voidage contribution than growth of previously undetected cells during proving.

The changes between the orthoslices at different proving times become greater with time, a result of yeast carbon dioxide production and cell kinetics. This makes it challenging to track individual cells between scans as time proceeds and the dough structure rapidly changes. The orthoslices illustrate the rapid and significant changes in structure from 30 to 110 minutes and the interaction of larger cells with each other after 110 minutes.

Examination of the cells seen in Figure 6.20(i) and 6.20(j), orthoslices obtained within 5 minutes of each other at a highly dynamic stage of proving, illustrates some of the cell mechanics. Changes in cell volume and number can be seen through cell growth and interaction. Some cells within these figures have been labelled to further illustrate some changes which occur. Cells A and B are examples of cells that coalesce. Cell C illustrates the non-spherical shape of a cell and the changes observed over 5 minutes are likely to be a result of the changes in dough volume altering the cell's position within the dough. Cell D is likely to have broken up, hence its presence in Figure 6.20 (i) and not (j). Cells breakup under high strain conditions on the cell wall and when the viscoelastic properties of the dough are no longer sufficient to retain the gas. Coalescence and cell breakup occur throughout proving, and in particular at later stages of proving when the cells are close enough to interact with each other, larger and thus less stable and more prone to change. Note that in support of the views of Dobraszczyk et al. (2001), disproportionation was not observed amongst these orthoslices. It is highly likely that if disproportionation did occur within cells, it would have been noticed amongst these orthoslices. Note also that no mention of observations of disproportionation have been made in published work where dough proofing was visualised.

Figures 6.20(k) and 6.20(l) illustrate a number of very large cells within the dough and some located within the dough perimeter. This extent of proving is undesirable in breadmaking. Here the dough consists mainly of gas in large cells and some support from the surrounding dough matrix. The fact that this dough contains a large quantity of gas contained within mainly large cells suggests it has high strain hardening properties and is good for breadmaking. Note also the polyhedral cell shapes seen in Figures 6.20(k) and (l). Figure 6.20(k) contains 60% gas. Vliet et al. (1992) noted that above 70% gas, cells begin to deform each other strongly and start taking on a polyhedral shape. It is likely that there will be some variation in this figure, depending upon the dough’s rheology.

Dough orthoslices can also be used to track the position of cells throughout dynamic processes. Work on tracking individual cells to monitor their growth, change in position, orientation, shape and interaction with neighbouring cells was conducted using visualisation tools by an individual from The University of Manchester’s School of Material Sciences. However, this work was not complete at the time of writing this thesis.
In Figure 6.21, two dimensional images of the dough at different proving times are illustrated. These illustrate the change in height of the dough sample and also suggest that the constraints created by the sample container affect the shape of the cells, causing them to favour upwards growth. It is likely that these constraints are also present in industrial breadmaking where dough is proved and then baked in a tin. Cells are also highlighted in the figure to illustrate how they change throughout the process.
Figure 6.21 Two dimensional images of dough slices with some cells highlighted and tracked throughout dough proving from 0-145 minutes at 21.5°C
Figure 6.22 Two dimensional images of dough slices with some cells highlighted and tracked throughout dough proving from 0-145 minutes at 21.5°C
**Figure 6.23** Two dimensional images of dough slices with some cells highlighted and tracked throughout dough proving from 0-145 minutes at 21.5°C
Figure 6.24 Two dimensional images of dough slices with some cells highlighted and tracked throughout dough proving from 0-145 minutes at 21.5°C
Figure 6.25 Two dimensional images of dough slices with some cells highlighted and tracked throughout dough proving from 0-145 minutes at 21.5°C
Figure 6.26 Two dimensional images of dough slices with some cells highlighted and tracked throughout dough proving from 0-145 minutes at 21.5°C
Figure 6.27 Surfacegen images showing the volume of the dough (blue) and the cells within it (red) during proving at 0 minutes, 30 minutes, 60 minutes and 140 minutes from left to right.
In Figure 6.22, images of the dough volume following 0, 30, 60 and 140 minutes of proving are presented. These illustrate how the dough volume changes as a result of its composition. Coloured in blue is the dough matrix and in red the cells. The figure shows the folds within the dough and the dough shape clearly and dough sides supported by tubing can be seen through the shape of the dough piece. The dough volume increases over the images, with the volume increase largely occurring in height. This is due to the tubing restricting expansion of the dough in other directions. At 0 minutes and 30 minutes, the number of cells in the dough allows the depth of the cells to be seen. By 60 minutes the change in dough composition makes it difficult to identify individual cells in the image and determine their location within the volume. At 0 minutes, it can be seen that a range of cell shapes and volumes have been entrained in the dough. On the left hand side of the dough piece there is also an L-shaped cell. This is likely an envelope of gas formed during mixing that has not had as much breakup from the mixer blade as the other envelopes of gas. The range in cell sizes and shapes can also be seen in the figure. The figure also shows that although the dough volume fills up with gas cells, there are relatively few cells at the edges and in particular at the top of the dough volume, compared to the centre of the dough piece. There are several possible reasons for this. Firstly, carbon dioxide molecules are likely to diffuse to nearby cells which are less likely to be those on the dough edge. Secondly, cells are less likely to occur at the top edge of the dough due to gravity. Also, because of gravity, cells higher up in the dough will be less stable, and therefore more likely to rupture. Thirdly and most likely, most commonly, when gas diffuses to the edge of the dough and in particular the top of the dough when proofed in a container, the dough matrix between the cell and atmosphere may thin and rupture, enabling the escape of cells into the atmosphere.

Figure 6.23 shows the cell number density observed in the dough volume at different times during proving. The graph shows an increase from approximately 20000 cells cm\(^{-3}\) at 0 minutes, to 54000 cells cm\(^{-3}\) at 30 minutes. The number of cells then decreases over the mixing session to less than 3500 cells cm\(^{-3}\). The increase in observed cells in the first 30 minutes cannot be due to the evolution of more cells. The increase is due to growth of cells previously below the resolution, as suggested by Figure 6.24 which shows a growth in cell volume with proving time. Between 0 and 145 minutes the mean cell volume increases by almost 156 times. As there were a large number of smaller undetected cells, in particular at earlier stages of proving, the increase in mean cell volume is likely to be underestimated. Other authors (Babin et al., 2008; Calderón-Domínguez et al., 2008) also found a decrease in cell number and an increase in mean cell size after longer proving times.

It is likely that the observed cell number density is lower than actual cell number density at all points, but more accurate at later stages as cells below resolution size interact with large neighbouring cells, and fewer cells below the resolution size are present. It was expected that with time, growth of the cells would cause them to coalesce and therefore the cell number density to decrease. The increase in cell volume over proving is illustrated in Figure 6.24. It shows a significant increase in mean cell volume over time.
Figure 6.28 Number density of cells at different times whilst proving at 21.5°C

Figure 6.29 Mean cell volume at different times whilst proving at 21.5°C

Figure 6.25 illustrates the cell number density per unit volume at different times during proving. A reduction in cell number density per unit volume is observed with an increase in cell volume. This is as expected due to less space being available for larger cells and the reduced stability of larger cells. Figure 6.24 also shows a change in the cell volume distribution with time. An increase in the number density of cells per unit volume of the full range of cell volumes from 0 to 30 minutes is observed. Following this, the number density of cells per unit volume decreases with time. The former is due to the resolution and growth of the cells enabling them to be detected at 30 minutes. The decrease in number density of cells per unit volume observed after 30 minutes is due to cell breakup and coalescence. These cell mechanics are reflected in the existence of larger cell volumes at 70 and 145 minutes. With the exception of the number density per unit volume of the cells around the resolution size the trend in the data sets is as expected.
Figure 6.30 Number density of cells per unit volume at different times whilst proving at 21.5°C

Figure 6.25 shows the cumulative percentage voidage of the different cell volumes for different proving times. The data sets at 0 and 30 minutes, and 70 and 145 minutes, share similarities, a reflection of the resemblance in voidage distribution at these times. At 0 and 30 minutes the voidage is distributed over a similar range of cell volumes. However, at later stages of proving, gas tends towards larger cells. At 70 minutes less than 4% of the voidage contribution is from cells smaller than $3 \times 10^9 \, \mu m^3$, and at 140 minutes the voidage contribution is less than 1%, compared to 25% at 50 minutes and 100% at 0 minutes. This quantitative data illustrates how quickly the voidage distribution changes over proving and highlights the timing at which significant changes in structure occur.

Figures 6.24 and 6.25 illustrate that a large number of cells does not necessarily mean a large proportion of gas.

Figure 6.31 Cumulative % voidage of different cell volumes at different times whilst proving at 21.5°C
Figure 6.26 illustrates the change in Feret shape of cells at different proving times. Figure 6.26(a) illustrates how the mean cell Feret shape changes, Figure 6.26(b) illustrates the cell Feret shape distribution through frequency per unit volume of different cell Feret shapes at different proving times and Figure 6.26 (c) the Feret shape of cells at different proving times. Figure 6.26(a) shows that in contrast to literature descriptions (Campbell et al., 1991; Bellido et al., 2006; Turbin-Orger et al., 2012), cells are on average non-spherical in shape when entrained. This shows cell volume to be more representative of cell size than cell diameter. In Figure 6.26(a) an increase in mean Feret shape from 1.59 to 1.91 can be observed, implying cells become less spherical and more irregular, a result of the increasing interaction of cells with each other over the proving time. As the shape of the gas cells deviate more from a sphere, they become less stable, as the forces acting on the cell become more unbalanced. The restriction to the direction of cell growth created by using a sample container is also likely to contribute towards the increase in Feret shape observed with time. Figure 6.26(a) also shows the decrease in the standard deviation of the cell Feret shape with proving time. The decrease in standard deviation illustrates that as proving time increases, there is less deviation in cell Feret shape. This is due to coalescence increasing the irregularity of the cell shapes, making the cells more elongated than the cell shapes entrained during mixing. In addition, when cells increase in size, it becomes easier for pixels to accurately follow the circular contours of the cell (Bellido et al., 2006). The number of cells were reduced as proving time increased. There is a lot of scatter in the data but single factor ANOVA tests confirm at a 95% confidence level that a significant difference exists between the cell Feret shapes at different proving times.

In Figure 6.26(b) the frequency per unit volume of groups of Feret shape of a range of 0.5 at different proving times is illustrated. The range of Feret shapes is smallest at 0 minutes, with a maximum mean cell Feret shape of 5.75. Literature has found that cell shape becomes less spherical over the breadmaking process (Babin et al., 2006; Babin et al., 2008). Figure 6.26(b) shows that at the most dynamic times during the proving session, 30-120 minutes, the maximum cell Feret shape increases to approximately 14.75. The maximum cell Feret shape at 145 minutes is approximately 10.75. This data point is made up from one cell as demonstrated in Figure 6.26 (c). This reduction in cell Feret shape late into the proving time is likely to be due to cells having had more time to reach a stable shape of lower Feret number or rupturing if unstable. Figure 6.26(b) also shows a greater number of cells with a smaller Feret shape at the start of the proving session than as proving progresses. This is due to there being fewer cells as they grow and interact with each other and become less spherical. As proving time progresses, the number of cells per unit volume decreases and the likelihood of the presence of cells with a larger Feret shape increases.
Figure 6.32 (a) Mean Feret shape of cells at different times whilst proving at 21.5°C. The data points are the mean of all cells present at the proving time. Error bars are one standard deviation on either side of the mean. A linear regression line is shown. (b) Feret shape distribution of cells per unit area at different times whilst proving at 21.5°C. The mean cell Feret shape are based on group sizes of 0.5. The volume is the dough volume at the proving time. (c) Mean cell Feret shape at different times whilst proving at 21.5°C.
distribution of cells at different times whilst proving at 21.5°C. The mean cell Feret shape are based on group sizes of 0.5. The volume is the dough volume at the proving time.

Mean cell Feret shape at different times whilst proving at 21.5°C

6.5 Summary

Work in this Chapter investigates the cellular structure of dough during mixing and proving, with variations in the dough formulation, and fits a population balance model to the measured cell size distributions. This confirmed validity of the model, and thus can be used to improve the understanding of the dynamics of aeration during bread dough mixing. Cell volume distribution in bread dough was presented, which is more representative than the 2D measurements of cell size typically published in academic papers, due to the non-spherical shape of cells.

To summarise this Chapter’s results, Section 6.2 investigated the cellular structure of dough with and without sugar. The results agreed with previous chapters, showing the sugared dough to have a lower voidage, and suggested this may be due to the smaller cell size and number. This observation is also the likely cause for a lower specific volume and greater firmness in finished Tweedy loaves, as a sufficient number of cells which are large enough to inflate must be present. It is likely that the higher disentainment rates observed in Chapter 5 in sugared doughs and in particular sugared dough made from weak flours are due to a weaker dough structure, making retention of bubbles more difficult.

In Section 6.3 an investigation was carried out into the dynamics of aeration and the change in cellular structure of the dough during pressure-vacuum mixing. This is the first time X-ray CT has been utilised to image changes in an opaque fluid during processing and the results integrated into a population balance model. The results support the no breakup population balance model previously reported by Martin et al. (2004a), as a reasonably accurate representation of the dynamics of aeration in bread dough, and demonstrates the model can give useful interpretations of the aeration process during pressure step change mixing, using just dough voidage measurements. In addition, the results found that over time in pressure-vacuum mixing, there are more cells of a smaller volume. This corresponds to the theory of pressure-vacuum mixing leading to a fine cell distribution, and due to increased cell breakup with time during mixing following expansion of cells on drawing a vacuum.

Finally, in Section 6.4, an investigation was carried out into the dough cellular structure throughout proving. The results showed the potential of a conventional X-ray source in monitoring in-situ changes in the dough. Cell volume, shape, number and dough voidage were monitored using sample volumes larger than those in comparable studies (Babin et al., 2006; 2008; Turbin-Orger et al., 2012), therefore allowing larger cells to form and be detected, and giving more representative quantitative data. This technique would be useful for monitoring the proving of yeasted bakery products to obtain the optimum proving time for the desired dough structure.
The three experiments in this Chapter illustrate the potential of X-ray CT in accurately determining dough voidage and illustrate a small proportion of the range of information that can be obtained on the cellular structure of dough using the technique.

The methodology developed from the conventional X-ray source obtained voxel resolutions greater than published work on dough samples imaged via X-ray CT and comparable to synchrotron X-ray sources. A 4D scan of proving dough, however, illustrated that the resolution was not high enough to detect all cells. This highlighted the incredibly small size of entrained cells and suggests that obtaining the cell size distribution via X-ray CT cannot get more accurate if representative dough samples are to be used.

The developed methodology illustrated that although it is known that synchrotron X-rays can obtain superior images to conventional X-ray sources, it is possible to image bread dough to a quality capable of obtaining accurate voidages, and that both 3D and 4D information could be obtained using conventional X-ray sources. Experimental work using a conventional X-ray source has some advantages over experimental work carried out on synchrotron beamlines. These include decreased set up time and costs, and increased availability.
Chapter 7 - Conclusions and further work

7.1 Conclusions

This thesis has investigated the effect of sugar on the aeration of bread dough, assessed the validity of an existing simplified population balance model of bread dough aeration and demonstrated the suitability of a non-synchrontron X-ray source to accurately investigate the microstructure of bread dough in both 3D and 4D processes. A literature review was first presented, discussing the significance of bread, its key ingredients, how it is made industrially and aeration during breadmaking. As the cells in bread are the subject of this thesis, how they are formed and how they change throughout the breadmaking process were explained, alongside a summary of published methods on investigating bread dough aeration.

Little work has been published on the effect of sugar on bread dough aeration, yet sugar containing breads make up a large proportion of the bread market, and a better understanding of its effect on bread will widen the opportunity for product development. The results show that extension of the industrial methodologies used on non-sugar containing formulations to sugared formulations was detrimental to the quality parameters of bread dough and bread, producing products with undesirable qualities and therefore that would be difficult to market. The presence of sugar changed dough aeration during mixing, by reducing the steady state voidage and increasing the gas free dough density, as a result of greater disentrainment during mixing. This is likely to be due to the weaker gluten structure within the dough, as reflected by the lower elasticity of sugared doughs. Assessment of a sugared dough found it contained fewer and smaller cells following mixing, compared to a non-sugared dough. This is likely to be responsible for the increase in specific volume and firmness of bread. Sugar was also found to increase dough proving time, most likely due to the osmotic stress applied to yeast, hence use of different strains of yeast worldwide depending on the type of bread being made. The results suggested that dual pressure and in particular pressure-vacuum mixing of sugared doughs would not produce bread dough with a finer cell distribution, but instead produce denser dough. Dense bread is perceived by consumers as undesirable.

X-ray CT was used to measure voidage and obtain cell size distribution throughout dough samples. Voidage is important as it is a measure of how much gas is in the dough and therefore its volume. Cell size distribution is important as these determine the quality parameters in a loaf, and can be the difference in texture between rye bread and UK sandwich bread. A comparison of the dough voidage obtained through X-ray CT and the established double cup method found X-ray CT to produce reliable although less convenient voidage measurements. The X-ray set up enabled the generation of high resolution and contrast images, easing the dough segmentation process using digital image analysis. This technique provides images non-invasively and can be utilised for future analysis of bread dough microstructure, helping understand the link between ingredients and processing, the structure and quality parameters.
In Section 6.2 pressure-vacuum mixing was shown to lead to a finer cell distribution, and the decrease in number density following pressure-vacuum mixing was found to be much more short-lived than the decrease in size, which was permanent. Cumulative voidage data was fitted to the simplified no breakup population balance model of aeration. The simplified model and experimental results showed good agreement, reflecting the robustness of the model. The model can be applied using only voidage data, making it useful for providing interpretations of the aeration process.

Having established a method of viewing the internal structure of bread dough, this method was utilised again to view the microstructure of bread dough during proving. Quantitative information and images of the dough were generated to get a full picture of the dough proving process, from immediately after mixing to the levelling off of cell proportion and volume. An increase in cell number over the first 0.5 hour of proving was found. This highlighted an insufficient resolution size for detection of all cells, and indicated the large range of cell nucleation volumes. Results showed an increased in both dough and cell volume, increase in cell Feret shape, and increase in dough voidage throughout the process.

Chapter 6 illustrated the potential of X-ray CT in monitoring both static and dynamic changes in dough structure, and discussed advantages of its use over synchrotron.

7.2 Further work

Investigations of the effect of sugar in bread highlighted the detrimental effects of simply extending existing methodologies from non-sugar containing formulations to sugar containing formulations, in particular in terms of loaf aeration. It also suggested the presence of insufficient water prevented optimal development of sugared doughs. It would be interesting to investigate the effect of sugar on optimally developed doughs. This would require adjusting the water content according to the quantity of sugar present and determining the quantity of work required to develop each dough, and mixing to this work input. ESEM micrographs should be used to confirm development of the gluten matrix. Working through each stage of the breadmaking process, from mixing to proving to baking, each process should be optimised for each dough to determine if use of the CBP would be feasible for the production of a high quality sweetened bread product. Optimisation of each stage would involve assessment of key quality parameters at that stage. Quality parameters would include product appearance (colour, volume, shape), cell volume distribution and eating qualities (firmness, resilience, flavour). It would be useful to measure a number of quality parameters throughout the process and determine if a quantity of sugar exists whereby the negative effects on quality are no longer acceptable. The negative effects should be monitored both experimentally and subjectively, as detection thresholds will vary between humans and machinery. Subjective methods would involve consumer panels. It is important that the flavour of bread, especially in terms of sweetness is assessed subjectively, as although it may be possible to process bread with 40% sugar for example, this may not be a desirable product. In addition, preferences vary across individuals.
This thesis assessed the effect of sugar on bread dough and bread through measuring a range of parameters throughout the breadmaking process. The sugar used was in solid form, and as noted in Section 4.4, during the mixing process, the sugar first dissolves into the water to form a syrup, which is mixed with the other ingredients to form a solid mass. If a liquid sugar was used, this should be a more efficient process, as dissolving the sugar is not necessary. Solid sugar is also abrasive, and could damage the starch in the dough, increasing the dough’s necessary water absorption for development. It would be interesting to compare the differences in the processing of sweetened bread using both a solid and liquid sugar. It is hypothesised that both would produce a denser dough and loaf, when added in certain quantities in certain dough formulations. However, the production of sweetened breads through using a liquid sugar, compensating for increased water levels by reducing quantities of water would be more practical industrially and produce better quality loaves due to lower levels of starch damage (Cauvain and Young, 2004). Quality indicators associated with increased levels of starch damage include larger cell sizes and greying of the dough matrix.

As artificial sweeteners are generally sweeter than sucrose, less of it would be required to sweeten breads. Its use as an ingredient in bread could be investigated as an alternative to sugar. Natural sweeteners, such as agave syrup and honey, could also be investigated. An interesting natural sweetener to investigate in sweetened breads would be the calorie free leaves of the stevia plant. Stevia is 250-300 times sweeter than sucrose (Food Standards Agency, 2013) so only a small amount of the product would be required, and it is hypothesized that the detrimental effects on the quality of bread would not be observed with the addition of finely ground leaves. It can be expected that bread’s appearance may take on a hint of green, depending on the quantity added. Although green bread is associated with mouldy bread, if marketed cleverly, a light green bread can be desirable, as seen in oriental countries where green tea bread is popular. However, although stevia is accepted, widely used and available in numerous countries worldwide, in the UK its use and to specified levels only is currently limited only to certain foods by law.

This thesis detailed a non-invasive and non-destructive method to investigate dough cellular structure dynamically. To obtain a more detailed and accurate picture of dough microstructure, it is important to ensure all the cells are detected. To complete this task X-ray CT of bread dough on the nanometre scale could be combined with X-ray CT on the micrometre scale, using two pieces of X-ray equipment. This is a challenging task as detection of smaller cells generally requires the use of smaller samples, which reduces the representativeness of the sample. However, it would determine the smallest cell nucleation volume and the minimum cell volume for growth, two parameters which could not be determined from this work, and are not known.

In the X-ray proving experiment, the results showed the dough to be overproved, but not to the extent that a significant proportion of the cells had begun to collapse and reduce its voidage. Although over proving is not desirable or beneficial in breadmaking, it would have
been interesting to capture how the cells collapse, and whether the collapse of a large cell would lead to an onset of cell collapse from surrounding cells.

Further work should include completion of the cell tracking. This would enable one to determine with confidence if disproportionation, a process disputed amongst some leading in the field, occurs within cells. Development of the fast reconstruction algorithm should also be completed to hasten the time consuming reconstruction process. The quality of results from the different dough proving data sets obtained via X-ray CT can then be compared.
Chapter 8 - References

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Hirning J., 2010. The significance of salt in bread dough processing, and compensating the effects of reduced salt levels by use of a novel dough additive, Masters dissertation, University of Manchester, UK


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Chapter 4, Section 4.10: Sugar content of strong flour doughs and proving time

ANOVA: Single Factor

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Chapter 4, Section 4.10: Sugar content of weak flour doughs and proving time

ANOVA: Single Factor

SUMMARY - weak dough proving time

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Chapter 4, Section 4.10: Sugar content of strong flour doughs and minimum density during proving

ANOVA: Single Factor

SUMMARY - strong min density

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Chapter 4, Section 4.10: Sugar content of weak flour doughs and minimum density during proving

ANOVA: Single Factor

SUMMARY - weak min density

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Chapter 4, Section 4.11: Sugar content of Tweedy loaves and baked loaf density

ANOVA: Single Factor

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Chapter 4, Section 4.11: Sugar content of breadmaker loaves and baked loaf density

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Chapter 4, Section 4.11: Sugar content of Tweedy loaves and percentage baking loss

ANOVA: Single Factor

SUMMARY – Tweedy baking loss

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Chapter 4, Section 4.11: Sugar content of breadmaker loaves and percentage baking loss

ANOVA: Single Factor

SUMMARY – breadmaker baking loss

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Chapter 4, Section 4.11: Sugar content of Tweedy loaves and force to compress 8mm
ANOVA: Single Factor

SUMMARY – Tweedy force to compress

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Total           | 3.005376| 215|

Chapter 4, Section 4.11: Sugar content of breadmaker loaves and force to compress 8mm
ANOVA: Single Factor

SUMMARY – breadmaker force to compress

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Total           | 9.929496| 215|

Chapter 4, Section 4.11: Sugar content of Tweedy loaves and cell number density
ANOVA: Single Factor

SUMMARY – Tweedy cell no. density

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Chapter 4, Section 4.11: Sugar content of breadmaker loaves and cell number density

ANOVA: Single Factor

SUMMARY – Breadmaker cell number density

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Chapter 4, Section 4.11: Sugar content of Tweedy loaves and mean cell diameter

ANOVA: Single Factor

SUMMARY – Tweedy cell diameter

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Chapter 4, Section 4.11: Sugar content of breadmaker loaves and mean cell diameter

ANOVA: Single Factor

SUMMARY – breadmaker cell diameter

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## Chapter 5

### Section 5.3.1 Raw data used to plot Figure 5.3

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### Chapter 5

#### Section 5.3.1 Raw data used to plot Figure 5.3

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## Chapter 5

### Section 5.3.1 Raw data used to plot Figure 5.4

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Chapter 6, Section 6.4: Proving time and cell Feret shape
ANOVA: Single Factor

### SUMMARY - cell Feret shape

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Bread dough aeration dynamics during pressure step-change mixing: Studies by X-ray tomography, dough density and population balance modelling

L. Trinh a, T. Lowe a, G.M. Campbell a, P.J. Withers b, P.J. Martin c

a The University of Manchester, School of Chemical Engineering & Analytical Science, Manchester M13 9PL, United Kingdom
b The University of Manchester, Henry Morsley X-ray Imaging Facility, School of Materials Science, Manchester M13 9PL, United Kingdom

c Corresponding author. Tel.: +44 161 200 6258. E-mail address: p.j.martin@manchester.ac.uk (P.J. Martin).

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ABSTRACT

Industrial bread dough mixing often involves a period of mixing under high headspace pressure to enhance oxygen availability, followed by a period of partial vacuum to favourably manipulate the final bubble size distribution. This paper presents the results of a study using X-ray tomography to measure the gas bubble size distribution in dough samples over the course of a pressure-step-change mix. The first objective of the current work was to measure bubble size distributions at points throughout a pressure-step dough mixing process using a non-synchronous X-ray source. The second objective was to fit a simplified population balance model to the measured size distributions. The third objective was to explore the validity of the assumptions within the simplified model and to consolidate understanding of underlying aeration and mixing phenomena and the resultant process dynamics. It was found that the dynamic changes in the bubble size distribution of bread dough during pressure-step mixing could be accurately measured using a laboratory X-ray source. The response of the cumulative dough volume to a pressure-step change during mixing could be reproducible very well using the simplified population balance model (which assumes all entrained bubbles are the same size, no bubble break-up or coalescence, and that bubble shearing is proportional to bubble volume). The measured response of the bubble number density and mean volume agreed reasonably well with that predicted by the simplified model (with parameters fitted to only cumulative volume data). It is demonstrated that the decrease in number density following a pressure step-decrease is much more short-lived than the decrease in size which is permanent.

HIGHLIGHTS

• A novel study of aeration in bread dough by density and X-ray tomography.
• First measurement of bubble size distribution with a population balance model.
• Bubbles size distribution in bread dough mixing has been accurately measured.
• Simplified population balance model represents measured dough volume well.
• Measured bubble number density and mean volume agreed with population balance model.

1. Introduction

The measurement of gas molarities, and other parameters, within a given fluid is challenging and this is particularly true when studying dynamic changes during their processing. However, such data is essential for understanding, modelling and controlling the size characteristics of these dispersions. The aeration of bread dough during mixing is a case in point.

Air bubbles entrained during dough mixing transport oxygen throughout the bulk simultaneously, the oxygen diffuses into the matrix and plays an essential role in the dough development chemistry. Furthermore, in the Cherrywood brand process the remaining bubbles at the end of mixing move into the final bread crumb cellular structure and thus influence its quality (Baker and Mizen, 1973; Baker and Mizen, 1981; Chamberlain and Collins, 1977; Spooner, 1990; Martin, 2004). Industrial bread dough mixing...
involved a period of mixing under high headspace pressure to enhance oxygen availability, followed by a period of partial vacuum to favourably manipulate the final bubble size distribution at the NHV Corporation Ltd., 2004). The difficulty of measuring bubble size distributions in the opaque and highly viscous dough mean that there is paucity of reported data and industrial practice relies on established art. The ability to engineer this process is advanced by measurement and modelling of the dynamic balance of entrainment, breakup, coalescence and dissolution (Campbell et al., 1996).

This paper presents the results of a study using X-ray tomography to measure the gas bubble size distribution in dough samples over the course of a pressure-step-change mix.

1.1 Imaging particles in bread dough and other opaque fluids

The objective of the current work was to measure bubble size distributions at points throughout a pressure-step dough mixing process using the Henry Moses X-ray Imaging Facility at the University of Manchester. The second objective was to fit a simplified population balance model to the measured size distributions. The third objective was to use the data set and fitted model to explore the validity of the assumptions within the simplified model and to consolidate understanding of underlying aeration and mixing phenomena, and the resultant process dynamics.

Direct visualisation is the predominant technology for measuring particles in transparent fluids. The main techniques which have been reported for measuring particles in opaque fluids are:

- X-ray ultrasonic scattering/transmission, electrical tomography (e.g. Elmore et al., 2003; Gent et al., 2004; Roeben et al., 2013); insufficient resolution and accuracy for bubbles in dough.
- ex situ or miniaturised in situ: X-ray tomography, magnetic resonance imaging (e.g. Brot et al., 2004; Stevens et al., 2010; Hold and Seto, 2011); M08 has insufficient resolution (voxel dimensions 100 μm) for dough bubbles.
- ex situ: Microscopy of physically sectioned samples (e.g. Cerny et al., 1991, 1998; Martin et al., 2004a; Menon et al., 2013); laborious with limited accuracy.

This leaves X-ray tomography as the most suitable method for each particle measurement. X-ray tomography of bread dough was first reported by Behnia et al. (2006) using a bench top microtomography scanner with a peak energy level 70 keV and a constant current 100 μA. Samples of 0.3 g were removed from a dough and were prepared for ex situ imaging by flattening to a thickness of 2 mm. Four frame averaging was used over 512 projections with 0.205 s exposure time resulting in 620 s scan times. Reconstructions resulted in voxels of (10 μm) over a 10 mm diameter by 2 mm height field of view. Bubbles size distributions were found by first removing bubble cross section diameters in each slice (assumed to be circles) and reconstructing the 3D bubble size distribution (assumed to be spheres). Reconstructions found around 4500 bubbles per sample, size and shape of each bubble were calculated individually. Mean bubble diameters of 100 and 109 μm with diameters of 0.0076 and 0.004 μm and bubble densities of 0.64 and 0.55 mm−3. Volume measurements were found to be within 95% of those calculated from density measurements, while cross-sectional anisotropy measurements indicated significant deformation of bubbles during sample preparation.

Behnia et al. (2006, 2010) reported a study of fast X-ray tomography for in situ measurement of bubble growth in doughs using a minimised dough proving apparatus. Experiments were performed on bread doughs at the European Synchrotron Radiation Facility (ESRF). Baked, at a beam energy of 18 keV with four frames averaging, 450 projections, 0.004 s exposure time per projection and 30 s scan times. The high photon flux of the synchrotron was reported to result in high contrast images which facilitated measurement of dynamic changes. The reconstruction comprised voxels of 15 μm dimensions over a 9 mm diameter by 4 mm height field of view. Thresholding followed by granulometry was used to measure the bubble size distributions, track individual bubble growth and quantify connectivity. The mean bubble diameter was 40 μm for three different dough formulations.

Turbin-Črner et al. (2012) followed the approach of Behnia et al. (2006) using fast X-ray tomography to measure the growth kinetics of bubbles in proving dough. Again, beamline ID15B was used with a monochromatic polychromatic beam of energy 17.6 keV, using 300 projections, at 0.004 s exposure time giving an overall scan time of 80 s. Local tomography was used to achieve high resolution over a region of interest within a small portion of the 30 mm diameter by 40 mm height samples. Reconstructions comprised (5 μm) voxels over a (5 mm) field of view. This is the highest resolution 3D imaging of dough reported to date. They noted that, while the volume fraction of the smaller bubbles was very low (< 0.01%).

The small size of these undetected bubbles also means that they can make a significant contribution to the number density of bubbles in the dough – and thus also the mean bubble size – whilst not impacting significantly on the viscosity.

1.2 Population balance modelling

When the mixer headspace pressure is changed from one pressure to another pressure P to another pressure P during pressure-vacuum mixing, or when dough is removed from the mixer to atmospheric pressure, the bubble volume, number density and viscosity change commensurately. Martin et al. (2004a) detailed these population balance changes and demonstrated that a population balance model was necessary to account for experimental observations of dough viscosity changes during mixing following a step-change in headspace pressure (Choi et al., 2004). The analysis in this paper is based on the bubble population in situ within the mixer under the mixer headspace pressure.

Martin et al. made the simplification that the number of bubbles entrained into the dough per unit volume of dough per second, N, was constant and that these bubbles all have the same volume, Vb, regardless of the headspace pressure. Coalescence was assumed to be negligible and they hypothesised that the probability of bubble break-up and dissolution phenomena were proportional to bubble volume. Thus the dissolution coefficient k for a bubble of volume b = Vb/Vb is given where k is the frequency of dissolution events per unit volume of dough per second.

In the mixing rate equation for the cumulative bubble volume data, the mixing rate coefficient K, the mixing rate constant, was determined by using a least squares fit to the experimental data.

\[ \frac{dN_b}{dt} = K_m \left( \frac{N_b}{t} - N_b \right) e^{-N_b/\lambda} \]

where t is the mixing time, t is the mixing time at which a cohere dough ball is first formed from the ingredients, N_b(t) is the number
density of bubbles of size $V_D$, when the dough ball is formed at $t_0$. Multiplying both sides of Eq. (1) by the bubble volume $V_D$ gives an expression for the transient voidage, $\phi$:

$$\phi = V_D \left( \frac{F_n V_D}{R} \right) \times 10^{(2-\alpha)}$$

(2)

where $\phi = \frac{V_n}{V_D} \frac{V_D}{R}$ is the voidage, $V_n = \frac{V_D}{R}$ is the volumetric gas entrainment rate per unit volume of dough and $\phi = \frac{V_n}{V_D} \frac{V_D}{R}$ is the initial voidage of the dough at $t_0$. This is of the same form as Campbell and Shanks (1990) mass balance model which did not account for bubble size. Following a pressure step-change at time $t$ all the bubbles previously of volume $V_D$ will change concurrently to $V_n$ and a new population of bubble of volume $V_D$ will start to be entrained.

$$F_n(V_D) = \frac{V_n}{R} \left( 1 - e^{-\phi \alpha} \right)$$

(3)

$$F_n(V_D) = \frac{V_n}{R} \left( 1 - e^{-\phi \alpha} \right)$$

(4)

$$\phi = \phi_e \left( 1 - e^{-\phi \alpha} \right) + \phi_{0} e^{-\phi \alpha}$$

(5)

where $\phi_{0}$ is the voidage immediately after the pressure step-change. This model was found to fit transient dough voidage measurements for mixing under constant, step-up and step-down headspace pressure using a single set of parameters. The first term on the right hand side of Eq. (5) models the contribution of bubbles entrained after the pressure step-change. The second term models the contribution of bubbles which were entrained before the pressure step-change and is a function of the pressure into $F_n(V_D)$.

The model was the first to enable prediction of voidage response to pressure step-changes and thus enable design of pressure/vacuum mixing procedures. It was the first to quantify the rate of turnover of air during dough mixing and to reveal that this is relatively slow, with an average residence time typically of the order of half of the total mixing time. It was also the first to provide a reliable measure of the air entrainment rate into the dough, and thus the oxygen availability for dough chemistry.

Model parameters may be fitted using a variety of iterative techniques to minimise a function such as the root mean of the sum of squared errors (RMSE) between the model values to the measured data. Press et al. (1986) introduce a method for quantifying the confidence of these fitted parameters by using Monte Carlo techniques. Simulated data may be calculated by randomly selecting values from the probability density functions (pdf) of an existing data set, using the fitted model value as the pdf mean and the RMSE as the pdf standard deviation. Model parameters are fitted to the sets of simulated data, and then the standard deviations may be calculated from the accumulated parameters.

Martin et al. (2004a) attempted to measure bubble size distributions by image analysis of microtomated frozen dough slices, but the method proved to be inadequate for reliable comparison of the model against bubble size distribution data. Thus the combined measurement of bubble size distributions by X-ray tomography and the application of this simplified population balance model offers an opportunity for demonstrating the model's utility and for consolidating understanding.

### 2. Materials and methods

A programme of experiments was conducted to mix dough under both constant 100 kPa (absolute) headspace pressure and step-down headspace pressure from 100 kPa (absolute) to 50 kPa (absolute) after 1 min of mixing. Designs were made from the formulation detailed in Table 1 using a strong flour (BS 5584, High Wycombe, UK) with measured protein content of 11%. The doughs were prepared as noted above for fermentation during the X-ray tomography scan. All doughs were mixed using a custom made laboratory scale Tweedy T1, a scaled down industrial pressure/vacuum mixer with a maximum angular speed of 70 rad s$^{-1}$ (Martin et al. 2004). The water temperature was controlled to ensure the final dough temperature after 180 s of mixing was 36 ± 2 °C (sufficient to develop the dough).

Transient behaviour was measured by mixing doughs for various amounts of time under the determined pressure conditions and then interrupting the mix to remove dough samples for analysis. Once interrupted, the dough would be discarded and a fresh experiment started for the next data point. One set of experiments was conducted to achieve a complete dough density response to pressure step-decrease. A separate set of experiments was conducted for samples to be taken for X-ray analysis – dough density was normally measured in these cases as well.

#### 2.1. ESEM microscopy

A FEI Quanta 200 (Hillsboro, Oregon, USA) environmental scanning electron microscope (ESEM) was used to obtain detailed images of the dough microstructure. A small sample of dough was removed following 230 s of mixing and immediately frozen in liquid nitrogen. The sample was fractured and a fragment placed into the ESEM for imaging.

#### 2.2. X-ray tomography

For each mix a dough sample approximately 1.5 cm$^3$ in volume was prepared for X-ray imaging. The dough sample was carefully inserted into a glass tube. A sufficient quantity of dough was inserted in the tube to ensure it would not slump during scanning. The tube had an internal diameter of 10 mm and a wall thickness of 0.5 mm. A film was placed over the top of the tube to minimise dehydration. The tube was then supported and secured on the X-ray sample holder of a Nikon X-TMS 225/320 kV custom X-ray CT system to obtain radiographs of dough samples. The detector was a 2K x 2K Perkin Elmer 1621-16-16 14-micron silicon flat-panel detector with 200-μm pixel pitch, which allowed fine differences in contrast to be detected. This enabled detection of a number of features which would not have been seen on the standard Nikon X-TMS 225 kV X-ray CT instrument.

A target material within the source is interchangeable to ensure the best signal and contrast for a given sample material. With these target materials, an energy range of 40-230 kV can be obtained, allowing the scanning of biological materials through to dense metal. For the X-ray scans conducted here a molybdenum target was used. The sample was rotated 180° and 180° projections were obtained over an approximately 45 min scan. Settings were varied for optimal imaging of each sample. Energy varied from 30-80 kV, current from 200-280 kV and voxel size from (2.1 μm)²(10.8 μm)³.
2.1. Dough density

The double cup density approach of Campbell et al. (2001) was used to measure dough density and thus calculate dough oil. Six dough pieces of approximately 11g were carefully cut from different regions of the dough ball and then weighed in air and in silicone oil at 80°C. The two weights and knowledge of the oil density enabled accurate calculation of the dough density of each sample. The mean of six measurements was calculated. A series of experiments mixed separate doughs under constant headspace pressure ranging from 25 to 250 kPa (absolute). The linear density-pressure plot was extrapolated to zero pressure to find the gas-free dough density of 1.253 g cm⁻³. Dough voidage was calculated using the gas-free dough density and dough density measurements. Voidage transformation with pressure followed the equations reported by Martin et al. (2004a).

3. Results and discussion

3.1. ESEM microscopy

Fig. 1 shows an ESEM micrograph of a fractured frozen sample of dough mixed under 100 kPa (absolute) constant pressure for 30 s. A roughly spherical bubble of 56 µm diameter is clearly visible in the centre of the image. Several other bubbles of 10-15 µm diameter also appear to be visible. These apparent bubbles have a similar scale to the starch granules which can also be seen; it could be that the same of the apparent small bubbles are recessed left by starch granules during fracturing.

3.2. X-ray tomography

Fig. 2(a) shows an orthoslice of dough mixed under 100 kPa (absolute) pressure for 30 s reconstructed from X-ray tomography. The pixel width is 10.8 µm in this image. The highlighted region shows features of around 50 µm diameter which appear to be bubble defects, but it is difficult to identify with confidence bubbles that are smaller than this diameter. While smaller bubbles look circular in cross section many of the larger bubbles appear elongated which indicate that bubble formation would be more representative of a sphere than diameter. Fig. 2(b) and (c) shows orthoslices for doughs mixed at 200 kPa (absolute) and at 100 kPa (absolute) followed by a step-decrease to 50 kPa (absolute). The bubble size distribution for the 100 kPa (absolute) cases look similar at first inspection. However, the dough which experienced a step change appears to have a significantly different bubble size distribution—with fewer and possibly smaller bubbles.

It was essential to have confidence in the measured bubble size distributions in order to quantitatively compare them with the population balance model. A particle size cut-off of 113 vessels was used to ensure that there were no false positives or negatives in bubble identification by the software. This equates to an effective spherical bubble diameter of 0.43 µm or 43.65 µm for the different samples. Below this cut-off no bubbles were recognised.

3.3. Dough density

Fig. 3 presents the transient dough voidage for both constant pressure and pressure step-down mixes. This and all other figures in this paper, refer to the state of the dough. The appearance of a bubble phase in X-ray tomography is only possible when the bubble burst during the measurement of the dough density. The agreement between the voidages from the same dough measured by density and X-ray tomography was typically within 7% with the X-ray tomography tending to underestimate the voidage size. A point on Fig. 3 for example at time t = 10 s, shows that the bubble size measured by X-ray tomography is smaller than the bubble size measured by dough density for the same sample. The bubble size difference between dough measured by the different methods is seen to drop immediately after the step-change in pressure at 10 s which is in agreement with previous results (Martin et al. 2004a).

The population balance model was fitted to the complete set of dough density data from both X-ray and density measurements, using a single set of parameters. The root mean square error (RMSE) was minimised over φo, p and k by using the Generalised Reduced Gradient (GRG) Non Linear Solver function in MS Excel 2010. Approximate values for the parameters may be found by noting that φo is the initial volume, p/k is the asymptote of the final voidage and the gradient immediately after the step change is given by:

\[ \frac{dp}{dt} = -v \cdot \frac{\phi_t - \phi_f}{p_k} \]  

The model appears to be in reasonable agreement using the fitted values of initial voidage φo = 0.064, entrainment rate \( v = 0.0012 \) s⁻¹ and entrainment coefficient \( k = 0.0157 \) s⁻¹, such that the steady state voidage was φs = 0.067.

Two hundred sets of synthetic data were calculated using the Monte Carlo technique presented by Pomm et al. (2006) and introduced in Section 1.2. The standard deviations \( \sigma_{\phi_o} = 0.0044, \sigma_{\phi_f} = 0.0030, \sigma_{v} = 0.0044, \) and \( \sigma_{k} = 0.0048 \). These standard deviations
represent 7.2%, 2.3%, 2.2%, and 5.4% of the fitted values, respectively. Thus it is apparent that there was reasonable confidence in the initial voltage $v_0$ and the ratio $r_0r$, but that there was considerable uncertainty about the individual values of the correlation coefficients $\gamma$ and $\lambda$. This uncertainty does not impact on the current study, which limits itself to assessing the potential of the model to represent measured bubble size distribution data. However, it does demonstrate the importance of obtaining experimental data over several different pressure steps, including step-ups and step-downs, in order to ensure the confidence with which $\gamma$ and $\lambda$ can be fitted.

3.4. Discussion

Fig. 4 presents the normalized bubble size distributions measured after the step-decrease in mixing pressure. The shape of the size distributions is strongly weighted by the choice of a number of volume fields, in all cases around 80% of the bubbles by number are smaller than the mean size, whereas around 70% of bubbles by volume are greater than the mean size. Dough bubble size distributions are typically sigmoidal in shape in agreement with previous reports, e.g. Euliss et al. (2012). The bubble resolution cut-off of 113 voxels (43-85 µm effective diameter) appears to exclude a part of the size distribution, as also evidenced by the smaller bubble visible in Fig. 1. The resolution cut-off has the advantage that the size distribution presented is accurate, but parameters which depend on the total number of bubbles will be affected, and must be considered as the value above the cut-off.
following a pressure step-decrease. The size distribution for a dough mixed at constant pressure for the full duration lies close to the size distribution before the step-decrease. The size distributions are based on measurement of a large number of bubbles (>2.5×10⁵) but only from a single dough piece from a single dough mix. Whilst the size distribution is considered to be accurate, a more extensive sampling method would yield results which are representative of the average dough bubble state.

The model was fitted to the data by selection of a suitable model for entrained bubbles (8.9×10² µm³), equivalent to a spherical bubble of diameter of 109 µm such that the model and measured data agree at mixing time 110 s. The model trends for mixing times after this point are only dependent on the parameters fitted to the dough viscosity data - the model was not fitted to the number density or mean bubble size data. Fig. 5(a) shows both the measured and modelled number of bubbles per volume of dough over the course of mixing for constant mixing pressure and following the pressure step-decrease. The number density ranged from 45 to 56 bubbles mm⁻³, these are the same order of magnitude as those reported by Boland et al. (2009). The measured bubble density is seen to decrease immediately following the pressure step-decrease, then increase again above the initial level by the end of the mix, but still remain lower than the number density for a dough mixed at constant pressure. The fit of the model is encouraging as it displays the same trends. The model under predicts the number density and may be a result of neglecting any bubble break-up.

Both the model and the bubble size distribution measurements neglect the effects of the smallest bubbles. The smallest bubble size incorporated in the model for the system studied is the entrained bubble volume (equivalent to a spherical bubble of diameter of 119 µm). The smallest bubble size measured had effective spherical bubble diameters of 43-65 µm for the different samples. Thus, neither the model nor the measurements incorporate the contribution of smaller bubbles and, as such, their results may be considered as being approximately commensurate. Significant numbers of sub 50 µm bubbles may well be present in the dough and their incorporation into measurements and modelling could dramatically affect number densities and mean volumes. However, the results show that these bubbles do not contribute significantly to the dough viscosity. In addition, previous studies (e.g. Shah et al., 1996) have shown that the increased internal pressure of such small bubbles limits their growth during proving and baking, and thus they are not principal contributors to final bread crust structure.

Fig. 5(b) shows both the measured and modelled mean bubble volume. The measured mean volume is seen to decrease for some time after the pressure step-decrease before levelling off after 180 s of mixing. The measured mean volume for constant pressure mixing is also seen to decrease, but by a much lesser extent. Again the model agreement to these trends is very encouraging - displaying the same trends and similar magnitudes. The model over predicts the mean
bubble volume and again this would be consistent with bubble break-up occurring during mixing which has been neglected in the model.

The agreement between the dough volume, bubble number density and mean bubble size for the model may be considered to be very good given the simplifying assumptions made. The model predicts the significant processing effect of vacuum mixing – the reduction in both number density and bubble volume. It is demonstrated that the decrease in number density is much more short lived than the decrease in size which is permanent. The model is fitted to volume data using only three parameters; such data are readily and inexpensively obtained from dough density measurements. Thus the ‘no bubble break-up’ model is shown to provide a practical and reasonably accurate characterisation of pressure vacuum mixing, and confirms that the assumptions made in the model are reasonable. A further characteristic based on only dough density measurements may be used to estimate aeration rate, oxygen availability and optimal time for pressure step-down to achieve the desired final bubble size distribution.

4. Conclusions

This paper has presented a novel study of the dynamics of aeration in an opaque, highly non-Newtonian fluid – namely bread dough – by a combined study of dough density and X-ray tomography. To the authors’ knowledge, this is the first time X-ray tomography has been utilised to image particles at points during the processing of an opaque fluid and to integrate the results into a population balance model. It has been shown that:

- Dynamic changes in the bubble size distribution of a bread dough during pressure-step mixing can be accurately measured using a laboratory X-ray source. However, dough viscoelasticity can be more conveniently and accurately calculated by measurement of dough density.

- The measured bubble size distributions show a continuous range of bubble sizes, with diameters as low as 3.5 mm, which is consistent with the measured distribution of dough density, which is known to be the result of significant break-up processes during mixing.

- The response of the cumulative volume of pressure-step change during mixing can be represented very well using the simplified population balance model of Martin et al. (2000a). This model assumes that all entrained bubbles are the same size, no bubble break-up or coalescence occurs and the likelihood of bubble bloom is proportional to bubble volume.

- The simplified population balance model has been shown to be robust and of widespread utility. The model gives useful and meaningful interpretations of the aeration process, even though it assumes a small number of discrete bubble sizes, and it can be applied using only dough viscoelasticity measurements without the need for measurement of the bubble size distribution.

- The measured response of the bubble number density and mean volume agrees reasonably well with that predicted by the simplified model (with parameters fitted to only cumulative viscoelastic data). The observed deviation is suggestive of the neglected bubble break-up events.

- It is demonstrated that the decrease in number density following a pressure step-down is much more short lived than the decrease in size which is permanent.

Nomenclature

Roman

- $F_b$: Bubble number density
- $k$: Bubble disentainment coefficient
- $N_k$: Number of bubbles entrained into the dough per unit volume of dough per second
- $P$: External pressure on dough
- $t$: Mixing time
- $V$: Bubble volume
- $v$: Volume of bubbles entrained per unit volume of dough per second
- $w_d$: Frequency of disentainment events per unit volume of dough per second

Greek

- $\phi$: Dough viscosity

Subscripts

- $i$: Immediately after
- $0$: Initial state
- $1$: First mixing period
- $2$: Second mixing period
- atm: Atmospheric
- B: Bubbles transformed by pressure step-change
- in: Flowing into dough
- step: Pressure step-change

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