POWDER COHESION AND STICK-SLIP FAILURE IN A SHEAR CELL

by

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Only a fool shall say there is no God.
EXECUTIVE SUMMARY

The overriding aim of this project is to gain an understanding of the flowability of powders. Industrial interest for this study was provided by Centura Foods, U.K. who supplied three samples for study. These powder samples are starch, maltodextrin and hydrolysed vegetable protein (HVP).

The first half of this project addresses the influence of adsorbed moisture on the flowability of these powders with strong emphasis on the value of cohesion. There is evidence in the literature suggesting that the prevailing relative humidity has an influence on powder cohesion. The general trend indicates an increase in cohesion with an increase in relative humidity. Some attempts have been made in the literature to relate this humidity dependence to powder properties notably the propensity for water sorption.

Material characterisation studies revealed the morphological and surface characteristics of the samples through Scanning Electron Microscopy (SEM). It revealed the angular and amorphous appearances of the maltodextrin and HVP particles respectively. The starch particles appear to have distinct groups of larger and smaller particles. The larger particles are ellipsoidal and the smaller particles are near perfectly spherical.

Water sorption studies using a gravimetric analyzer showed that the starch and maltodextrin powders had on average 19 and 25 % mass increase respectively when subjected to a relative humidity change from 0 % to 90 %. However, the HVP powders had the largest percentage mass uptake of over 100 %. This was supported by in-situ humidity microscopy results that showed significant swelling with dissolution when exposed to saturated air. Comparatively but to a lesser degree, similar behaviour was observed for maltodextrin powders. Starch particles also exhibited swelling under saturation conditions.

Powder flowability studies were carried out using a Jenike-type shear cell. Commissioning of the shear cell was conducted using limestone powders. Measurements
were carried out at dry, ambient and saturated conditions. This was facilitated by a newly
designed in-situ humidity control system. Values of cohesion were obtained by
constructing a family of yield loci for each material. HVP powders showed a significant
increase in cohesion with humidity. Maltodextrin and starch powders showed no
significant change in cohesion with humidity. However, both powders were more
difficult to stir with changes to the relative humidity thereby highlighting that the shear
cell in this study is not sensitive enough to detect the changes in flowability.

The phenomenon of stick-slip failure was observed for starch at all humidities, and for
maltodextrin but only at 0 %RH. Published information implies that stick-slip behaviour
is linked to the rate of stored elastic energy and frictional dissipation. A study of stick-
slip observed with starch powder was conducted under four levels of consolidation over
four different velocities.

A mathematical model has been applied which describes the various stages of stick-slip
and consequently identifying the parameters that influence stick-slip. A comparison of
the model with experimental values highlighted the effectiveness of the model.

Results from this study identified the levels of consolidation and drive velocities as the
governing parameters influencing stick-slip, with the level of consolidation argued to
have a greater impact.

It was anticipated that the study would identify conditions to eliminate stick-slip.
Although this was not achieved, the study however presents \( \Omega \), a measureable which
represents the extent of stick-slip. On this basis, stick-slip is argued to be minimized
under a combination of high consolidation loads and velocities.

The study has identified possible areas for future work. Firstly, some irregular trends
were observed when comparing some results under the two intermediate consolidation
levels (4 kN/m² and 12.5 kN/m²). It is possible to further investigate stick-slip failure
with smaller increments to the level of consolidation. It is possible to add to the work
done in this study by investigating the effects of higher consolidation and velocities and testing on other materials. Finally, a parallel study repeating the work done in this study (and incorporating the proposed ideas for future work) using a different type of shear tester remains a good candidate for research.
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To my family – I thank God for you and I am indebted to you for all your love, prayers and support over the years. I love you all. Mum & dad I am especially pleased to be able to share this milestone with you. Your investments in us will not fail but will blossom in the name of Jesus – Amen! I dedicate this landmark to you both.

To my son Christian – thank you for the smiles and the drive to keep going on. This achievement pales in comparison to the heights you shall reach.

To my wife Sarah – bone of my bone and flesh of my flesh. A virtuous woman who can find? By His grace you surpass them all. Thank you for your relentless sacrifice and support. I dedicate this book to you, Christian and the others to follow.

To Him who has done exceedingly, abundantly, above all I could have ever asked or fathomed; Almighty God I give you all the glory for with you all things are possible. Again I say, only a fool shall say there is no God!
CONTENTS

EXECUTIVE SUMMARY I
ACKNOWLEDGEMENTS IV
CONTENTS V
NOTATION XI
LIST OF FIGURES XII
LIST OF TABLES XXIII

1. INTRODUCTION 1
   1.1. Background 1
   1.2. Industrial Challenge 2
   1.3. Report Summary 3

2. LITERATURE SURVEY 6
   2.1. Powder Flowability Measurement 6
       2.1.1. Introduction 6
       2.1.2. Funnel / discharge flow tester – flow time tester 6
       2.1.3. Shear Testers 7
       2.1.4. Description of other shear testers 10
       2.1.5. Conclusion 17
   2.2 Parameters Influencing Powder Flowability 18
       2.2.1. Introduction 18
2.2.2 Effect of ambient relative humidity on powder flowability

2.2.2.1 Adsorption and interparticle forces

2.2.2.2 Influence of relative humidity on powders

2.2.3 Influence of powder physical properties on cohesion

2.3 Understanding Stick-Slip Behaviour in Powder Flow

2.3.1 Introduction

2.3.2 What is stick-slip?

2.3.2.1 Static and kinematic friction

2.3.2.2 Particle-particle configuration

2.3.2.3 Bulk powder behaviour

2.3.3 Phenomenological analysis of parameters governing parameters

3. MATERIALS AND METHODS

3.1 Experimental Materials

3.1.1 Introduction

3.1.2 Description of experimental materials

3.2 Material Characterisation Studies

3.2.1 Introduction

3.2.2 Scanning Electron Microscopy (SEM)

3.2.3 Water sorption studies

3.2.4 In-situ microscopy test

3.3 Shear Cell Studies – Part 1

3.3.1 Introduction

3.3.2 Design development and commissioning of instrument
3.3.2.1 The design 63
3.3.2.2 The development 66
3.3.3 Validation of shear cell technique with limestone powder 69
3.3.3.1 Equipment 69
3.3.3.2 Experimental procedure 70
3.3.4 Variation of cohesion with humidity 74

4. COHESION MEASUREMENT 76
4.1 Introduction 76
4.2 Results from Shear Cell Study 76
   4.2.1 Limestone - Validation of technique 76
   4.2.2 Starch 79
   4.2.3 Maltodextrin 82
   4.2.4 HVP 85
4.3 Discussion 88
   4.3.1 Introduction 88
   4.3.2 Cohesion Measurement 86
   4.3.3 Starch 90
   4.3.4 Maltodextrin 92
   4.3.5 HVP 93

5. EXPERIMENTAL EVIDENCE FROM STICK SLIP STUDY 95
5.1 Introduction 95
5.2 Trace Evaluation across different Normal Stress Levels 96
5.3 Analysis of Experimental Traces from Shear Cell Studies 114
Appendix E  Graph of Shear Cell Velocity as a function of Motor Setting
Appendix F  Graphs of Effect of Shear Cell Velocity on Observed Stick-Slip Frequency
Appendix G  Graphs of Effect of Shear Velocity on $F_{\text{max}}$
Appendix H  Graphs of Effect of Shear Velocity on $F_{\text{min}}$
Appendix I  Graphs of Effect of Shear Cell Speed on Average Amplitude
Appendix J  Graph of Shear Modulus (G) as a function of Normal Stress (over a range of Velocities)
Appendix K  Graphs of Elastic Constant (k) as a function of Shear Cell
Appendix L  Graphs of Static Coefficient of Friction
Appendix M  Graph of Dynamic Coefficient of Friction
Appendix N  Graph of Duration of Stick – Measured and Calculated
Appendix O  Graph of Duration of Slip – Measured and Calculated
Appendix P  Graphs of Dynamic Coefficient of Friction – Measured and Calculated
Appendix Q  Graphs of Yield Time – Measured and Calculated
Appendix R  Graphs of (i) Stick-Slip - Measured and Calculated values, and (ii) corresponding graphs showing the Slip Phase using the Dynamic Model

Appendix S  Spreadsheet of Stick-Slip Study
**NOTATION**

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>$A$</td>
<td>Constant in dynamic model – Chapter 6</td>
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<tr>
<td>$B$</td>
<td>Constant in dynamic model – Chapter 6</td>
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<tr>
<td>$b$</td>
<td>thickness of powder (in shear cell)</td>
</tr>
<tr>
<td>$C$</td>
<td>Cohesion</td>
</tr>
<tr>
<td>$D$</td>
<td>Diameter of orifice in funnel/discharge flow tester</td>
</tr>
<tr>
<td>$d$</td>
<td>Diameter of top ring (shear cell)</td>
</tr>
<tr>
<td>$F$</td>
<td>Generally used to denote force</td>
</tr>
<tr>
<td>$F_d$</td>
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<td>$F_k$</td>
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</tr>
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<td>Duration of initial slip</td>
</tr>
<tr>
<td>$t_y$</td>
<td>Duration of initial stick (yield time) using simple model</td>
</tr>
<tr>
<td>$T$</td>
<td>Tensile strength</td>
</tr>
<tr>
<td>$\tau_y$</td>
<td>Yield stress using simple model</td>
</tr>
<tr>
<td>$\tau_{max}$</td>
<td>Maximum shear stress equivalent to critical shear stress</td>
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<td>Minimum shear stress equivalent to shear stress at $t_{end\ of\ slip}$</td>
</tr>
<tr>
<td>$UYS$</td>
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<tr>
<td>$v$</td>
<td>Velocity</td>
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<td>$\omega$</td>
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<td>$\mu_d$</td>
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</tbody>
</table>

$A$, $B$, $C$, $D$, $d$, $F$, $F_d$, $F_k$, $F_s$, $F_{end\ of\ slip}$, $G$, $g$, $k$, $M$, $RH$, $t$, $t_{slip}$, $t_{stick}$, $t_{end\ of\ slip}$, $t_y$, $T$, $\tau_y$, $\tau_{max}$, $\tau_{min}$, $UYS$, $v$, $\omega$, $x$, $\mu_d$, $\mu_k$
<table>
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</tr>
</thead>
<tbody>
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<td>Static coefficient of friction</td>
<td></td>
</tr>
<tr>
<td>$\sigma$</td>
<td>Principal consolidation stress</td>
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</tr>
<tr>
<td>$\gamma$</td>
<td>Shear strain</td>
<td></td>
</tr>
<tr>
<td>$\Omega$</td>
<td>Extent of stick-slip</td>
<td></td>
</tr>
</tbody>
</table>
# LIST OF FIGURES

<table>
<thead>
<tr>
<th>Figure</th>
<th>Title</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.1</td>
<td>Examples of shear testers.</td>
<td>8</td>
</tr>
<tr>
<td>2.2</td>
<td>Basic features of the Jenike shear cell.</td>
<td>11</td>
</tr>
<tr>
<td>2.3</td>
<td>Cross-section of a rotational shear tester.</td>
<td>13</td>
</tr>
<tr>
<td>2.4</td>
<td>Theoretical tensile strength of agglomerates with different bonding mechanisms.</td>
<td>19</td>
</tr>
<tr>
<td>2.5</td>
<td>Pendular state of water content for an assembly of spherical particles.</td>
<td>25</td>
</tr>
<tr>
<td>2.6</td>
<td>Funicular state of water content for an assembly of spherical particles.</td>
<td>25</td>
</tr>
<tr>
<td>2.7</td>
<td>Capillary state of water content for an assembly of spherical particles.</td>
<td>26</td>
</tr>
<tr>
<td>2.8</td>
<td>Simplistic sketch of a drop state of water content for an assembly of spherical particles.</td>
<td>26</td>
</tr>
<tr>
<td>2.9</td>
<td>Schematic of the rearrangement of solid spherical particles during shear.</td>
<td>34</td>
</tr>
<tr>
<td>3.1</td>
<td>SEM image of starch powder.</td>
<td>45</td>
</tr>
<tr>
<td>3.2</td>
<td>SEM image of maltodextrin powder showing the different types of granules.</td>
<td>46</td>
</tr>
<tr>
<td>3.3</td>
<td>SEM image of a single maltodextrin granule.</td>
<td>46</td>
</tr>
<tr>
<td>3.4</td>
<td>SEM image of HVP powder.</td>
<td>47</td>
</tr>
<tr>
<td>3.5</td>
<td>SEM image of a single HVP granule highlighting its hollow structure.</td>
<td>48</td>
</tr>
<tr>
<td>3.6</td>
<td>Water sorption isotherm for starch powder.</td>
<td>50</td>
</tr>
<tr>
<td>3.7</td>
<td>Water sorption isotherm for maltodextrin powder.</td>
<td>51</td>
</tr>
<tr>
<td>3.8</td>
<td>Water sorption isotherm for HVP powder.</td>
<td>52</td>
</tr>
<tr>
<td>3.9</td>
<td>In-situ microscopy images of dried starch.</td>
<td>55</td>
</tr>
<tr>
<td>3.10</td>
<td>In-situ microscopy image showing starch under saturation conditions after 24 hrs.</td>
<td>55</td>
</tr>
<tr>
<td>3.11a</td>
<td>Optical micrograph of maltodextrin particles under dry conditions, ((t = 0 \text{ hours})).</td>
<td>56</td>
</tr>
<tr>
<td>3.11b</td>
<td>Optical micrograph of maltodextrin particles under dry conditions, ((t = 24 \text{ hours})).</td>
<td>56</td>
</tr>
<tr>
<td>3.12a</td>
<td>Optical micrograph of maltodextrin particles under saturation conditions, ((t = 0 \text{ hours})).</td>
<td>57</td>
</tr>
<tr>
<td>3.12b</td>
<td>In-situ microscopy images showing the rapid swelling response of maltodextrin granules after 1 hr. exposure to saturation humidity.</td>
<td>57</td>
</tr>
<tr>
<td>3.12c</td>
<td>In-situ microscopy images showing the changes to the maltodextrin granules following 2 hrs. exposure. No further changes were observed after this time.</td>
<td>58</td>
</tr>
</tbody>
</table>
3.13a Optical micrograph of HVP powder under dry conditions (t = 0 hours).

3.13b Optical micrograph of HVP powder under dry conditions (t = 24 hours).

3.14a Optical micrograph of HVP powder under saturation conditions, (t = 0 hours).

3.14b In-situ humidity microscopy images showing the rapid response of an HVP particle when exposed to saturated conditions.

3.15 Schematic of the shear cell (not drawn to scale).

3.16 The modified shear cell in assembled form (a), and close up of the circular base and top ring with reference to the sintered brass plate.

3.17 Process flowsheet of the system design.

3.18a Photograph of the mixing chamber.

3.18b Inside view of the mixing chamber showing the stainless steel baffle used for effective mixing.

3.19 Schematic design of the modified shear cell.

3.20 Graph showing the calibration results comparing experimental to published values.

3.21 Initial and final offset position of the shear cell before and
after shearing respectively.

3.22 An example of preshear stress-strain curves for under, critical and over-consolidated samples.

3.23 An example of a chart recorder output showing the stress-strain curves for preshear and shear stages as observed in a shear cell experiment.

4.1 Graph comparing the yield loci of experimental data conducted in the current study to published results.

4.2 Yield Loci for limestone under variable humidity using average values.

4.3 An example of the stick-slip failure pattern for starch.

4.4 Yield loci for starch powders under variable humidity using mid-point values of the stick-slip oscillations.

4.5 Yield loci for maltodextrin powders under variable humidity using mid point values for 0 %RH data (stick-slip) and peak values for ambient condition data.

4.6 Evidence of over-consolidation-type response during preshear by maltodextrin powder (0% RH).

4.7 Yield loci for HVP powder under variable humidity.

4.8 Evidence of over-consolidation type response during preshear by HVP powder.

4.9 Effect of granular size change on contact area/number
in a fixed volume (i.e. shear zone).

5.1 Observed Frequency as a function of shear cell velocity for all consolidation loads studied.

5.2 Critical shear stress $\tau_{max}$ as a function of the shear cell velocity.

5.3a-d $\tau_{max}$ as a function of the shear cell velocity.

5.4 $\tau_{min}$ as a function of shear cell velocity.

5.5a-d $\tau_{min}$ as a function of shear cell velocity.

5.6 Change in average amplitude as a function of shear cell velocity.

5.7 Percentage ratio of amplitude of stick-slip to $\tau_{max}$ as a function of shear cell velocity.

5.8 Schematic of bulk powder used to illustrate the relationship between $k$ and $G$.

5.9 Shear Modulus (log) as a function of shear cell velocity under all consolidations investigated.

5.10a-d Shear Modulus as a function of shear cell velocity.

5.11 Static Coefficient of friction $\mu_s$ as a function of shear velocity for all consolidations.

5.12 Dynamic coefficient of friction $\mu_d$ as a function of shear velocity.
5.13a The trace captures the initial stick peaking at a steady constant value $\tau_{\text{max}}$ as identified in the experiment marked “2”.

5.13b The next sequence of flow patterns for these runs shows stick-slip failure for the runs marked 3 and 1. Observe that both runs maintain $\tau_{\text{min}} = 0$ at the end of slip.

5.13c The next sequences shows a fast reoccurring stick slip for the run marked “1” with steady failure maintained for the other two runs.

5.13d Next sequence showing stick-slip occurring for run marked “2” with stable failure maintained for long periods for the other two runs.

5.13e This next sequence shows the two responses to slip observed where the run marked “1” shows prolonged $\tau_{\text{min}} = 0$ followed by a second slip, and runs marked “2” and “3” begin stick immediately following slip.

5.13f Next sequence showing a good example of prolonged stable failure exhibited by all examples.

5.13g In this next sequence, experiment “1” shows a clear example of a “pair” of stick-slip occurring.

5.14a Start of a sequence of failure patterns highlighting reduced duration at $\tau_{\text{min}} = 0$ compared with Fig. 5.13.

5.14b Trace represents the last portion in this series (not in sequence with Fig. 5.14a) which highlights measurable shear stress for
the second of the “pair” of stick-slip (i.e. $\tau_{\text{min}}$ is not equal to zero).

5.15a Start of a sequence of failure patterns.

5.15b Next sequence showing “pairs” of stick-slip occurring in between periods of stable failure. The second of the “pair” of slips can be seen to be clearly above $\tau_{\text{min}} = 0$.

5.16 Trace showing the failure pattern at maximum velocities tested. Observe the reduction in the amplitude of the second of the “pair” of stick-slips.

5.17a Trace highlighting the observed elongated slip starting at $\mathbf{X}$ and prolonged stick ending at $\mathbf{Y}$.

5.17b Trace showing similar observations at an increased velocity of $13 - 16 \text{ mm/min}$.

5.18a Failure pattern at the start of two experiments showing two different failure patterns.

5.18b Trace showing next sequence highlighting irregular failure.

5.18c Trace showing a section of the irregular failure patterns observed.

5.18d A portion of the trace highlighting the irregular failure pattern observed.

5.19a Trace showing initial slip at shear stress lower than $\tau_{\max}$ $(3.4 \text{ mm/min})$.

5.19b Trace showing next sequence of slip occurring at higher shear stress, $\tau_{\max}$. $(3.4 \text{ mm/min})$. 
5.20a Trace showing initial slip at shear stress lower than $\tau_{\text{max}}$ (5 - 6 mm/min).

5.20b Trace showing next sequence of slip occurring at higher shear stress, $\tau_{\text{max}}$ (5 - 6 mm/min).

5.21 Trace showing slip occurring before the powder reaches $\tau_{\text{max}}$ when shear is carried out at maximum drive velocities of 22 - 23.5 mm/min.

5.22a Trace highlighting stable flow prior to initial slip (2.5 - 3 mm/min).

5.22b Next sequence highlighting significant reduction in amplitude (2.5 - 3 mm/min).

5.22c Trace showing failure as shear progressed highlighting an increase in the frequency of stick-slip and with reduced amplitudes (2.5 - 3 mm/min).

5.23a Trace highlighting stable flow prior to initial slip (7.6 mm/min).

5.23b Next sequence highlighting significant reduction in amplitude (7.6 mm/min).

5.23c Trace showing failure as shear progressed highlighting an increase in the frequency of stick-slip and with reduced amplitudes (7.6 mm/min).

5.24 Trace capturing the development of the failure pattern as shear progressed highlighting an increase in the frequency of stick-slip and with reduced amplitudes (16.4 mm/min).
5.25 Comparison of the extent of stick-slip, $\Omega$, over the range of data for measured and calculated values. 135

6.1 Schematic of bulk powder used to illustrate the relationship between $k$ and $G$. 144

6.2 Schematic of pulley driven system. 146

6.3 Schematic of shear cell setup highlighting forces in action. 149

6.4 Sketch of the model in operation. 153

6.5 Sketch of a typical saw-toothed stick-slip trace annotated with some parameters together with their definitions where possible. 155

6.6a-d Comparison of the duration of stick. 158

6.7a-d Comparison of the duration of slip 160

6.8 Comparison of the measured and calculated values of dynamic coefficients of friction. 163

6.9 Graph showing an example of an actual trace alongside a theoretic trace using the dynamic model. 164

6.10a Start of sequence showing initial failure characterised by classic saw-toothed stick-slip. 165

6.10b Next sequence showing apparent stable failure characterised by plateaus before slip occurs (also refer to Section 5.3). 165

6.10c Final sequence showing rapid saw-toothed stick slip with noticeable reduced amplitude. 166
6.11 Graph showing an example of an actual trace alongside a theoretic trace using the dynamic model. A scan of the experimental trace is included and discussed in Fig. 5.18 marked “2”.

6.12 Graph showing an example of an actual trace alongside a theoretic trace using the dynamic model. A scan of the experimental trace is included and discussed in Fig. 5.17b marked “3”.

6.13 Graph showing an example of an actual trace alongside a theoretic trace using the dynamic model. A scan of the experimental trace is included and discussed in Fig. 5.16 marked “3”.

6.14 Applying the Dynamic Model, \( F = \mu_d Mg + (\mu_s - \mu_d) Mg \cos(\omega t) \), to show frictional decay during the slip phase during “Run 44”.
## LIST OF TABLES

<table>
<thead>
<tr>
<th>Table</th>
<th>Title</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.1</td>
<td>Results from the calibration of the RHM squirrel using salt solutions compared with the range of values reported by Young [39]</td>
<td>68</td>
</tr>
<tr>
<td>4.1</td>
<td>Comparison of experimental values of cohesion with published values</td>
<td>82</td>
</tr>
<tr>
<td>6.1</td>
<td>An example of the application of the model (“Run 44”)</td>
<td>157</td>
</tr>
</tbody>
</table>
CHAPTER 1: INTRODUCTION

1.1. BACKGROUND

The evaluation of powder flow properties is essential for designing and optimising powder processing operations [1]. A change in the relative humidity of the environment is known to influence powder flow properties by modification of inter-particle forces. This modification is directly linked to the adsorption of moisture on the particle surface. One of the most significant and rigorously defined powder flow properties is bulk cohesion. This can be considered to represent the inherent strength or resistance of a powder to deformation when it is unconfined. Orband and Geldart [2] describe it more concisely, as the shear strength needed to cause failure of the powder when no normal load is applied to the plane of shear.

Powder manufacturing and handling plants are susceptible to climatic change. A common feature is the fluctuation of the ambient relative humidity. The modification of powder cohesion by water sorption is significant for powder product quality, and for the effectiveness of process operations such as powder flow, compaction, agglomeration, mixing and fluidisation. The rate and extent of water sorption in powders are therefore important when attempting to understand the mechanisms underlying powder cohesion. As part of understanding cohesion in food powders, an arm of this research study is to investigate the influence of adsorbed moisture on powder cohesion.

The variance in the cohesion of bulk powder is underlined by a change to the flow pattern i.e. the ease or difficulty of the powder to flow with a change in ambient humidity. Some bulk powders display stick-slip behaviour under flow. This intermittent action of flow to no-flow action has been linked to handling problems such as silo honking and silo quaking. Evidence of stick-slip prompted a thorough study on the governing parameters. Starch powder which showed stick-slip under all humidity conditions tested was chosen as a candidate for this study.
Chapter 1: Introduction

For the purpose of gaining a broad understanding of cohesion, this survey has addressed evidence obtained from a broad spectrum of granular materials. However, the initial motivation for the work came in response to a quest for more detailed understanding of powder behaviour from Centura Foods. Centura process a large number of cohesive powders, blending these ingredients with fat and extruding to produce gravy granules. Emphasis has therefore been placed on obtaining relevant published information on food powders, and particularly on specific powders of interest to the study, i.e. potato starch, maltodextrin and hydrolysed vegetable protein.

1.2. INDUSTRIAL CHALLENGE

Centura use an extrusion process in order to produce gravy granules from a mixture of cohesive powders and oils. The procedure is a batch system and is outlined below (see Fig: 1.1) in a process flow diagram. The flavours are arranged in layers on trays exposed to ambient temperature and humidity. The flavours are fed into a sack tip unit (STU) together with salt, flour and maltodextrin. From the STU, these ingredients are mixed with bulk starch in the mixer for 60 seconds before being stored in a storage bin. Oil is added at 40 – 45 °C to the mixture in preparation for extrusion. The oil added acts as a binder holding the particle matrix together. It also acts as a lubricant in the extruder. The inlet and outlet temperatures for this process are 30 and 33 °C, respectively.

The major challenges facing Centura are the unwanted production of oversized granules, and “caramelised” granules. These problems are likely to result from a change to the properties of the powders at any stage of handling i.e. storage, tipping, flow, extrusion etc. Oversized granules can be the product of a highly cohesive mixture in the extruder. The mixture binds together emerging rather like spaghetti instead of cylindrical granules. It is possible that the caramelised product arises from excessive heating of the product.
Chapter 1: Introduction

The project aims at identifying how much change occurs to the powder flow properties with changes to the ambient humidity. As there are a number of powder handling steps, it is important for the manufacturers to be aware of the expected change to the properties of the powders before processing takes place.

![Diagram of the production process]

Fig. 1.1: Process line diagram for the production of gravy granules.

The objective of the current study is to investigate the influence of adsorbed moisture on powder cohesion and to conduct a stick-slip study on starch powder in order to identify the governing parameters.

1.3 REPORT SUMMARY:

The project unfolds in Chapter 2 with a survey of the literature identifying some of the different instruments available to measure powder flowability. This comprises the development of powder flow measuring devices, a description of various shear testers, and a critical analysis of measuring the flowability of particulate material. In addition to
this, a review of the parameters that are reported to have possible effects on flowability are discussed. Some of these parameters include ambient relative humidity and powder physical properties. Though flowability is viewed on a bulk scale, a review of the interactions on a single-particle scale is carried out by analyzing mathematical models of cohesion.

Chapter 2 concludes with a review of stick-slip from literature with a view to garner an understanding of the phenomenon and governing parameters.

In Chapter 3, the materials of interest are introduced. These materials are starch, maltodextrin and HVP powders supplied by Centura. Material characterisation tests are performed on these samples in order to gain better understanding of the nature of the materials, and their possible responses to shear under a given condition, such as a varied humidity. These characterization tests include SEM, water sorption tests and in-situ microscopy studies. In this chapter, a description of the step-by-step procedure to carry out a shear test using a Jenike shear cell is provided, together with listings of the modifications made to the Jenike-type shear cell used in this project to facilitate in-situ humidity supply to the test powder.

The results and observations from the shear cell studies are discussed in Chapter 4. The discovery of stick-slip shear behaviour demands a fundamental understanding of its origins and this is also included in this chapter. Particular interest has since developed in starch for scientific and industrial reasons, i.e. it was the only powder to exhibit stick-slip behaviour under all conditions, and it has a large proportion in the final product.
Chapter 5 reports on the stick-slip study conducted on starch powder. Results from this study are vital to identifying experimentally the parameters governing stick-slip. Also discussed in this chapter are irregular failure patterns observed throughout the study.

In Chapter 6 the stepwise development of the mathematical model is presented. It also includes a comparison of experimental results with a theoretic results obtained using the model. The limitations of the model are also presented.

A general study conclusion is presented in Chapter 7 which identifies the key contributions of this study with scope for future work outlined also in the same chapter.
Chapter 2: Literature Survey

CHAPTER 2: LITERATURE SURVEY

2.1 POWDER FLOWABILITY MEASUREMENT

2.1.1 Introduction

This chapter reviews the wide range of techniques available for measuring powder flowability. These range from simpler techniques using the funnel/discharge flow tester, through to the more thorough approach using shear testers. A review of the available techniques allows for an evaluation of the suitability of choice of technique to employ for the current project. It will be shown in this document that the technique to be employed would need to be suitable to allow for calculation of the powder strength (taken to be cohesion), and the detailed analysis of the effect of normal stress and strain rate on the failure pattern.

2.1.2 Funnel / discharge flow tester – flow time tester

The dynamic flow properties of a powder are often assessed by determining the rate of powder flow from a standardised conical hopper. This method is frequently used in the pharmaceutical industry to see if a given powder flows at a sufficient rate. The funnel flow tester consists of a series of miniature glass hoppers, or funnels, each with a different sized orifice, and each partially filled with powder [3]. The powder is shaken vigorously by several inversions and re-inversions of the hopper, and then left to stand. The time taken for the powder to flow is recorded.

de Jong et al., [4] describe the principles and operation of the flow funnel. They state that in general, the mass flow rate of a bulk powder flowing through an orifice of diameter $D$ is proportional to the bulk density of the powder. If a fixed orifice size is used, the volumetric flow rate of the powder can be used as an indication of its flow
properties. Another approach is to find the minimum orifice diameter necessary for flow and use this as a flowability characteristic.

According to Sutton [3] the funnel flow tester effectively measures the strength of a free arch of powder. It can be used as a quick method for evaluation of time consolidation [3]. This can be evaluated by a simple method of measuring the time taken to empty a fixed mass of powder flowing through the same orifice size contained in the funnel over different lengths of time before being discharged.

This equipment is simple but useful for preliminary design work or ranking different powders. It is also relatively inexpensive compared to other shear testers available presently. However, the information generated is empirical and does not lead towards the development of any mechanistic understanding of flow behaviour.

2.1.3 Shear Testers

The use of shear testers is widely acknowledged to originate from soil mechanics. Early work done to study the strength, resistance to flow and flow patterns of soil paved the way for the development of shear testers as used today. Civil engineers use shear testers to conduct studies on soil material used in building, construction, foundation laying etc. There is a range of flow measuring devices that have been developed, and their selection depends upon the application and type of material. Schwedes and Schulze [5] report that shear testers range from simple practical devices which are easy to use, to more sophisticated instruments which give complete information on all stresses and strains acting on a sample.

Shear testers can be ‘direct’ or ‘indirect’. For direct shear testers the shear zone is defined explicitly by the movement of one part of the instrument relative to a stationary part, whereas in indirect shear testers the shear zone develops unhindered according to
the applied state of stress, [5]. Direct shear testers can be translational or rotational in operation, and are also known as shear cells. Examples of some shear cells will be described later in this section.

![Diagram of shear testers](image)

**Fig 2.1: Examples of shear testers [5]**

**Direct shear testers:**
There are a number of shear cells available with differing operational properties. The principles governing most types of shear cells have strong similarities. Shear cell testers measure the resistance to flow of critically consolidated powders under a varied normal load. The results from shear cell testers can be used to determine the intrinsic strength of a powder i.e. cohesion and tensile strength. Industrial powder handling relies on accurate shear testing for design and quality control purposes. Shear testers are used, [1], to provide the basic data for design of silos and powder handling equipment like chutes and screw feeders. Feise [1] reports that negligence at this stage of design generally leads to costly modifications (e.g. ‘no-flow’, failure of equipment walls) and/or unsatisfactory performance of the equipment ('partial flow-partial blockage').
Chapter 2: Literature Survey

There are two types of direct shear testers as shown in Fig. 2.1, these are the translational and rotational type shear testers. The basic features of both shear testers are summarised:

The translational shear tester shears bulk powder within a cell volume by lateral linear displacement of the top half of the sample relative to the bottom half. Examples of these types of shear cell are described in Section 2.1.4.

The rotational shear tester uses an annulus or disc-shaped trough filled with bulk powder. The trough is covered with a lid subject to a normal stress. The lid is rotated at a constant angular velocity causing shear to the powder due to the relative displacement of the shear cell to the lid. The torque necessary to rotate the lid is measured from which the shear stress is calculated.

Indirect shear testers:

The biaxial and triaxial testers are cells that allow the sample to be subjected to well defined external stresses and strains. The shear plane is free to develop internally within the sample. Biaxial and triaxial testers are examples of indirect shear testers commonly used in soil mechanics. According to Wood [6] triaxial shear testers are the most widely used testing apparatus for investigating the stress-strain behaviour of soils. In general, indirect shear testers are designed for application of high stresses and small strains. They are therefore not best suited to powders in a processing context, [7] and will not be considered further.

Another example of an indirect shear tester is a uniaxial tester. A typical uniaxial tester is the unconfined yield stress tester which is described later in this chapter. For these types of devices the stress and strain is controlled in one direction only.
2.1.4 Description of other Shear Testers

Reviews of the performance of shear testers have been widely published in the literature, e.g. [5, 8]. A summary of some of the more 'popular' shear testers is documented here. These include:

Jenike shear cell
Annular shear cell (or ring shear tester)
Direct shear box
Warren Spring-Bradford cohesion tester (WSBCT)
Computer controlled shear tester
Powder rheometer (Freeman)
Unconfined yield stress (UYS) tester

Jenike shear cell
The Jenike shear cell was developed by Jenike [9]. It is a well-known instrument often used [10] in characterizing powder flow properties. There have been variations to the original design by Jenike. It can also be argued that the limitations posed by Jenike’s design spawned the development of other shear cells e.g. annular shear cell [11], Warren-Spring Bradford cohesion tester [2] (see below).

The Jenike shear cell is a classical example of a translational shear tester as described previously. The cell is split into two parts; a base and a top ring (see Fig. 2.2). The powder within the cell is covered with a lid upon which weights of known masses are placed creating a normal stress downwards on the bulk powder. In some cases, consolidation is created by hanging weights from a hanger attached to the cell. The consolidation of the bulk powder is enhanced by twisting of the lid. The cell is driven mechanically at a constant speed with the top ring acting against a force transducer. The top ring is displaced from the base and the force required to displace the two halves is
recorded as a function of time. The shear force is converted to shear stress by dividing by the cell cross-sectional area.

![Diagram of Jenike shear cell]

**Fig. 2.2: Basic features of the Jenike shear cell**

The standardised procedure to carry out an effective shear test using the Jenike shear cell has been published by the IChemE [12]. A standard operating procedure is necessary because the results obtained are operator dependent. In addition to the publication by the IChemE [12], some reports in recent years [8, 13] have referred to the report by Akers [14] which gives a thorough description of the procedure, as an alternative standard method. The results obtained from the study [14] on limestone have been used as a reference to calibrate Jenike shear cells used by others [13].

The Jenike shear cell poses an operational limitation by way of a limited shear displacement [15], thereby necessitating a laborious and time consuming procedure to determine the conditions required for critical consolidation. Another effect this has is the unsuitability of the Jenike shear cell for use on elastic bulk solids (saw meal, plastic products) or products such as sewage sludge or moist dry clay [16], thereby creating material limitation.
It is reported [14] that yield locus points that lie on the left side, outside of the Mohr circle (UYS) are invalid. This can have an effect on the accuracy of the value of cohesion obtained by extrapolation of the yield locus. Using an instrument such as the Warren Spring-Bradford Cohesion Tester (WSBCT) the value of cohesion can be obtained directly. Similarly, the Unconfined Yield Shear (UYS) tester can only be obtained by the construction of Mohr circle otherwise unnecessary if an instrument such as the UYS tester was used.

Although the Jenike shear cell has its limitations, it has remained an industry-standard [16] and is still widely used [16], [11], [13], [17], [18]. As would be shown further in this section, the development and calibration of other shear testers have been referenced to the Jenike shear cell. The Jenike shear cell provides a wide range of flow properties that are not always entirely available from other shear testers. It is suggested that this quality, in addition to its tried-and-tested history, makes the Jenike shear cell a popular choice for powder flowability characterisation.

**Annular shear cell (ring shear tester)**

The annular shear cell was originally proposed by Carr and Walker [15] as an alternative powder testing apparatus to remove limitations associated with the Jenike shear cell, as described above. The annular shear cell is a rotational type shear cell (Fig. 2.3) which permits the study of flow characteristics prior to, and after failure. The design and operation of this type of shear cell has been reported previously.

The main benefits of the annular shear cell are the unlimited shear displacement and the removal of a manual preconsolidation step. Compared to the Jenike cell, tests can be conducted without the laborious and operator-dependent sample preparation to ensure that the critical state is reached.
However, in his work on the development of the ring shear tester, Schulze [16] reports on the possible drop in shear stress with increasing shear displacement. The shear displacement is the circumferential displacement between lid and shear cell. The reason for the decrease is reported to be an assumed rearrangement of the particles and the build up of horizontal shear planes with decreasing shear resistance [16]. In addition to this, the possibility of sample coarsening as a result of degradation, and modification due to prolonged strain are proposed as principal contributors to the drop in shear stress. This potential effect must be borne in mind when conducting tests with an annular shear cell.

In addition, prior to steady-state failure, the stress-state in the powder across the annulus is not uniform given by the variation in linear strain with radial position across the annulus, i.e. material at the outer edge of the annulus will reach its critical state before the material at the inner edge. This is a potential issue during incipient failure, but once critical state failure is established the variation in linear strain is not consequential.
Chapter 2: Literature Survey

**Direct shear box**

The direct shear box is an apparatus used to measure the consolidated shear strength of soil material in direct shear. It is also used to determine wall friction angles and angles of internal friction and cohesion. It is traditionally used for soil mechanics and is associated with large consolidation loads. The direct shear box cannot be used to determine the flow function of a material [17]. Its use for powder process applications is limited and therefore is not considered further.

**Warren Spring-Bradford Cohesion Tester (WSBCT)**

This device measures the cohesion of a particulate material directly, unlike other common shear cells (e.g. Jenike, annular shear testers) which allows for an indirect determination of cohesion by extrapolation of the yield locus [2]. The development of the WSBCT was used in a study to obtain a qualitative comparison as regards the flowability of powders of different sizes. It is suggested that the WSBCT in operation can be compared to the annular shear cell without a normal stress acting on the bulk powder, the benefit of which as mentioned earlier is a direct measurement of cohesion.

Though a direct measurement of cohesion is obtained, however, the WSBCT does not provide additional information needed to characterise fully the flowability of a powder, i.e. a yield locus. This would enable the determination of the unconfined yield stress and the major principal consolidation stress, thereby fixing the flow function of the powder.

A parallel drawn to the method used in the WSBCT in relation to the annular shear cell or Jenike shear cell is the assumption that critical consolidation is achieved prior to measuring the shear stress at a lower load (in this case the lower load is zero). In their account [2], Orband and Geldart did not report on a method to determine the consolidation state of the powder before shearing.
Computer controlled shear cell (CCSC-YLT)
The CCSC as described by Ladipo and Puri [20] works using the same principles as a Jenike shear cell with added computer controlled features such as cyclic loading at a monitored rate to create a normal stress. Ladipo and Puri [17] report that the CCSC can be used following the same procedure as the conventional Jenike shear cell (CCSC).

The CCSC has the advantage of being able to be used as a dynamic yield locus tester (CCSC-YLT) for which it was designed. However, the results produced using the CCSC-YLT show parity with CCSC results for more cohesive powders, and less so for less cohesive powders (i.e. granulated sugar). Ladipo and Puri [17] attributed this to a discrepancy in the determination of the point of critical consolidation. The shear phase, using the CCSC-YLT, is initiated following constant volume during preshear, as opposed to no change to the preshear stress value practised conventionally. For this reason, the points on the yield locus will fall on different values depending on the nature of the powder.

This instrument appears suitable for the current study. Work carried out using this instrument, like the majority of the shear cells, have used the results from the Jenike shear cell as a benchmark for their work, and as a key tool in evaluating their experimental data. This instrument (including the computerised package) can thus be seen as a Jenike shear cell with added control features arguably to ease protocol and to make the measurement less operator-dependent. From the viewpoint of the current study, the added features included will incur a significant amount in cost, and installation time. A significant amount of effort has been put in to minimise any operator dependency in the present work.

Powder Rheometer
The design and commissioning of the FT3 Powder Rheometer was presented by Freeman [18] as an empirical approach to the measurement of the flowability of powders. This instrument measures the torque required to stir the powder, as a means to characterise
flowability. The operating principle is to force a twisted blade along a helical path through a column of powder or other material contained in a circular vessel having a closed bottom [18]. Parallels can be drawn with the WSBCT approach though the FT3 predominantly monitors flow after failure. The energy required to induce flow and maintain a desired flow rate is taken to be a measure of the flowability of the powder [18].

This quick and easy method is a convenient way to compare qualitatively the degree of flow of powders. If the energy required to induce flow and also to maintain steady state flow is taken as a measure of flowability of powders, then the FT3 is a strong candidate. This approach rivals the use of the flow function as a measure of powder flowability used conventionally.

The FT3 has been described above to give a measure of powder flowability based on the energy required to effectively turn the blades. In his report, Freeman [18] links the effect of blade tip speed on the total energy consumed. However, as with the annular shear cell, the FT3 possesses varying radial speed along the length of the blade. As will be seen in this study the “drive” speed causing shear has an impact on the flowability measured, therefore the energy consumed is argued to be characterised by an average flowability over the varying radial speed along the blade.

Unlike the WSBCT the effect of additional normal stress acting on the bulk powder by the instrument is not accounted for. Similar to the WSBCT a normal stress can not be applied to the powder during shear. It is therefore not possible to obtain a yield locus using this instrument. Consequently the FT3 is not considered as a suitable instrument for use in quantifying the flowability of powder in the present study. However, it could find application as a secondary device used to compare any changes in the energy required to induce and maintain steady state flow.
Unconfined yield stress (UYS) tester
This is a simple technique for direct measurement of the unconfined yield stress. A bulk powder enclosed in a mould is consolidated to a known value. The original bulk powder is such that it has a low height to diameter ratio. Upon consolidation the diameter of the powder is reduced significantly by carefully slicing using a knife (or equivalent tool) leaving a column of powder. The unconfined yield strength of the cylindrical test sample is then measured by placing a polythene flask on the top. The weight is gradually increased by filling the flask with water until the sample fails [5]. The UYS is a point on the Mohr circle that describes the onset of failure of a bulk powder for a given consolidation. This is used as a measure of the cohesivity of bulk powders. This rather simple but useful method of determining the UYS does not give additional flow properties and is therefore not considered suitable to be used as the test instrument for this project.

2.1.5 Conclusion

The suitability of an instrument to be used for powder characterisation is dependent on the application of the data, i.e. the purpose for which it will be used. For the purpose of this study an instrument that is able to provide a family of yield loci is required. Both the Jenike and annular shear cell meet this criterion. The limitations associated with both instruments have been described. However, in a review of various measuring devices, Schwedes [10] pointed out that the most widely used and versatile instrument for powder characterisation was the Jenike Shear Cell.

The Jenike shear cell is a well tested instrument that has served as an industrial standard for powder characterisation, and shear cell development. On this basis, a shear cell based on the idea by Jenike is recommended for use on this project.
2.2 PARAMETERS INFLUENCING POWDER FLOWABILITY

2.2.1 Introduction

This section describes the factors affecting the flowability of powders, uncovering the effect of ambient relative humidity on the powder flowability by looking into the process of water adsorption and the effect this has on bond strength.

Also in this section, the influence of powder physical properties on cohesion is reviewed. This covers the effects of particle size and shape, and surface characteristics.

2.2.2 Effect of ambient relative humidity on powder flowability

Investigations have shown that the cohesion of a powder is influenced by the extent of water sorption, e.g. [20] and [21], which in turn is governed by the ambient relative humidity of the surrounding air. This can be attributed to a modification of inter-particle forces due to the presence of adsorbed water at the particle-particle contact points. The mechanism by which this occurs is complex, involving a combination of capillary meniscus formation, surface chemistry, surface plasticisation, surface roughness and possible dissolution or solid phase transitions. According to Tennou and Fitzpatrick [20], small differences in moisture content, particle size, storage time, and even temperature can make a significant difference in flowability.

In general, the greater the water content of a powder, the more cohesive the powder becomes and the more difficult it is to promote flow [22]. However, it must be borne in mind that water may also act as a lubricant between individual particles undergoing mutual shear. Thus there can be no hard and fast rules for predicting cohesion.
2.2.2.1 Adsorption and interparticle forces

Water adsorption is the process by which moisture from the air collects on the surface of powders, and in voids between neighbouring particles. The equilibrium moisture content of a powder is related to the relative humidity of the surrounding atmosphere by the sorption isotherm. The form of the isotherm depends upon the temperature and the affinity of the particle surface for water molecules.

In general, two regions of the isotherm can be distinguished. At low relative humidities, the moisture associated with the particles of a powder exists as a thin condensed layer, or clusters of water molecules adsorbed at specific sites. As the relative humidity increases, the thickness of the adsorbed layer also increases, until eventually condensation occurs at the contact points, generating liquid bridges, [23]. In the latter region it is widely accepted that the flow behaviour of the powder substantially deteriorates, [24]. This is due to the large increase in inter-particle adhesive force associated with liquid bridges.

![Fig. 2.4: Theoretical tensile strength of agglomerates with different bonding mechanisms [4]](image-url)
Fig. 2.4 above can be used to identify the different types of bond strengths for a given particle diameter. The bond strengths, as described by the measure of the tensile strength, can be seen to increase with the amount of water present between particles. Therefore, as described previously, for an assembly of powder particles under dry conditions, van der Waals forces dominate. Upon slight adsorption by the particles, the force of attraction increases as shown in Fig. 2.4. If the relative humidity is increased further causing the formation of liquid bridges, the force of attraction would be expected to increase even further. In some cases dissolution of some parts of the particles may occur within the liquid bridges. These may form a solid bridge between the original particles if the relative humidity is later reduced and drying occurs.

In addition, for many powders, it is proposed by Teunou et al. [21] that there exists a value of relative humidity at a given temperature above which the powder will cake, and this is very detrimental to powder flowability. This value of relative humidity is called the critical relative humidity, also called the caking point [22]. Another definition of the critical humidity is given by Murtopaa et al. [24], i.e. the humidity, above which the material absorbs excess water so that it eventually dissolves. This definition would only apply to deliquescent materials. Earlier studies by Harnby et al. [25], describe the critical humidity as the relative humidity where liquid bridge bonds are initiated, causing a marked increase in powder structural strength or cohesivity. Work performed by Harnby et al. [25] with other powder types suggests that this critical humidity can occur anywhere within the range of 50% to in excess of 90%RH. Any process crossing the boundary is likely to have control problems. There is clearly much ambiguity surrounding the term critical humidity, and caution should be exercised when encountering it.

When considering single particle interactions, the role of particle surface roughness must be considered. Irregular and rough surfaces result in multiple contact points between the protruding asperities. This has a number of consequences; firstly the effective contact area is reduced compared to the idealised case of two perfectly smooth and spherical particles. Secondly, any adsorbed water will fill the valleys surrounding the asperities. If
the contacting asperities are sufficiently prominent the formation of a single, large capillary bridge between the particles is unlikely until enough water is adsorbed to totally engulf the asperities.

A further consequence of surface roughness is identified by Orband and Geldart [2], and Coelho and Harnby [22] who report that rougher particles result in higher friction and interlocking forces. This translates into an increase in the cohesivity of the powder. However, the arguments based on area available for capillary condensation suggests otherwise, i.e. the smoother powder particles tend to form stronger bonds and consequently become more cohesive due to the strength of liquid bridges.

2.2.2.2 Influence of relative humidity on powders

As the relative humidity of the surrounding air is increased, powders tend to adsorb water which may form liquid bridges between powder particles and result in greater powder cohesion and reduced flowability. As the relative humidity decreases, powders tend to desorb water, and liquid bridges will evaporate for insoluble materials, such as glass beads. However, according to Teunou and Vasseur, [13] most food powders are soluble materials, and solid bridges may remain, causing the powder to cake. Adsorption of water from the atmosphere by a mass of powder is time dependent because water must diffuse from air into the powder bulk. Furthermore there may well be diffusion of water within the particles, or specific surface reactions with water that will combine to influence the overall water sorption kinetics. Eventually, a moisture content will be attained that provides a dynamic equilibrium between water in the powder and water in air. The rate at which this equilibrium occurs is dependent on temperature and the particle chemical and physical properties.

Teunou et al. [21] studied the flowability of four food powders (flour, skim-milk, tea and whey-permeate). Each powder was characterised by particle size analysis, bulk and particle densities, water sorption isotherms and differential scanning calorimetry (DSC). (DSC thermograms of a powder are reported [21] to provide an insight to the phase
structure of powders. It shows crystallisation and glass transition peaks indicative of the
presence of amorphous structures in the material. The flowability of the four food
powders was compared and discussed with reference to their physical properties and the
relative humidity of the surrounding atmosphere. The tea powder was the most
hygroscopic because it took up the most water with increasing relative humidity and
because it caked at a low relative humidity (Critical Relative Humidity – CRH = 33%).
The flour was much less hygroscopic than tea because its water uptake was less sensitive
to changes in relative humidity and it caked at a higher relative humidity (CRH = 66%).

According to Teunou et al. [21], the critical relative humidity is defined as the minimum
relative humidity at a given temperature at which the powder cakes. This was determined
by shaking the sample on a dish that is in equilibrium with the surrounding relative
humidity and observing any stickiness or clumping of the particles. The relative
humidity at the point where the particles stick together is described as the critical relative
humidity for that sample.

Following on, the skim milk and whey permeate powders appeared to be not particularly
hygroscopic powders based on their respective isotherms. However, they caked at low
relative humidities (CRH for skim milk = 44% and CRH for whey-permeate = 33%)
which suggested that the powders were hygroscopic. The DSC thermogram for flour
showed a decomposition peak at 190°C. On the other hand, the whey-permeate
thermogram had glass transition and crystallisation peaks, which indicated the existence
of lactose in its amorphous state. Lactose in its amorphous state is very hygroscopic and
readily adsorbs water on to the powder surface. It was thought that the water caused
dissolution of lactose and formed liquid bridges between powder particles. The lactose in
solution then crystallised and formed solid bridges between the particles with some of the
free water vapourising back into the air. The formation of the solid bridges between the
particles caused the caking which was observed. It should be noted that amorphous
lactose adsorbs a significant amount of water but much of this water will be released to
the air as crystallisation occurs, so the sorption isotherm does not show a significant
uptake of water. This sort of behaviour was also observed for the skimmed milk.
According to Teunou et al., [21] characterisation of food powder flowability is useful because it quantifies the strength of the powder that has to be overcome to cause the powder particles to move over each other and flow. Following on from their study [21], it was reported that food powders are complex due to their large distribution in particle size and the presence of solid-liquid-gas phases in the particles. Among these phases, it was reported that water has a great influence on flowability and its percentage in the powder depends on the relative humidity of the surrounding atmosphere.

The flow properties of the powders were characterised using an annular shear cell at 20 °C and 25 %RH for this study by Teunou et al., [20]. The flowability of the powders was evaluated by the slope of the flow function. The skim milk was found to be free flowing owing to its low water content and large particle size. The whey powder was reported to be easy flowing though its flow index is less than the skimmed milk. This was reported to be due to the smaller size particles of the whey powder. The flour was found to be cohesive with difficult flow as a result of high water content. The tea powder exhibited moderate flow as a result of having small particles and low water content.

Further experiments were carried out to evaluate the effect of relative humidity on the flowability of flour and whey-permeate powders. The experiments were carried out at 33 and 66 %RH. The unconsolidated powders were placed in the chamber for 15 hours before shear testing, to allow equilibrium to be reached.

In general, the unconfined yield strength (UYS) increased with increasing relative humidity, resulting in a decrease in powder flowability. The presence of water was reported to lead to the possible caking of powders at higher relative humidities. Both powders caked at 66 %RH. The flour formed a relatively weak cake resulting in a slight change to the flow function. On the other hand the cake formed by the whey-permeate powder at the higher humidity was very strong and resulted in a great increase in flow function.
Chapter 2: Literature Survey

It is important to note that Teunou et al. [21] do not compare the slopes of the flow function (or flow index) for these materials at different humidities. This is used conventionally as a classification of cohesivity from the flow function graphs.

In earlier work, Teunou and Vasseur [13] investigated the effect of water content on the flow function of an unnamed moist soluble material. Powder flow characterisation was performed using a shear cell based on the design by Jenike, as used by Akers [14], and following the method proposed by the IChemE [12]. The Jenike shear cell was calibrated using limestone by analysing the CRM 116 limestone powder as used by Akers [14].

Four groups of powders at 0.03% (dry product), 3%, 10% and 15% water content were used for this test. A plot of the UYS against the major consolidating stress revealed a general trend of a decrease in flowability with increase in water content. However, the powder at 15% water content appeared to be more free flowing than at 10%, but more cohesive than the other powders. Teunou and Vasseur [13] drew parallels to a similar observation by Walker [11] with coal. However, it was added [13] that the “anomaly” in the trend was not evidence to suggest that above 10% water content the material flows more easily.

In an attempt to explain the observations, the four states of water content for an assembly of spherical particles were described. At 0.03% water content, the force of attraction is reported to be mainly van der Waals, which according to Rhodes [26] (see also Fig. 2.4) has a low tensile strength for agglomerates.

At 3% the powder is reported to be in a pendular state (Fig. 2.5 below). The unconfined yield strength is reported to increase owing to the pendular adhesion of the liquid film at the surface of the particles. According to the chart by Rhodes [26] (Fig. 2.4) this would correspond to an increase in tensile strength owing to the presence of liquid layers.
At 10% the powder is described [13] to be in a funicular or capillary state (Fig. 2.6). Fig. 2.6 highlights an example of water gathering at the contact points between two contacting particles indicated by the red rings rather than just on the surface. This suggests possible liquid bridges caused by increased water content. The onset of liquid bridges would suggest an increase in the tensile strength [26] (see also Fig 2.4).

In addition, Teunou and Vasseur [13] reported on the possible contribution caused by powder plasticisation to the observed decreased flowability.
At 15% water content, Teunou and Vasseur [13] suggest that the powder may be in a drop (Fig. 2.8) or a capillary state (Fig. 2.7) and the adhesion force due to liquid bridges is reduced due to the increasing distance between the particles. Furthermore, it is suggested that the water acts as a lubricant favouring the sliding of particles over each other.
A short test showed [13] that at high compression loads, the lubricating effect observed at 15% water content would be observed for all the water contents tested thereby suggesting that flowability is not strongly influenced by moisture levels. There are two reasons proposed for this observation. Firstly, at high stresses (> 10 kPa) adhesion forces due to liquid bridges have been reported to be very low (negligible) [13], perhaps owing to the relatively larger distance between the actual particles. It is important to point out that this research does not share this view. However, it will be shown in this document that compression loads have a significant contribution to flowability than that which could possibly be contributed by a change in moisture level from 0.03% to 15%. Therefore at the same high compression loads it is not surprising that the flowability was observed to be similar.

Secondly, the excess water is suggested to be rejected in the shear cell through shear splits when twisting during preconsolidation thereby reducing the final water content to a similar value for the powders.
2.2.3 Influence of powder physical properties on cohesion

Powders or granular materials can be described by a wide range of different physical properties. These physical properties must be investigated in order to establish a link with powder cohesion. Physical properties are measured because they help to define the powder and because they can provide an understanding of the flow behaviour of the powder. Some of these properties include particle size, bulk and particle densities, water sorption characteristics, and chemical stability [22].

In addition to the mean particle size and the particle density, Orband and Geldart [2] report that powder cohesion depends on a multiplicity of other factors such as:

- the width of the particle size distribution,
- the particle shape,
- the surface texture and composition.

It is of interest that Orband and Geldart [2] mentioned the surface properties of the individual particles, as we would expect some possible change to the texture of a material upon ‘wetting’ by adsorbed moisture. The influence of the surface texture and composition is not reported explicitly by Teunou et al., [21]. However they argue that a DSC thermogram gives an insight into the structure of the powder particles such as detecting the presence of amorphous regions in a predominantly crystalline material.

Particle size has been said [2] to be one of the most important physical properties that affects the flowability of powders. It is generally considered that powders with particle sizes larger than 200 μm are free flowing, while finer powders are subject to cohesion and their flowability is more difficult due to increased effect of interparticle forces. With respect to adsorbed moisture, the surface area per unit mass of bulk is a critical parameter [21]. Powders with smaller particle diameter have a larger surface area per unit mass for adsorbing water from the atmosphere. Therefore the flowability of fine powders will be expected to be more affected by the effects of relative humidity change.
Unless a particle surface is perfectly smooth, there is a minimum value of the humidity below which only occasional moisture bonding will occur, with a negligible macroscopic effect on the powder behaviour, [23]. The work carried out by Coelho and Harnby [22] found that surface roughness strongly affected bonding due to adsorbed layers instead of particle size. It is worth mentioning that the background of their work relates to "moisture bonding in powders" on a microscopic scale. However, it is possible to relate moisture bonding in powders to food powder flowability as reported by Teunou et al., [21]. The strength of bonding ultimately determines the flowability.

An accurate determination of the magnitude of these so-called single-particle characteristics is rarely a simple matter, and involves sophisticated and expensive laboratory instrumentation. Furthermore, this knowledge will generally not be sufficient to predict the overall powder behaviour [2]. To gain a more complete insight into the nature of flow problems it is important to establish the link between single-particle characteristics and the properties of the powder as a bulk mass, i.e. as an assembly of particles interacting in complex ways with each other.

The bulk density of powders can give some indication of the flow characteristics of the powder. Coelho and Harnby [22] found that the ratio of the aerated to the tapped bulk density of a powder provided a very good guide to the likely fluidisation characteristics of the powder. The bulk density of powders depends on the filling and handling process, however, relative changes in bulk density can be very sensitive indicators of changes in the structural strength of a loosely compacted powder and hence of its flow characteristics in many process operations [26]. A powder with a strong structural strength will resist collapse when dispersed in a container and will have a low bulk density whilst a structurally weak powder will collapse easily and have a high bulk density. The structure of a cohesive powder will collapse significantly on tapping whilst the weak or free flowing powder has little scope for further consolidation.

The change in the bulk density measured as the ratio between the tapped and the aerated bulk density is sometimes known as the Hausner ratio. It is an indication of the
incremental, volumetric, or structural collapse induced by subjecting the powder to two standardised and increasing external forces and has been found to be a sensitive guide to powder cohesivity.

Kohler and Schubert [27] studied the influence of the particle size distribution on the flow behaviour of fine powders. They found for narrow particle sizes distributions, there was a pronounced decrease in the bulk density with decreasing particle size and that the bulk density was strongly influenced by the width of the distribution. For a given external stress, with decreasing particle size, the stress per contact diminished. This becomes apparent in a reduction of the area and the strength of the individual contacts. However, the increasing number of particle contacts per unit volume due to decreasing particle size may compensate for this tendency. In conclusion, Kohler and Schubert [27] pointed out that in actual powders the random orientations and geometries at the particle contacts and their adsorption layers make feasible only rough calculations of the parameters of interest.

Sutton [3] gave a full account of the influence of the aforementioned physical properties bar the bulk density. He reported that any attempt to sum the microscopic contribution made by the particles themselves to the flow properties of the powder must take into account three factors:

1. The inter-particulate forces.
2. The three dimensional shape of the particle.
3. The way in which the particles, via their surfaces, interact geometrically to form a packed structure (interlocking).

All the above phenomena are associated with the nature of the particle surface which highlights the importance of the surface characteristics of the particles. Most powders are not easily characterised in terms of these microscopic quantities.

Different methods of particle production can give particles of different roughness, leading to changes in the inter-locking or friction forces between particles. Sutton [3] mentions
the importance of the packing arrangement of the particles. There are a number of three-
dimensional packing structures open to the particles. Particle size and shape, and inter-
particle attractive forces both modify the situation, so the packing structure of fine, 
irregular particles is extremely complex geometrically.

Sutton [3] concluded that the best one could hope for at that time were meaningful 
theoretical results based on rudimentary models on the one hand, and a thorough 
experimental investigation of the phenomena on the other.
2.3 UNDERSTANDING STICK-SLIP BEHAVIOUR IN POWDER FLOW

2.3.1 Introduction

Initial shear cell tests in this present study revealed stick-slip behaviour for two materials, namely starch and maltodextrin. With the observation of stick-slip behaviour and in some cases its dependency on sample condition (i.e. apparent humidity dependence), it was decided to probe further in order to understand the origins of stick-slip, to identify governing mechanisms, and the parameters which influence such mechanisms.

2.3.2 What is stick-slip?

In a general sense, stick-slip is the phenomenon of intermittent motion between two contacting surfaces. Displacement therefore occurs as a series of regular jerks rather than continuous motion.

It is important to distinguish between the relative motion of continuous planes, and motion between sections of a granular solid. When considering two planes moving relative to each other, it is likely that tribological effects dominate assuming the surfaces are rigid. This will involve accounting for frictional forces occurring at the interface. In the case of two sliding planes, attention is paid to the nature of the surfaces, i.e. the presence and size of asperities etc. The case of shearing bulk powder is more complex, as no single contact plane is clearly defined due to the discrete nature of powder grains. The behaviour is likely to be dominated by bulk powder properties such as the particle size distribution and shape distribution, as well as single particle mechanical properties, roughness, and interparticle forces.

2.3.2.1 Static and kinematic friction:
van de Velde et al. [28] in their study of friction force during stick-slip describe this effect
to be caused by a velocity-dependent friction force. When two surfaces in contact move relative to each other, a force acts immediately so as to oppose the movement. Prior to any displacement the immediate force opposing motion is called the static friction force \( F_s \). In order to overcome this resistive force, the push/pulling force must exceed \( F_s \). Once the force is greater than \( F_s \) the surfaces will move relative to each other. If the force is constant, the surface will move at a constant velocity against a lower frictional force called the kinematic frictional force \( F_k \).

The static frictional force represents the "stick" phase in stick-slip followed by a rapid "slip". In some cases this duo-action is repeated continuously, giving rise to "stick-slip" behaviour. A classic example of the relative movement of two surfaces is that of a block sliding on a plane [28], [29]. In the work of van de Velde et al. [28], a box of mass \( m \) is attached by a linear spring with stiffness \( k \) to a frame. A plate is set beneath the mass and allowed to slide. The sticking phase occurs until the spring force equals the static friction force of the surfaces. This is followed by an acceleration of the mass (slip) so as to contract the spring with decreasing velocity until the spring force equals the kinematic friction force of the surfaces. Another example given by Persson [29] considers the block on a slope. The force component of gravity acting down the slope must exceed the static friction of the interface in order to induce movement. The jerky movement down the slope is indicative of the stick-slip motion. It is important to note that Persson’s work centred on stick-slip motion induced by shear melting of a lubricated surface, however the principles of friction of a sliding block can be applied.

2.3.2.2 Particle–particle configuration:
Consider a two-dimensional powder bed with rigid, smooth and mono-sized particles arranged evenly as shown in Fig. 2.9. A simple model can be used to describe the relative movement of the top row moving over a static bottom row. Assuming no deformation or rearrangement occurs upon shearing, two steps occur in order to observe movement. Particles in the top row will require a force in order to "climb" (Fig.2.9a) adjacent particles in the bottom row (stick). Once perfectly aligned in a face-centred lattice
arrangement (Fig 2.9b), the particles then slide (slip) freely into a new arrangement (Fig. 2.9c). The shear force required when lifting the top row is larger than the shear force required during the "drop" of the top row into the new position. The climbing and dropping of the top row is repeated if movement of the top row relative to the bottom row is to continue. This intermittent "climb" followed by "drop" movement is described as stick-slip.

![Fig. 2.9: Schematic of the rearrangement of solid spherical particles during shear.](image)

A similar example can be visualised with poly-sized rough particles, as follows. According to Ross et al. [30], the macroscopic value of friction force obtained experimentally is an average of individual forces at asperity-protrusion contact points. A plethora of friction forces, compared to an average friction force, would develop along the interface depending on the asperity-protrusion size. Shear movement would be sustained by a combination of overcoming high friction forces (stick), and the average friction force (slip). Conceptually, this analogy is grossly over simplistic. It will be shown later in this review that asperities (roughness) can not solely determine whether stick-slip occurs.

The two-dimensional model described above accounts for fluctuations in shear cell data with large particles. For small particles the fluctuations are negligible. These fluctuations are random compared to the regular and periodic fluctuations observed with stick-slip behaviour.
Ross et al. [30] proposed that in many cases the stick-slip frequency is determined by the mechanical characteristics of the bodies or of their supports. In their work, they report that predicting macroscopic behaviour such as stick-slip depends upon the assumptions made about the mechanical interactions between individual particles.

All powders will exhibit bulk elastic deformation tendencies to some extent prior to shear failure. Under given conditions, a proportion of the work done on the powder will be absorbed by the particles as stored elastic energy. If the rate of accumulation of elastic energy in the powder-cell force transducer drive system exceeds the rate of friction dissipation of the system, then a peak shear force is reached at which the material suddenly yields and frictional dissipation occurs. This is followed by a re-accumulation of elastic strain. This mechanical instability repeats giving rise to the characteristic stick-slip motion. Tribological effects occurring during this motion are not dominant compared to the effect of elastic energy storage. The association between stick-slip and elastic energy is recognised by Bouissou et al. [31]. The role of elastic material properties in stick-slip will be discussed at length in Section 5.2, as they form the basis of the adopted modelling approach (see Chapter 6).

2.3.2.3 Bulk powder behaviour:

Powder handling includes the filling and emptying of silos. One of the problems faced by manufacturers using tall silos is silo honking which occurs during the emptying of a silo. This phenomenon is characterised by a loud noise (100 dB) every few seconds accompanied by significant vibrations of the silo walls [32]. Tejchman [32] reports that silo honking occurs as a result of resonance between the silo contents and silo structure. If the frequencies of pulsations of a powder during flow coincide with the natural frequency of the silo structure, resonance effects can be induced. Vibrations occur at the surface of the silo walls due to overcoming friction forces as the granules flow out. Powders that exhibit stick-slip are likely to cause a pulsating downward flow notably at the walls of the silo. The flow would be seen to be a fast intermittent action of sticking and slipping. This intermittent stick-slip action is reported to contribute significantly to
vibrations as described by Tejchman [32].

Similarly, another phenomenon observed is that of *silo quaking*. This is a recurring problem in bin and silo loading due to cyclic and pulsating-type flow which arises as a result of density change [33] during flow and by varying degrees of mobilisation of the internal friction and boundary wall friction. It is believed [33] that stick-slip at the boundary relates to the quaking experienced in silos.
2.3.3 Phenomenological analysis of governing parameters.

Stick-slip motion appears to be associated with a combination of tribological effects and mechanical effects pertaining to the material. In their study on the dynamics of stick slip of interfacial friction, Rozman et al. [34] reported that the origin of stick-slip motion and the related phenomena are still unclear and under some debate. Sliding friction is reported to be one of the oldest problems in physics. Many aspects of sliding friction are still not well understood, [29] and an understanding of friction on an atomistic level is just starting to emerge. Bouissou et al. [31] report that the ability of a sample to store elastic energy is a condition required for stick-slip to occur.

In their work to identify the transition from stable sliding to stick-slip, Bouissou et al. [31] showed that normal pressure, slip rate and roughness influence the transition. Ross et al. [30] added that the presence of surface films together with the aforementioned variables tends to affect the type of friction observed. Persson [29] has reported on the origin of stick-slip motion of lubricated surfaces. As part of this study, the motion of a block sliding on a substrate with a molecularly thick lubrication layer has been investigated. Unless the lubricated layer has a low melting point, friction occurs at the interface between the molecules of the lubricant and the moving layer.

The work documented in [31] has been performed on polymethylmethacrylate (PMMA). The samples with dimensions of 100 × 65 × 5 mm³ were cut from the same plate to ensure the same physical properties. The dimensions of the sliding surface were fixed at 15 × 5 mm². Work was carried out for five grades of surface roughness (3-128 RMS μm), five slip rates (0.1-10 mm/min), and six normal pressures across two orders of magnitude (0.5-50 MPa). Each experiment was repeated five times at a temperature of 23°C and a relative humidity of 30 %.

It was found that the transition from stable friction to stick-slip friction is strongly influenced by normal pressure. At low normal pressure (0.5-1.5 MPa) the motion is stable for all slip rates and grades of roughness investigated. At high normal pressures
(25-50 MPa) stick-slip was observed for all slip rates and grades of roughness investigated. In the intermediate normal pressure (5-10 MPa), the slip rate and roughness had more influence on the results. The slip motion was stable at high slip rates for all grades of roughness, and for all slip rates for the roughest surfaces. Stick-slip was observed for other conditions outside this boundary within the intermediate normal load.

Perhaps counter to intuition, the smoother surfaces tended to show stick-slip more than the rough surfaces (at high normal load). A possible explanation for this is the important role adhesion plays [31]. At low normal pressures, only the tops of asperities are in contact [31]. It is argued that the asperities are able to slide over each other without plastic deformation due to slight displacement in the normal direction to the sliding. Only the tops of the higher asperities are broken. The effective contact area is low at low normal load, yielding low adhesion forces. Therefore, the contact strength is very low and the specimen cannot store enough elastic energy to permit stick-slip. Under these loading conditions, the motion would be stable [31]. At high normal pressures the contact strength is high due to an increased asperity contact area and pronounced asperity interlocking [31]. During stick, the high force required to overcome contact strength is stored as elastic energy. A part of this elastic energy is released during the slip phase when the applied shear load equals the contact strength. At intermediate normal pressures, the sliding velocity becomes an important parameter. Adhesion and interlocking are facilitated by slow velocity displacement [31] that enables asperities to fit together by elasto-plastic deformation. Therefore high sliding velocities [31] prevent the asperities interlocking and ultimately reduce adhesion.

For particulate systems, it is argued that the size of the particles can affect stick-slip indirectly. Consider a sample investigated at two size distributions. The overall number of contacts and hence the overall contact area per unit volume is likely to be higher for the smaller sized particles compared to the larger sized particles. This difference will manifest itself as a change in adhesion force, over the slip plane in the material as adhesion force increases with contact area. The argument can be extended to wider size distribution particles.
Chapter 2: Literature Survey

The effect of ambient humidity depends on the water sorption characteristics of the particles. In some cases, as noticed in starch in the present study, the majority of the water diffuses into the starch granule causing particle swelling. A thin film of water may form on the particle surfaces leading capillary bridge formation.

In the study reported by Persson [29] on the origin of the stick-slip motion of lubricated surfaces, the connection between melting temperature of the lubricant and the onset of stick-slip is discussed. Organic lubricants may have a low melting temperature [30], and can also be rate sensitive [29]. In the case of a low melting temperature lubricant such as hexadecane, Persson [30] observed stable motion at 25 °C but stick-slip occurred at 17 °C. It was deduced that the melting temperature of hexadecane layer was between 17 - 25°C. It was further suggested [30] that in some cases where stick-slip is not observed as a result of particle-particle interaction, stick-slip type of processes will occur within the lubrication layer.
3.1 EXPERIMENTAL MATERIALS

3.1.1 Introduction

Three materials have been selected for study in this project; starch, hydrolysed vegetable protein, and maltodextrin. These products are of industrial interest hence the desire to investigate the influence of adsorbed moisture on the cohesion of these powders. The materials have been selected based on their widespread commercial relevance, and range of properties that are represented by them. An outline of the material manufacturing procedure is presented in Appendix A.

Results from characterization studies will be presented, including sample images using scanning electron microscopy (SEM) i.e. shape, size and morphology. Results from water sorption studies are addressed, supported by observations from in-situ humidity microscopy tests.

Flowability studies are to be carried out using a Jenike-type shear cell. A description of the development of the instrument to facilitate flowability tests under various relative humidity conditions and the technique employed is reported.

Lastly, a thorough study was conducted investigating stick-slip failure of starch powder. The method employed and results are reported in Chapter 5.

3.1.2 Description of experimental materials

STARCH

Starch occurs as insoluble granules in many plant tissues, and is widely distributed in nature, cereal grains, peas, beans, roots and tubers being particularly rich sources [35]. Starch from tubers and roots such as potato and cassava (tapioca) are important commercial starches.
For the purpose of this project, potato starch has been selected. The commercial name for this product is “farina”. Farina is produced from starch extracted from potato pulp. The farina is dried, sieved and stored before packing.

Upon initial inspection, it was found that the powder is white in colour. The bulk of powder does not appear to be free flowing. Manufacturers quote maximum moisture content of 12% (w/w). The powder has remained stable with no apparent signs of caking or deterioration with time.

Starch grains can be induced to swell enormously by a process known as gelatinisation, hence farina is used as a thickening agent in cooking. This process occurs by heating in excess water to about 65 °C [36]. According to Flint [35], at room temperature, starch will only swell if damaged. However, in the following section (Section 3.2), evidence is presented that demonstrates swelling of apparently undamaged starch by up to 47% volume when placed in saturated air at ambient temperature. All experiments are to be conducted at room temperature, therefore gelatinisation due to high temperature is not expected to occur.

Water sorption studies have been carried out on samples of the potato starch supplied using a gravimetric analyser1. The water sorption isotherms are shown in Section 3.2.

According to Flint [36] potato starch has a round or ovoid shape. The size ranges for granules are typically; rounded 10 – 35 μm, and ovoid 30 – 100 μm. This is comparable to figures investigated (see Section 3.2) which showed granule sizes of 8 – 25 μm rounded, and 30 – 60 μm ovoid.

**MALTODEXTRIN**

Maltodextrins are partially hydrolysed maize (corn) starch products [37]. Starch hydrolysis products are commonly characterised by their degree of hydrolysis expressed as dextrose equivalent (DE), [36] which is the percentage of reducing

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1 Hiden IGAsorp
sugars calculated as dextrose on a dry-weight basis. A range of commercial maltodextrins has become available for use in a wide range of applications. Maltodextrin is used in foods for example as a carrier or bulk agent, texture provider, spray-drying aid, fat replacer, film former, and freeze-control agent to prevent crystallisation. It can also be used as a convenient source of energy in food or drinks. In these applications [36], various physical and biological properties are required.

The powder is white in colour, and it is free flowing. The manufacturers quote maximum moisture content of 6% (w/w). The powder has remained stable during storage with no apparent signs of caking or deterioration.

The maltodextrin constituent in gravy granules as produced by Centura, is added generally as a bulking agent. Mechanically, the addition of maltodextrin aids the dispersion of starch by coating and physically separating the starch granules. This yields huge process operation benefits and increases the quality of the end product.

**HYDROLYSED VEGETABLE PROTEIN (HVP)**

In comparison to the other powders, there is little information about vegetable protein powders. Vegetable proteins are the richest source of protein in higher plants. These include seeds such as peas, beans, nuts, soya and wheat flour. Once hydrolysed, these powders provide a variety of flavours for end products. There are two main types of HVP powders based on the process method - spray dried or vacuum dried. Six samples of HVP powders were received labelled HVP1–6. HVP1–4 are spray dried powders, and HVP5 & 6 are vacuum dried. Those that are vacuum dried have a higher propensity to pick up moisture. For the purpose of the project, HVP1 has been chosen as it is expected to remain stable (i.e. not pick up moisture with time leading to caking), and it is a major constituent in the end product make-up (in terms of weight). Details on the origin of HVP1 are not available from the manufacturers.

HVP1 powder is of clay colour with a distinct odour (as it is a flavour). The powder bulk appears to be highly cohesive. The manufacturers report a maximum moisture
content of 6% (w/w). The powder has remained stable in sealed bags with no apparent signs of caking, discolouration or deterioration.

There is little available in the literature on HVP powders. However, Flint [35] comments that generally protein powders tend to be more elongated and fibrous perhaps as a result of the effect of food processing. The results of SEM tests amongst other characterisation tests are presented in Section 3.2.
3.2 MATERIAL CHARACTERISATION STUDIES.

3.2.1 Introduction

Material characterisation gives an insight into the flowability of the powders. The influence of particle physical properties, and humidity on flowability discussed in Chapter 2 prompts an investigation to these factors.

The material characterisation carried out includes scanning electron microscopy, measurement of water sorption isotherms, and in-situ humidity controlled microscopy.

3.2.2 Scanning Electron Microscopy (SEM).

SEM work was conducted for all three materials (starch, maltodextrin and HVP1). The aim of performing SEM on these materials was to identify the general shapes, size and surface structure of the granules. All samples were sputter-coated with gold prior to examination, to prevent charge accumulation. At high magnification the intensity of electron bombardment was sufficient to cause local damage to the powders. The observations and images for each powder sample are described below with additional SEM images presented in Appendix B.

STARCH

Fig. 3.1 below is an SEM image of the starch grains which shows the typical bimodal size range. The manufacturers gave a size range of 5 – 100 μm which is thought to cover both size groups. From the figure below, it can be seen that the smaller granules are rounded with a size range between 8 – 25 μm. The larger granules appear to be ovoid in shape, as described by Flint [36], see Section 4.1. A larger majority of these granules fall in the size range 30 – 60 μm.
MALTODEXTRIN

Maltodextrin particles are irregular in shape. Large proportions of them are angular, with rough surfaces. There also appears to be many fragmented particles present (Fig. 3.2 below). Manufacturers quote 73% (by mass) of granules are greater than 100 μm, 3% greater than 200 μm, and 1% greater than 300 μm. A close-up scan of a single granule (see Fig. 3.3) reveals detail of the irregular structure particularly for the smaller fragments.
Fig. 3.2: SEM image of maltodextrin powder showing the different types of granules

Fig 3.3: SEM image of a single maltodextrin granule
HVP

Fig. 3.4 below is an SEM image which shows a clustering of irregularly shaped HVP particles. There is evidence that the majority of the particles were originally spherical. However a large number of them are fragmented. It is interesting to note that some of the granules appear to be hollow (see Fig. 3.5). It proved difficult to separate the granules on to the SEM stub, suggesting that the material is very cohesive. SEM results reveal a particle size between 20 μm and 100 μm. There are some fragments of the order of 10 μm in size. Conversely there are agglomerated granules of the order of 200 μm in size. Size information from the manufacturers only mentions that all granules should be less than 2000 μm in size. From the SEM work carried out, there is no evidence to suggest that the granules are likely to be as large as stated by the manufacturers.

![SEM image of HVP powder](image)

Fig. 3.4: SEM image of HVP powder
3.2.3 Water Sorption Studies

Water sorption studies were conducted using a Hiden IGASorp (Hiden Isochema, Warrington, England). The IGASorp is a gravimetric analyser that can be used to detect mass change of a given sample enclosed in a chamber with temperature and humidity control. The mass of the powder changes as a result of moisture sorption or desorption.

Information gathered from this study gives an idea of the powder’s response to various relative humidities as proposed for shear cell studies. For example, a powder showing a high propensity for absorbing moisture may exhibit significant changes in flowability over a range of relative humidities as discussed in Chapter 2. In addition,
the time taken for adsorption (or/and desorption) to equilibrate is used to determine the minimum time required for the powder to remain within the shear cell with every change in relative humidity.

The samples are initially dried within the instrument by subjecting the sample to zero relative humidity for a given time period. Adsorption is then allowed to occur by increasing step-wise the relative humidity within the chamber. After adsorption is complete, the process is reversed causing desorption. The mass of sorbed water at equilibrium is recorded for a given ambient relative humidity, for both the adsorption and desorption processes. At the end of each cycle, it is possible to detect the possibility of bound moisture held by the powder.

The shapes and features of the isotherm provide information of the powder’s affinity for water. For the purpose of this study, each test for a given sample was carried out at a specified temperature of 20°C.

Procedure:
In order to establish a suitable drying time samples were left drying for 24 hours. It was found that the majority of drying was complete after 10 hours. This value was therefore used for all subsequent experiments.

Immediately after drying, the powders underwent adsorption followed by desorption. The air was set to increase from 0%RH to 90%RH, with 10%RH increments, and returned to 0%RH in similar order. Using the data collected, the % mass change was plotted against %RH for the adsorption and desorption steps.

The mass of the powder lost during drying indicated the water content of the powder in its equilibrium state (as-received). The percentage mass gained by the powder during adsorption determines the sample’s affinity for water. The desorption phase indicates the extent of irreversibility. In some cases the adsorbed moisture can be held as bound moisture as previously mentioned. This is reflected by a larger mass at the end of the run compared to the original powder mass. In extreme cases the powder may not return to its original form. For example, at the end of the
experiment, a “free-flowing” powder could return in the form of a cake, or a paste-like sample.

Below is an account of the water sorption characteristics of the powders together with the corresponding graphs of the sorption isotherms:

**STARCH**

The adsorption of water (Fig. 3.6) is steady and nearly linear. The overall water uptake at 90%RH was around 22.5%. Note that significant hysteresis is apparent, with the sample retaining nearly 5% of its water uptake on drying. This is thought to be a consequence of the comparative slow kinetics for diffusion of the water out of the starch grains.

![Starch isotherm at 20 °C](image)

*Fig. 3.6: Water sorption isotherm for starch powder*
MALTODEXTREN

The water sorption isotherm for maltodextrin powder is shown in Fig 3.7 below. There is a steady water uptake from dry conditions up to around 50 %RH, causing over 5.5 % mass increase. Following this, the amount of water adsorbed increases at a higher but steady rate until the completion of the adsorption phase. The isotherm shows a total of approximately 19 % mass change.

From the isotherms in Fig. 3.7, significant hysteresis is observed, with the desorption isotherm significantly above the adsorption isotherm. In addition, a 4 % mass increase is noticed at the end of the desorption phase which suggests bound moisture trapped by the powder. It is interesting to note that the desorption isotherm follows a very different trend from the adsorption isotherm. The large offset upon returning to 0% RH suggests that a substantial amount of water becomes locked in the sample, perhaps due to the collapse of the amorphous structure.

![Maltodextrin isotherm @ 20 C](image)

*Fig. 3.7: Water sorption isotherm for maltodextrin powder*
Chapter 3: Materials and Methods

HVP

In comparison to the sorption isotherm of the other powders, Fig. 3.8 shows a large water uptake for the HVP powders. The isotherm shows the powders increases in mass by up to 115 % RH due to adsorption of water. The water uptake increases with humidity in a non-linear trend, showing a dramatic increase in uptake after approximately 60 %RH.

It is suspected that the amount of water adsorbed may exceed that recorded as shown in Fig. 3.8 if the powder was left to remain in this environment for a longer time period, thus highlighting its high propensity for water. This is supported by the apparent small mass loss upon desorption between 90 – 80 %RH. It is likely that the point taken at 90 %RH did not fully reach equilibrium. It is suspected that the powder either does not immediately desorb the water, or the powder continues to absorb more water after the data point has been recorded by the instrument.

The desorption of HVP powders follows a similar trend from 60%RH to 0%RH. The isotherm shows a permanent increase in mass of approximately 5% which is thought to be bound moisture held by the powder, again due to restructuring and fusing of the amorphous particles.
3.2.4 **In-Situ Humidity Microscopy Test.**

This test has been developed to observe any changes that may occur when powders undergo humidification or drying on the microscope stage, at room temperature. Some powders like starch have been reported to gelatinise well above room temperature and humidity [36]. However, the reaction to a high humidity environment for starch powders at ambient temperature is of direct interest here. There was no information to predict the outcome for HVP1 and maltodextrin powders.

These tests can provide vital evidence in linking single particle properties to bulk powder behaviour. For example, a sample that swells significantly under an increase in ambient humidity may become "mushy" and gel-like within the shear cell causing high cohesion. This might provide an upper %RH ceiling for tests performed in the shear cell for that particular powder. Conversely a material that shrinks at low humidity indicates a change in the powder physical properties (which may also influence flowability). Loss of natural moisture from a powder may weaken the particle structure causing mechanical degradation. Such powders may be more susceptible to breakage under stress rather than deformation to accommodate added stresses.

**Procedure:**

In-situ microscopy tests were carried out using an optical microscope with a standard 35-mm camera attached to it, and a petri dish modified to provide two separate compartments. The petri dish is divided by a glass slide. In one compartment air at a specified humidity is prepared for drying or humidification processes. This can be achieved using a range of salt solutions, or, distilled water for saturation humidity, and silica gel for dry conditions. In the other compartment, the powder granules are carefully spaced out on a suspended slide in the focal plane of the microscope lens. The petri dish is sealed, and the sample particles are viewed through a window of optical quality glass.
Chapter 3: Materials and Methods

The idea is to carry out the whole experiment focusing on the same granule or set of granules of interest for both the drying and humidification stages. At regular intervals spaced over a maximum of 24 hours, a series of photographs is taken. For each powder sample the test begins with a drying stage followed by humidification. Drying is performed before humidification because for some samples, irreversible changes occur at high %RH. The characteristics of the material in its dry state would therefore not be seen.

The experiments were conducted in this order; starch, maltodextrin and HVP. With the exception of starch, the powder samples were initially monitored every half-hour for the first two hours, then hourly depending on the observations. This pattern was repeated for both stages of the test. The observations from the tests along with key photographs highlighting structural changes are presented.

Below is an account of the reaction of the powders to the test:

**STARCH**

The particles were dried for a total of 24 hrs and there were no apparent changes to the size or form of the starch particles (Fig. 3.9).

The particles were exposed to saturation conditions for a total of 24 hrs. The particles did not appear to show a dramatic size change in comparison to the other powders (see below). However, a simple analysis of the starch particles showed an overall volume increase of ~ 47% when subjecting dry starch to 100% RH conditions. The analysis used the optical microscope to obtain the three principal dimensions of the particle, and assumed an ellipsoidal shape to determine the volume. The difference in size is difficult to detect with the naked eye as can be seen comparing Fig. 3.9 and Fig. 3.10. The observed swelling suggests that water is diffusing into the starch grains.
Fig. 3.9: In-situ microscopy images of dried starch.

Fig. 3.10: In-situ microscopy image showing starch under saturation conditions after 24 hrs.

MALTODEXTRIN

The particles were dried over a total of 24 hrs. During this time period the particles did not show any change in size or form as shown in Fig. 3.11a&amp;b.
Fig. 3.11a: Optical micrograph of maltodextrin particles under dry conditions, ($t = 0$ hours).

Fig. 3.11b: Optical micrograph of maltodextrin particles under dry conditions, ($t = 24$ hours).
The particles responded to saturation conditions by dissolving. Signs of dissolution were noticed after an hour following some swelling. More water was absorbed causing increased dissolution after 2 hours of exposure. After this point the amount of water causing dissolution does not appear to increase significantly. The sequence of responses as described above is illustrated in Fig. 3.12 below.

**Fig. 3.12a:** Optical micrograph of maltodextrin particles under saturation conditions, \((t = 0 \text{ hours})\).

**Fig. 3.12b:** In-situ microscopy images showing the rapid swelling response of maltodextrin granules after 1 hr. exposure to saturation humidity.
Chapter 3: Materials and Methods

Fig. 3.12c: In-situ microscopy images showing the changes to the maltodextrin granules following 2 hrs. exposure. No further changes were observed after this time.

HVP

The particles did not show any apparent changes during drying. The images shown in Fig. 3.13a,b show the particle at the start and end of the drying experiment. However, there was a greater response upon exposure to saturation as illustrated in Fig. 3.14 overleaf.
The particles responded rapidly to the saturation condition by swelling and dissolving within half an hour of exposure (see Fig. 3.14 below). Dissolution continued after an hour owing to the deliquescent nature of the powder. The experiment was stopped after 20 hours.

Fig 3.14a) Optical micrograph of HVP powder under saturation conditions, \((t = 0 \text{ hours})\).
Fig. 3.14b: In-situ humidity microscopy images showing the rapid response of an HVP particle when exposed to saturated conditions
3.3 SHEAR CELL STUDIES – PART I

3.3.1 Introduction

The principal experimental study is the measurement of bulk cohesion and the analysis of stick-slip shear failure. A shear-cell based on the design by Jenike has been used. Whilst this instrument has a number of limitations, as mentioned in Chapter 2, it is still regarded as an industrial standard for characterising powder flow and failure properties. As the project includes an investigation of the influence of adsorbed moisture on powder cohesion, a means to facilitate this effect was needed. In-situ humidity control within the shear cell has been introduced. The design details of this feature are discussed in this section.

In order to generate a yield locus using the Jenike shear cell, a rigorous procedure has to be followed. As mentioned in a review of the literature (see Chapter 2), the results obtained can be operator dependent, and irreproducible. Therefore, it was imperative to carry out a study using a test powder to standardise the procedure, and compare the results with published data [13]. Limestone powder was chosen for this purpose. Following a comprehensive standardisation test, shear cell work was carried out on the food powders at ambient conditions, progressing then to variable humidity. This is reported in the second half of the shear cell studies (Section 3.4).

3.3.2 Design, Development and Commissioning of instruments.

The shear cell used is based on the design by Jenike with some modification (see Fig. 3.15 & 3.16 below). The shear cell has a circular base and top ring (5.75 cm in diameter) which form the cell containing the powder sample. The lid covering the powder and the circular base are made of sintered brass to facilitate air percolation. The lid has two screws fitted to facilitate wrench action and lid-lifting. Weights of known masses are
placed on the lid to provide normal stresses (consolidation). The base is driven towards a force-measuring bracket at a steady rate by a motor-driven stem. Upon contact with the bracket the top ring (approx. 1 cm thick) is displaced from the base causing shearing of the powder in the cell. The resistive force opposing the displacement of the top ring is a measure of the shear force mobilised in the powder within the cell.

![Schematic of the shear cell](image)

**Fig. 3.15: Schematic of the shear cell (not drawn to scale)**

![Modified shear cell in assembled form](image)

**Fig. 3.16: The modified shear cell in assembled form (a), and close up of the circular base and top ring with reference to the sintered brass plate.**
In previous studies (unpublished work as part of academic study by the author), a common method for the purpose of humidity change was to house the powder, and the shear cell in a sealed polythene tent. A salt solution creating a specific humidity would be placed within the tent. The powder would be allowed to equilibrate to the conditions in the tent for an arbitrary minimum time of 24 hours. At this point the powder is scooped into the cell in order to commence shear cell studies. All work is carried out within the tent until the end of the study.

Due to the sensitivity of the technique employed, it is imperative to minimise all errors posed by the practical difficulties of working in a tent. The above method leaves uncertainty as to the extent of equilibrium throughout the entire bulk of powder and the respective conditioned ambient relative humidity. The long time taken for the powder to equilibrate, and the use of different salt solutions for every different humidity makes the method very time consuming. A new design was therefore introduced which enables more rapid control of water activity equilibrium, without the inconvenience of using a tent.

### 3.3.2.1 The Design:

The aim of the new design is to develop a shear cell with in-situ humidity control for powder flow property measurement. The objectives are two-fold:

1. Design a workable system to supply a constant flow of air at a required relative humidity -- namely “conditioned air”.
2. Modify a Jenike-type shear cell to allow for direct contact of conditioned air to the powder within the shear cell.

The flow diagram for the RH control system is shown below in Fig. 3.17. The RH control system consists of:

- (i) 5 valves
- (ii) PSA air dryer
- (iii) Dreschl bottle (containing distilled water)
(iv) RH probe
(v) RH chamber.

Compressed air supplied to the laboratory passes through a PSA air dryer to provide dry air at 2 bar. The dry air is controlled by a flow valve (V2) supplying air for the purpose of this study measured between 0.2 to 0.6 bar. For tests under dry conditions valve V4 is shut off and V3 is open completely thereby allowing the dry air to flow through the RH chamber and into the cavity of the shear cell. For saturated conditions the reverse is performed with V3 shut and both V4 and V5 are left fully opened.

All other intermediate humidity conditions rely on careful adjustment of the valve settings to allow for the correct blend of saturated and dry air into the shear cell cavity. In all cases the humidity of the air going into the shear cell cavity is measured by the humidity probe.

Fig 3.17: Process flowsheet of the system design
Chapter 3: Materials and Methods

Mixing chamber:
Conditioned air supplied to the cell is a blend of dry air and saturated air. The desired humidity is achieved by careful adjustment of both air streams. Dry air is available from the utility mains. Saturated air is obtained by passing dry air through a Dreschb bottle filled with distilled water.

Both inlet streams are fed into a chamber. A stainless steel gauze baffle for effective mixing separates the inlet streams and the outlet stream. The humidity of the outlet stream (conditioned air) is monitored by a humidity probe (see Fig. 3.18 a,b) for details. The chamber and components have been constructed from stainless steel to avoid corrosion due to moisture. The chamber is able to withstand pressures well above the operational level.

Fig.3.18a: Photograph of the mixing chamber
3.3.2.2 The Development:

Shear Cell:
The bottom plate of the shear cell comprises a sintered brass plate (see Fig. 3.19). This feature allows for air to flow through the bottom of the cell, and percolate through the powder and out via the sintered brass lid. The air flow rate is restricted to avoid fluidisation of the powder.
Chapter 3: Materials and Methods

Fig. 3.19: Schematic design of the modified shear cell.

A cavity was cut into the base of the cell creating a plenum chamber. Conditioned air enters the cavity via a port connected to the mixing chamber by a plastic pipe.

The Jenike shear cell method involves twisting the top lid whilst avoiding unwanted lateral displacement from the top ring. In practice this is very difficult to achieve. Occasionally, the edge of the top lid might cut into the powder in the cell. Movement of the top ring or powder while twisting would yield incorrect results. In order to restrict the movement of the top ring, a stainless steel collar was manufactured, as it was essential to devise a means to minimise movement of the top ring while twisting.

The mixing chamber was tested independently to produce a blend of air between 0%RH and 100%RH. The initial arrangement was set up with one Dreschl bottle, however, a top ceiling of 84%RH was observed. This prompted the use of two dreschl bottles arranged in series providing saturation humidity.

RHM Technology, UK, supplied the humidity probe used to monitor the relative humidity in the mixing chamber and squirrel data logger. A calibration check was performed using two salt solutions, silica gel and distilled water. These tests were carried out in a sealed desiccator. The results from the calibration are reported below (Table 3.1). The published values of RH are the range of values obtained from Young [38], and three references cited by Young [38]. Young reported that the relative humidity
measured above a salt solution can be considered to be within 2% of the nominal humidity. A graph of published values (using median values) against experimental values has been presented (Fig 3.20). The results suggest a 'workable' agreement between the squirrel and the salt solutions.

<table>
<thead>
<tr>
<th>(Salt) solution</th>
<th>Range of published values for % RH (Young, 1967)</th>
<th>Experimental % RH</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silica Gel</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Potassium Acetate</td>
<td>19 – 22.5</td>
<td>23.5</td>
</tr>
<tr>
<td>Sodium Chloride</td>
<td>75 – 76.5</td>
<td>78.5</td>
</tr>
<tr>
<td>Distilled Water</td>
<td>100</td>
<td>100</td>
</tr>
</tbody>
</table>

Table 3.1: Results from the calibration of the RHM squirrel using salt solutions compared with the range of values reported by Young [38].

Fig. 3.20: Graph showing the calibration results comparing experimental to published values.
3.3.3 Validation of shear cell technique using limestone powder

In the study by Akers [14], five separate shear cell studies were carried out in different laboratories on the same sample of limestone by different research groups. Using exactly the same type and grade of limestone, shear cell studies were conducted as part of the current project, for the purpose of commissioning the instrument and developing the experimental method. The results of the study conducted show satisfactory parity with the published results [14]. This gives confidence in the instruments and technique employed in the current study. The method and results of the study are reported in Chapter 4.

The limestone powder used in the current study was supplied by Prof. Geldart of Bradford University, from the work by Verlinden [8]. For the purpose of a comparison exercise, it was sufficient to rely on available sample information [8], [14] without carrying out a characterization study on the limestone.

3.3.3.1 Equipment

The layout of the instruments used is as described above. The motor driving the stem is set to a shear rate of \(~2.5 \text{ mm/min}\). The recommended speed [12] is approximately \(1 - 3 \text{ mm/min}\). The use of a steady shear rate is a crucial feature in this study. Irregular or sudden jerky movement while shearing will yield inaccurate results. An analogue chart recorder registers the output force on the force transducer bracket during shear.

The shear force transducer was calibrated by tilting the shear cell on its side and placing weights of known masses on the bracket. An initial offset due to gravity acting on the bracket is accounted for by adjusting the dial setting to zero reading before placing the weights. Using a range of weights, the results from the chart recorder could be interpreted as force measurements. The results from the calibration are presented in Appendix C.
3.3.3.2 Experimental procedure

Experiments were conducted in a series of steps. These have been outlined below:

1. Selection of normal stress levels.
2. Sample preparation.
3. Optimisation (of condition to create critical consolidation).
4. Shear testing
5. Humidity change (covered in Section 3.3.4).

Selection of normal stress levels.
The normal stress levels were selected to coincide with the values published by Akers [14]. Akers used the wide range of 3 kPa, 6 kPa, 9 kPa, 12 kPa and 15 kPa. Experiments were limited to normal stress levels under 4.5 kPa. This is because for the majority of powders in process situations, the level of compacting stress rarely exceeds this value with the exception of operations such as tabletting and briquetting. Therefore, for the purpose of a comparative study, the critical normal stress was set at 3 kPa. Interim normal stress levels were set so as to mirror published values as close as possible.

Sample preparation.
The top ring is aligned with the base in an initial offset position (Fig. 3.21). This is aided by the use of the collar. The alignment of the top ring over the base is such that at a given point during shearing, the top ring and base are concentrically aligned before moving towards the other offset position. The base moves relative to a stationary top ring upon contact with the bracket.

Initial Offset Position. Final Offset Position

Fig. 3.21: Initial and final offset position of the shear cell before and after shearing respectively.
**Filling the cell:**
The cell is filled uniformly in small horizontal layers using a spoon or spatula without applying pressure to the surface of the material. The filling procedure should avoid voids throughout the bulk of powder in the cell. The cell is filled to mild overflow and excess material scraped off using a blade or spatula. Similarly, care is taken to avoid exerting pressure on the powder while scraping.

**Preconsolidation:**
The lid is carefully placed on the powder heap to avoid material being ejected from the cell. The preconsolidation load is carefully placed on the lid. The powder is topped up as it was observed that limestone reduces in volume considerably upon consolidation. The preconsolidation load is a factor (n) of the critical consolidation load. Typically, the ratio, n, of the preconsolidation load to critical consolidation load is 1.5 [14].

The majority of the laboratories that participated in the study published by Akers [14] carried out the experiments using n = 1.5. A load corresponding to a preconsolidation normal stress of 4.5 kPa would therefore correspond to a critical consolidation load of 3 kPa.

**Twisting:**
The lid is fitted with two screws that constitute handles. The screws do not protrude through the plate, therefore do not disrupt the smooth powder surface underneath. The lid and preconsolidation load are twisted using the screw handles in a wrench motion. Each twist consists of a 90° clockwise and anti-clockwise rotation at a speed of one twist per second [12]. The number of allowable twists ranges between 5 and 50 going up in 5 twist increments [12].

It is important not to further compress the powder while twisting. Care is also taken not to lift or spill the powder. The use of the collar minimizes movement of the top ring. Practice is required in order to be able to twist effectively.
After twisting, the lid is removed to inspect the surface of the powder. If the filling and twisting is satisfactory, there should be no significant slope to the surface of the consolidated powder. The surface is also expected to be smooth. The preconsolidation load is carefully replaced with the critical consolidation load. The powder is partially sheared in order to evaluate the state of the powder. The chart recorder output provides the shear pattern.

Optimisation

The output of the shear pattern provided by the chart recorder is evaluated by comparison to the chart shown in Fig. 3.22 below:

![Diagram](image_url)

Fig. 3.22: An example of preshear stress-strain curves for under, critical and over-consolidated samples.

The ideal shape to obtain is that of a critically consolidated sample as indicated above. To develop the technique, a larger mass and high number of twists (e.g. 30 twists) were used for preconsolidation. This showed an over-consolidated sample. The number of twists was reduced to 10 and step 2 was repeated in full. The output of the repeated run is evaluated accordingly. If the sample is still over-consolidated then the number of
twists is reduced to 5. Below this number the mass used is reduced and the shear tests repeated until a critically consolidated state is achieved. It is argued based on experience that this operation can be lengthy to achieve the conditions for critical consolidation.

The output from the chart recorder may not be perfectly smooth, but instead may contain various irregularities due to the formation of shear planes [12]. For the purpose of technique development, it is recommended that one performs the preshear thereby obtaining distinctly under-consolidated and over-consolidated samples. From this, an intermediate condition is selected for critical consolidation tending towards the under-consolidated state and not the over-consolidated condition [12].

\* Shear testing

Optimisation helps to determine the operating conditions and the technique required for shear tests. For every preshear yielding critical consolidation, the base is retracted by reversing the direction of the motor thrust. The critical consolidated mass is replaced by a smaller mass. For the purpose of the comparative study using limestone, the lower load was used according to the published study [14]. The base is again driven at the same rate towards the bracket. Upon contact, the force will rise to a maximum and fall rapidly to a constant value. This is illustrated in Fig. 3.23 below:

![Fig. 3.23: An example of a chart recorder output showing the stress-strain curves for preshear and shear stages as observed in a shear cell experiment](image-url)
Chapter 3: Materials and Methods

After each shear phase is completed, the mass of powder in the top ring is carefully poured (scraped) into a container and weighed as it contributes to the normal stress acting on the shear plane. The weighed powder is then returned into the powder bulk, stirred, and ready for preconsolidation.

Each shear phase as shown above is preceded by a preshear phase. For a given preshear and shear condition, the procedure is repeated in order to have 5 reproducible results within a preshear error tolerance of ~ 5 %. It is not uncommon to require a number of preshear attempts in order to obtain 5 acceptable shear results. The average of the measured shear stresses for a given normal stress corresponds to one point on the yield locus. This procedure is repeated for 5 lower shear loads. These points describe the yield locus of the sample at a given critical consolidation (see Fig. 2.4). The experiment can be repeated using a different critical consolidation in order to generate a family of yield loci. This will require following the steps outlined in this section.

The results from the shear cell studies are presented in the following chapter (Chapter 4).

3.3.4 Variation of cohesion with humidity

The aim of this portion of the commissioning work was to improve the technique using the newly developed in-situ humidity controlled shear cell. The result from this study is reported in Chapter 4.

The sample is prepared using the procedure as described previously. Conditioned air is fed to the cell with careful adjustments in order to obtain the operational humidity. Air from the mains is supplied at an adjusted pressure between 0.2 – 0.6 bar. This is split to serve the dry and saturation feed to the humidity chamber. Depending on the adsorption mechanism of the powder given by results from water adsorption studies (as described in Section 3.3), the powder is left to equilibrate for a minimum of 12 hours. At this stage
the powder is said to be conditioned and the procedure outlined above is replicated in
order to generate a yield locus.

There are some vital modifications that have been implemented to the conventional
technique to accommodate this novel method. The air supply is cut during preshear,
twisting and shearing. This is to avoid the influence of air affecting the consolidation of
the powder as the powders can not be exposed to ambient conditions, while stirring,
conditioned air is passed through the sample. This also has the benefit of minimising the
effect of exposure to ambient air.

Extra care is required when conducting these experiments to avoid loss of "conditioned
powder" from the cell. After a number of experimental runs more powder can be added
to "top-up" significant powder loss. Powder used to "top-up" must be laid thinly on the
top surface. This is usually a small amount. The cell is then left to further equilibrate for
approximately an hour or less depending on the amount of "top-up" powder used.

The nature of the set-up would not permit for powder in the top ring to be continuously
weighed in the ambient environment after each run due to prolonged exposure to the
ambient humidity. Therefore, at the end of the study, the mass of powder in the top ring
is weighed. This is taken to be the average mass of powder in the top ring for calculation
purposes.
CHAPTER 4: COHESION MEASUREMENT

4.1 INTRODUCTION

In Chapter 3 the Jenike type shear cell to be used for this study is introduced, together with the modifications to facilitate in-situ humidity conditioning of powder within the shear cell. In addition, the technique to be employed using limestone is described following the steps described by Akers [14].

In order to validate the shear cell to be used, and the experimenter’s technique, the tests carried out by Akers [14] were closely replicated and the results compared. Furthermore, shear cell tests under varied humidity were conducted on limestone powders before similar tests were carried out on the food powders.

The present chapter reports on the findings from the tests described above. A portion of this chapter offers some explanation to some of these observations and results.

In this chapter, the reader is introduced to stick-slip failure as first observed in this study. This served as a platform for further investigations as detailed in Chapters 5&6.

4.2 RESULTS FROM SHEAR CELL STUDIES

4.2.1 Limestone - Validation of technique

Fig. 4.1 below shows all yield loci data produced from experiments performed in the current study, alongside the average values of the tests carried out in the 5 separate studies as reported by Akers [14] labelled Lab A to E. The technique employed has been reported in the previous chapter.
Chapter 4: Cohesion Measurement

Fig. 4.1: Graph comparing the yield loci of experimental data conducted in the current study to published results [14].

The cohesion of a bulk powder as defined in Chapter 2 is the value of shear stress given by the yield locus when the normal stress is extrapolated to zero. It is important to appreciate that the yield locus is not necessarily linear. However, for the purpose of a comparative study, linear regression has proven useful to determine the cohesion.

Note from Fig. 4.1 that there is significant variation in the results from all laboratories. The current shear stress data add to the variation by being slightly higher than the rest in the data set. This highlights the inherent inconsistency in shear cell testing.
Chapter 4: Cohesion Measurement

The yield loci for the limestone powder under 3 different humidities have been plotted below (Fig. 4.2). The trend lines have been fitted by linear regression.

Fig. 4.2: Yield Loci for limestone powder under variable humidity using average values.

Comparisons of the yield loci in Fig 4.2 above show an increase in cohesion with increase in relative humidity on limestone which is reputedly insensitive to adsorbed moisture [8]. Under a relative humidity of 26 %RH the cohesion is approximately 450 N/m². An increase in relative humidity to 47 %RH is followed with an increase in cohesion to approximately 600 N/m². Further increase to the relative humidity (68 %RH) causes the cohesion to increase to approximately 770 N/m².

As mentioned in the introduction to this chapter, this test was performed to validate our capability to condition powders to varied humidity and perform a shear test on them. The proof of this is shown by a change in the flowability of the powder according to the trend described in Chapter 2 (i.e. powder cohesion increases with increased relative humidity).
4.2.2 Starch

Starch powder exhibited "stick-slip" behaviour, as shown in the shear cell trace of Fig. 4.3. This motion observed during shearing is a recurring dual action: Firstly, the shear force increases sharply (stick) until it is large enough to promote slip, or plastic failure of the bulk material. This is accompanied by a sudden reduction in the shear force to a value that cannot sustain the failure and the material is described to then slip. The static material then re-accumulates elastic strain energy and the process begins again. A full account of stick-slip failure is presented in Chapter 5.

Fig. 4.3: An example of the stick-slip failure pattern for starch

Fig. 4.4 shows a graph of the yield loci for starch under all three humidity conditions. The trend lines are drawn using linear regression curve fitting. The stick-slip nature of the shear stress makes it difficult to determine a suitable value for constructing yield loci. It has been decided that the most suitable interpretation would be to take a mid-point value between the maxima and minima of the oscillations.
Though a comparison of the yield loci above shows small changes in the cohesion with change in humidity, it was noted that the powders felt increasingly more difficult to stir with increased humidity during the experiments. This would imply that the shear cell test is not particularly sensitive to the subtle changes in the powder, at least under the range of consolidation loads studied.

A comparative study of the cohesion values for starch obtained in the present study with published values in the literature would be useful in order to validate our results. It must be borne in mind that there are a range of different starches arising from different plant sources, which have differing shapes and sizes. In addition, the material condition (e.g. moisture content), sample preparation (i.e. level of compaction) and experimental conditions (e.g. type of shear cell tester and shear cell velocity) to name a few all contribute largely to the values of cohesion reported.
Cohesion values for various starch and wheat powders are tabulated below together with the critical consolidation stress. An arbitrary ratio of the value of cohesion to critical consolidation is introduced for the purpose of comparison.

Experimental cohesion values have been reported below for ambient conditions. Unless otherwise stated, the cohesion values reported below are for powders under ambient ("as-received") condition i.e. there are no reports of intended changes to the ambient relative humidity or moisture content of the powders.

<table>
<thead>
<tr>
<th>Powder</th>
<th>Cohesion, N/m$^2$ (Approx.)</th>
<th>Critical Consolidation Stress, kN/m$^2$ (Approx.)</th>
<th>Ratio Cohesion / Critical Consolidation Stress (N/m$^2$ / kN/m$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Potato Starch (from the present study)</td>
<td>300</td>
<td>3</td>
<td>100</td>
</tr>
<tr>
<td>Potato Starch [39]</td>
<td>200</td>
<td>N/A</td>
<td>-</td>
</tr>
<tr>
<td>Corn Starch [40]</td>
<td>200</td>
<td>6</td>
<td>33.3</td>
</tr>
<tr>
<td>Starch Fraction in Chickpea flour [41]</td>
<td>7000</td>
<td>80</td>
<td>87.5</td>
</tr>
<tr>
<td>Corn Starch [18]</td>
<td>32 – 600</td>
<td>Expressed in terms of a range of porosities (45 – 58 %)</td>
<td>-</td>
</tr>
<tr>
<td>Wheat Flour [42]</td>
<td>1700</td>
<td>5.6</td>
<td>300</td>
</tr>
<tr>
<td>Wheat Flour [42]</td>
<td>2000</td>
<td>10.6</td>
<td>190</td>
</tr>
</tbody>
</table>
Chapter 4: Cohesion Measurement

| Wheat Flour [42] | 1900 | 15.6 |

Table 4.1: Comparison of experimental values of cohesion with published values.

The difficulty associated with a direct comparison of the values of cohesion has been described above, however despite these objective difficulties it is intended where possible to show comparison of experimental flowability data to within an order of magnitude [43] of published values. Based on this, it is possible to conclude that the values of cohesion are comparable to published values for starch. As previously mentioned, the values of cohesion are not expected to be the same owing to number of parameters as outlined above.

Dilation of the powder bulk was observed during shearing. This was evident by the gradual rise of the top ring from the base of the shear cell. This effect was consistent throughout the various stages of humidity change.

The individual yield loci for different humidity conditions have been included in Appendix D.

4.2.3 Maltodextrin

Shear cell studies could not be carried out using the same critical consolidation (~ 3 kPa) load as used for the starch powders. At this critical consolidation load the corresponding shear stress exceeded the torque available from the motor. This reflects the much more frictional nature of this material due to its angular nature. At this point the motor would cease to drive forward. Progress was made under a critical consolidation load of ~ 1.5 kPa.
Experiments were carried out under dry (0% RH) and ambient relative humidity (25 – 30% RH). Attempts made to carry out a study at 100% RH failed due to caking of the powder. The powders initially formed a paste notably at the base of the chamber before caking for the first set of results. The condition of the powder worsened with time causing the study at this humidity to be aborted.

At 0% RH, maltodextrin exhibited stick-slip behaviour. This was not observed at ambient (25 – 30% RH) and 100% RH. The occurrence of stick-slip under selective humidity conditions sparks an avenue of interest, and is discussed further in Section 4.3.4.

A search of the literature did not reveal any reported values of cohesion for maltodextrin powder. It has therefore not been possible to compare the present data with those of others for this material. The keywords: maltodextrin + cohesion + shear cell used in search engines such as Google and Science Direct do not generate published values of cohesion useful for a comparison with values of cohesion reported in the present study.

As with starch powder there are a number of different grades of maltodextrin adding to the complexity in carrying out a quantitative comparative analysis.

For effective comparison of the cohesion under different conditions, the yield locus under dry conditions was represented using mid point values from the stick-slip oscillations. As with starch, this method takes into account the minima and maxima values giving an average of the shear force experienced. The yield locus under ambient conditions (25-30% RH) was plotted using the standard “peak” values as it did not exhibit stick-slip.

The yield loci (Fig. 4.5) below show that the results for the 0%RH and 25-30% RH coincide. The trend line drawn to the ordinate was constructed using linear regression. The trend lines suggest that there is negligible influence on cohesion with change in humidity. Counter to this finding, the powder bulk was noted to be more “cohesive” while stirring under dry conditions than under ambient conditions. This may be due to
the angular nature of the material which is more pronounced at dry conditions. Under 30% RH it is likely that some rounding of the angularity has occurred making it easier to stir. However when consolidated under a load, it is likely that the interparticle force will increase as a result of the absorbed water.

![Yield loci for maltodextrin powder using mid point values for 0% RH and peak values for ambient conditions, @ 1.5 kPa consolidation](image)

**Fig. 4.5: Yield loci for maltodextrin powders under variable humidity using mid point values for 0% RH data (stick-slip,) and peak values for ambient condition data.**

The powder bed was observed to dilate significantly during shearing causing the top ring to lift gradually irrespective of airflow through the powder bed. This is a consequence of the angular and irregular nature of the particles. More expansion of the assembly is required to accommodate relative movement between individual grains.

Despite employing the standard technique as used for all other shear tests, it was observed that the chart recorder output showing shear stress vs. time (or distance) suggests that maltodextrin powders under dry and ambient humidity conditions are regularly over-consolidated during preshear (see Fig. 4.6). Several failed attempts were
made to achieve critical consolidation before arriving at the conclusion that this could be a feature for maltodextrin powders, and not experimenter technique, as reported by Cleaver et. al. [44] for angular powders, and for food powder as reported by Juliano et al. [45] and van der Kraan et al. [46]. The angular nature of the powders requires extra dilation which requires extra energy.

Fig. 4.6: Evidence of over-consolidation-type response during preshear by maltodextrin powder (0% RH)

4.2.4 **HVP**

The studies for HVP powders were carried out using the same critical consolidation of ~1.5 kPa as used for maltodextrin for the same reason. The yield loci (Fig. 4.7) show that there is a marked increase in cohesion with increase in humidity. The trend lines have been fitted using linear regression. It can be seen that the cohesion increases by over double the original amount from 0 %RH to 25-30% RH.
As with maltodextrin, the keywords: HVP/vegetable protein + cohesion + shear cell were used in search engines such as Google and Science Direct but there were no reported values of cohesion for HVP. The protein fraction from chickenpea flour is reported by Emami and Tabil [41] to have a value of cohesion of 3220 N/m$^2$ when subject to a critical consolidation stress of approximately 80 kN/m$^2$. Applying the arbitrary ratio of value of cohesion (N/m$^2$) to critical consolidation stress (kN/m$^2$) a value of approximately 40 is obtained. This value is appreciably less than the experimental arbitrary ratio of approximately 600 (using a value of cohesion of 900 N/m$^2$ and critical consolidation stress of 1.5 kN/m$^2$ – see Fig. 4.7). The difference is argued to be heightened due to the complexity of protein powders.

![Yield loci for HVP powder under variable humidity (using average values), @ ~1.5 kPa consolidation.](image)

**Fig. 4.7: Yield loci for HVP powder under variable humidity**

The number of data points are less for this study due to the practical difficulties faced with handling HVP powder. Many experimental runs had to be aborted due to increased blockage of the pores of the porous plate after the first ten results.
HVP powder did not display stick-slip behaviour under any given humidity tested unlike starch and maltodextrin. An interesting point to note from the chart recorder output, (shear stress vs. time or distance), is that at 0 %RH, the graphs displays similar signs of being over-consolidated (Fig. 4.8 below) during preshear as observed with maltodextrin. This was not noticed under ambient humidity conditions where the graph followed conventional critical consolidation shape.

Negligible dilation was observed for this study. In general HVP powders posed severe handling problems due to clogging of the porous plate and motor-driver system, and stickiness due to hand moisture.

The powder was not humidified above ambient humidity as handling problems were severe, even at ambient (25-30% RH) relative humidity. The water sorption isotherm for HVP powder shows that HVP has a high affinity for water (Section 3.2).

Fig. 4.8: Evidence of over-consolidation type response during preshear by HVP powder
4.3 DISCUSSION

4.3.1 Introduction

This section is a review of experimental results from shear cell studies as described in the previous section. This section serves two distinct purposes. Firstly, it provides a review of the results obtained from investigating the effect of adsorbed moisture on flowability (cohesion) measurements of the three food powders (where cohesion is adopted as a measurable parameter depicting the extent of flowability). Secondly, this chapter identifies experimental evidence of stick-slip failure exhibited during shear cell studies. Evidence of stick-slip at this stage provides a platform for a more in-depth analysis of stick-slip study in Chapter 5.

4.3.2 Cohesion Measurement

According to the literature, the general trend for most powders, as described in Chapter 2, would indicate an increase in cohesion with increase in relative humidity of the surrounding air. The level of influence can be characterised by the material’s affinity for water, and the subsequent effect this can have on the particle assembly, through the formation of capillary bridges between the particles. Other effects include increased plasticity, collapse of amorphous structure and particle dissolution.

The survey of the literature presented in Chapter 2 has so far not made a distinction between the food and non-food powders. Food powders represent a large variety of powder materials that differ in their chemical composition and physical characteristics [47] which is not generally experienced in non-food powders. Food powders are usually biological in origin and comprise typically a mixture of proteins, carbohydrates, fats, fibre and water [48]. Their complex composition and biological origin make food powders especially difficult to handle predictably. In addition they are often in a meta-stable state, containing a substantial amount of amorphous phase which may undergo transitions as a result of temperature or humidity changes, [49].
Chapter 4: Cohesion Measurement

Food powders can therefore be more of a challenge to the process engineer than more conventional powders.

During flow, the individual particles of non-food powders predominantly slide across each other in a shearing action. However, food powder particles have a high propensity to stick together and form stable structures, preventing flow due to mechanical forces [45]. Mechanical and chemical interactions among powders have been reported [45] to depend on a number of parameters including moisture content, particle shape and size, and the manner in which the particles geometrically interact to form stable structures. The strength therefore among particles can be due to mechanical interlocking, chemical bonding, and van der Waals and electrostatic forces. The forces opposing flow are friction, cohesion and interlocking [45]. It should be noted that many amorphous food powders have glass transition temperatures that are close to ambient. [49] Food powders are therefore at risk of being naturally sticky if the powder temperature rises above the glass transition temperature.

HVP powders show a marked increase in cohesion with increase in humidity. The cohesion under ambient conditions (25 – 30% RH) is over twice that when the material is totally dry. It is suspected that a similar trend would be observed for relative humidities in excess of 30% RH (i.e. ambient humidity). However practical limitations made these tests difficult to conduct.

Counter to intuition, the trend of increasing cohesion with increasing relative humidity has not been adhered to by starch and maltodextrin, according to the shear cell data in Fig.4.4 and 4.5.

However in the case of starch powders, it was noted that during the experiment the powders became progressively difficult to stir with increasing humidity. This qualitative observation supports the notion that cohesion increases with increasing water adsorption, although this is not supported by the shear cell data.

Contrary to this, it was also noted that there was increased difficulty experienced in stirring maltodextrin powders when reducing the relative humidity from ambient (20 –
Chapter 4: Cohesion Measurement

35% RH) to dry conditions. Documented below are possible explanations for the observations recorded from shear studies of all three powder samples.

4.3.3 Starch

The adsorption isotherm for starch powder shows a water uptake of ~23% between dry starch and starch exposed to an ambient humidity of 95% RH (see Chapter 3). This relatively large water uptake would intuitively suggest a detectable change in the cohesion of the starch powder with change in humidity. Results from the in-situ microscopy tests for starch suggest that the mass change was associated with swelling of the grains. Size analysis of single starch particle revealed a volume change of up to ~47%, on increasing the relative humidity from totally dry to totally saturated conditions.

A search of the literature presented in Chapter 2, suggests the influence of moisture on cohesion is largely attributed to the formation of capillary bridges between contacting granules. Water adsorption studies showed a mass increase of ~23% during adsorption. However in this case, it is argued that the bulk of the water is absorbed into the individual granules causing swelling as observed during in-situ humidity microscopy tests (see Fig 3.10). The presence of water on the surface is acknowledged, however the effect of liquid-liquid bonding between the granules as would be suggested by the 23% mass change is argued to be reduced. Therefore, it is suggested that the expected effect of an increase in cohesion with increase in ambient humidity due to surface water is greatly reduced.

Given that the size of the granules changes notably with water sorption, within a finite volume, as provided by the shear cell, a change in granular size causes a change to the overall contact area between granules. See Fig 4.9 below:
Fig. 4.9: Effect of granular size change on contact area/number in a fixed volume (i.e. shear zone)

In the figure above, two schematics have been drawn showing an equal number of granules within a marked square. The marked squares represent the volume of the shear zone within which a shear plane exists. For the purpose of this analysis the volume of the shear zone remains fixed. As the volume of the granules increases with humidity, the number of contacts per unit volume between the granules is reduced. This would suggest that within the shear zone, there is a reduction in the overall contact area as the humidity increases. In the extreme case of a 47% increase in particle volume, the number of particle contacts per unit volume would decrease correspondingly by a factor of 0.67. It has to be borne in mind that the increase in particle size will increase the individual contact area between particles, thereby increasing cohesion. In addition the absorption of water into the particles is likely to modify their mechanical properties making them more ductile. This will promote cohesion. An in-depth analysis of these phenomena is outside the scope of this current study.

In addition, it is thought that the presence of water on the surface between the granules may provide a lubricating effect causing increased “sliding” between granules during shear.
Though not conclusive, it is suggested that the arguments presented could account for the possible “attenuation” of an expected increase in cohesion typically caused by increase in adsorbed moisture.

4.3.4 Maltodextrin

The adsorption isotherm for maltodextrin as shown in Chapter 3 shows a mass increase of ~19% during adsorption. The isotherm shows marked irreversibility during the desorption phase. At the end of the desorption phase a residual mass change of ~4% was recorded arguably due to water being locked into the fused amorphous structure.

Maltodextrin powders have a high affinity for water. In-situ microscopy (Chapter 3) results reveal dissolution of the granules under saturated conditions. There were no changes to the granules when moving from ambient to dry conditions.

An account of the results from shear tests detailed in Section 4.1 shows stable flow under ambient conditions (and saturation conditions), but stick-slip failure under dry conditions. A yield loci plot as illustrated in Fig 4.5 reveals no apparent change in the cohesion. The failure patterns were also observed to show a consistent “over-consolidation” type curve during preshear despite conventional attempts to rectify this pattern as directed [12]. This involved reducing the number of twists and the consolidation normal stress. In addition, the dry powders were observed to be more difficult to stir than the ambient powders.

The over-consolidated failure pattern during preshear is suggested to be as a result of inter-locking due to the angular morphology of the granules. When a shear force is applied to a consolidated maltodextrin powder bulk, (e.g. during preshear), the granules are unable to roll over each other and significant dilation must occur to accommodate shear. This dilation requires that work is done against the weight of the lid. The extra work manifests itself as an augmentation in the recorded shear stress whilst dilation is occurring [44].
A secondary observation point for discussion is the selective nature of maltodextrin powders to exhibit stick-slip. SEM images of the granules shown in Chapter 3 and Appendix B reveal an angular morphology. The experiments conducted under dry conditions used powder samples dried from ambient conditions. The isotherm also suggests bound moisture remains within the bulk of the powder when dried. Under dry conditions, the angular structures of the granules are pronounced. It is suggested that the interlocking effect [3] is enhanced when the granules are dry. The effect of interlocking, as proposed in a review on stick-slip (Chapter 2), is suggested to contribute to the stick-slip mechanism. Adsorbed water is known to act as a plasticiser, and therefore dry samples are likely to be harder and more brittle. This may also contribute to the observed behaviour.

It is possible that moisture present under ambient conditions provides lubrication between granules. This acts to reduce friction between contacting granules. When dry, the lack of a lubrication layer results in an increase in frictional resistance.

4.3.5 HVP

The water sorption isotherm for HVP powder (Chapter 3) can be identified by two distinct regions when comparing the adsorption and desorption phases. At a humidity less than ~ 55 % RH the isotherm appears reversible. Above this humidity, water sorption is clearly irreversible with a distinct hysteresis. At the upper humidity limit of 90 %RH, the powder sample is reported to have an enormous mass change of ~ 115 %. However the material is significantly deliquescent and more water may have been adsorbed if the test had been conducted for longer.

The affinity of these powders for water is reflected in the results of the in-situ microscopy tests. These tests show that within 30 minutes of exposure to a saturated condition, the powders adsorb enough water causing swelling and possible dissolution of the powder granules.
SEM images for HVP (Chapter 3 and Appendix B) reveal a clustering of amorphous granules with a relatively wide size distribution. The powders have a sticky nature when handled, with the tendency to absorb moisture readily from the hands. The handling difficulties associated with spray dried powders such as this sample of HVP have been reported by Downton et al. [50].

The study on HVP was carried out under ambient and dry conditions. A comparison of the yield loci reveals a marked increase in cohesion from dry to ambient. An attempt at saturation conditions was not conducted as in-situ microscopy showed an excessive uptake of water.

It was also noted that the powders exhibited an over-consolidated style shape curve during preshear under dry conditions as observed with maltodextrin. As with maltodextrin, it is possible that this is as a result of interlocking of a consolidated dry bulk powder unable to move freely over each other under an initial shear stress. Once a shear band has been created, the powders are able to move with more “freedom” over each other.

It was noted that HVP powder did not exhibit stick-slip behaviour as displayed by the other two powder samples. There were also no signs of significant dilation during the experiments suggesting no expansion of the bulk powder during shear.
CHAPTER 5: EXPERIMENTAL EVIDENCE FROM STICK-SLIP STUDIES

5.1 INTRODUCTION

Stick-slip behaviour was observed for two materials, namely starch and maltodextrin, during shear cell studies. With the observation of stick-slip behaviour and in some cases its dependency on sample condition as observed with maltodextrin, it was decided to probe further in order to understand the origins of stick-slip, to identify governing mechanisms, and the parameters which influence such mechanisms.

As described in Chapter 3, stick-slip study was conducted using starch powder owing to its widespread industrial use and constant display of stick-slip failure under all conditions during shear cell studies.

From the survey of the literature presented in Chapter 2, the normal stress acting on the shear "plane" and the respective strain rate are among the contributors to the extent of stick-slip. The study was set up to identify the effect of both factors.

Modification to the instrument used for shear cell studies was necessary in order to accommodate the extent of the factors to be investigated. Refer to Chapter 3 for details of the modification and a description of the experimental procedure.

The observation of stick-slip in starch powders during shear has been published in the literature [51] and [46]. In both publications the values of cohesion were not presented. In the study on the effect of porosity on bulk powder, Scarlet et al. [51] report on examples of stick-slip for a starch powder occurring as a possible result of the property of the powder. However they make no attempt to explain or discuss the phenomenon in any depth that will contribute to the current study.
In this chapter a review of the raw data is first presented. The raw data obtained from the shear cell are in the form of chart recorder traces. Data from 46 useful traces covering four normal stresses (open lid i.e. \( \sim 0.075 \text{ kN/m}^2 \), \( \sim 4 \text{ kN/m}^2 \), \( \sim 12.5 \text{ kN/m}^2 \) and \( \sim 28 \text{ kN/m}^2 \)); where each normal stress is evaluated over four strain rates, are reviewed in two parts. Firstly the general trends between the different operating conditions (normal stress) are presented followed by identifying disparities within the same operating condition that may serve to reveal more on stick-slip phenomena.

Manipulation of information from the traces provides useful parameters. These include friction coefficients, shear modulus, a measure of the elasticity of the system and the duration of the separate stick and slip phases. These parameters identify the effect of normal stress and strain rate on stick-slip.

### 5.2 Trace Evaluation Across Different Normal Stress Levels

For a given normal stress level, the powder was tested over 4 motor settings (3 V, 6 V, 12 V and 24 V). The velocity range recorded under each setting for all normal stresses are as follows:

Under:
- 3 V, 0.5 – 1.2 mm/min
- 6 V, 2.3 – 3.4 mm/min
- 12 V, 5.7 – 7.7 mm/min
- 24 V, 13.2 – 23.6 mm/min.

A graph of shear cell velocity as a function of motor setting (V) is presented in Appendix E. The range of velocities per voltage represents the minimum and maximum values for all normal stresses investigated. The velocity is calculated by recording the time taken for a fixed point on the motor to travel alongside a graduated measuring strip.
Chapter 5: Experimental Evidence from Stick-Slip Study

The recommended velocity for the Jenike shear cell is 1-3 mm/min [12]. The velocity range observed envelopes the recommended range, and more importantly exceeds it. This was done in order to observe notable effect to stick-slip across a wide range of velocities, especially at relative extremes of velocities.

The results are presented with a view to providing evidence of the effect of changing the normal stress and shear cell velocity on stick-slip. Where appropriate, descriptions of these parameters are given. Graphical evidence is included in support of these results.

Stick-slip study commenced with an open lid thereby minimising the normal stress on the starch powder i.e. the only normal load on the powder was due to the weight of the powder in the upper ring. The failure pattern appeared to show the least evidence of stick-slip based upon the ‘observed frequency’ of stick-slip occurring over the evaluated period. The observed frequency is based on the number of slips occurring over a given time period.

![Graph](image)

**Fig. 5.1: Observed Frequency as a function of shear cell velocity for all consolidation loads studied**
Fig. 5.1 above may appear to show inconclusive evidence of the effect of shear cell velocity on frequency over the normal stresses studied. At low shear cell velocities (under 3 mm/min) the observed frequencies appear not to be influenced by the level of consolidation. Further increase in consolidation creates two frequencies bands. The lower frequency band occurring under 0.075 kN/m² and 12.5 kN/m², and the upper band for tests performed under 4 kN/m² and 28 kN/m². However, the slip phases resulting from increasing the normal stress (and shear cell velocity for a given normal stress) were observed to occur rapidly causing the instrument to jerk sharply.

As would have been expected, Fig. 5.1 also shows that within a given consolidation load, the frequency increased steadily with an increase to the drive velocity. This is largely due to an increased rate of accumulation of elastic energy beyond the static friction force and the subsequent release as indicated by slip. Within a given time frame this process will be observed to be repeated more often at a higher velocity than at a lower velocity. Individual graphs under each condition are included in Appendix F.

Fig. 5.2 below shows the change in $\tau_{\text{max}}$ with shear cell velocity. $\tau_{\text{max}}$ is the critical shear stress reached and has been plotted on a log scale for an effective comparison. $\tau_{\text{max}}$ is also the shear stress corresponding to the static friction force, $F_s$. 

98
Fig. 5.2 Critical shear stress $\tau_{\text{max}}$ as a function of the shear cell velocity

Generally, the magnitude of the maximum shear stress $\tau_{\text{max}}$ increased with increasing normal stress. Within each normal shear stress band, it is argued that $\tau_{\text{max}}$ decreases with an increase to the shear cell velocity based on evidence provided in Fig 5.3 (a-d) below. Note that the scales on some of these graphs have been adjusted to display the data to best effect. The Corresponding graphs of $F_{\text{max}}$ as a function of velocity is presented in Appendix G.
Chapter 5: Experimental Evidence from Stick-Slip Study

Fig. 5.3a: $\tau_{\text{max}}$ as a function of the shear cell velocity

Fig. 5.3b: $\tau_{\text{max}}$ as a function of the shear cell velocity
\( \tau_{\text{max}} \) as a function of the shear cell velocity

\( \tau_{\text{min}} \) is the shear stress required to maintain "steady" flow which ought to correspond to the kinematic friction value at the end of the slip phase. Counter to intuition, it is
proposed that the true value is not described by the shear stress at the end of each slip (as seen in Chapter 4 where mid-point values were taken as a simple measure to represent steady flow). It is argued that the shear stress reached at the end of the slip phase reflects both the shear stress required to maintain steady flow as well as a measure of stored elastic energy released.

The true value of $\tau_{\min}$ has not been investigated in this research and remains a strong candidate for future research. However for the purpose of calculating the kinematic friction force as defined by Bouissou et al. [31] $\tau_{\min}$ is taken as the shear stress at the end of each slip.

As with $\tau_{\max}$, there is a general increase in $\tau_{\min}$ with normal stress as illustrated by the graph in Fig. 5.4 following a similar pattern with the graph in Fig. 5.2.

![Graph showing the effect of shear cell velocity on $\tau_{\min}$](image)

**Fig. 5.4: $\tau_{\min}$ as a function of shear cell velocity**

A closer examination of the effect of velocity on $\tau_{\min}$ for a given consolidation indicates an increase in $\tau_{\min}$ with velocity. This effect is consistent throughout the velocities tested under 'open lid' and 4 kN/m$^2$ consolidation. Under 12.5 kN/m$^2$
consolidation, $\tau_{\text{min}}$ increases with a steeper gradient at velocities under 1 mm/min followed by a continuous increase with a lower gradient. A similar trend is observed under 28 kN/m$^2$ with an even steeper increase in $\tau_{\text{min}}$ for velocity conditions under 2.5 mm/min. From this point $\tau_{\text{min}}$ appears to reach a maximum asymptotic value for the remainder of the velocities tested (See Fig. 5.5 (a-d)). The graphs showing the corresponding $F_{\text{min}}$ variation with velocity is included in Appendix H.

Fig. 5.5a: $\tau_{\text{min}}$ under 0.075 kN/m$^2$ (open lid) as a function of shear cell velocity
Chapter 5: Experimental Evidence from Stick-Slip Study

Fig. 5.5b: $\tau_{\text{min}}$ under 4 kN/m$^2$ as a function of shear cell velocity

Fig. 5.5c: $\tau_{\text{min}}$ under 12.5 kN/m$^2$ as a function of shear cell velocity
The graph showing the change in amplitude with normal stress and velocity (Fig. 5.6) shows a general trend of a decrease in amplitude with increase in shear cell velocity. Individual graphs under a given consolidation load showing the change in amplitude with velocity is presented in Appendix I. The amplitude of the stick-slip wave is the half distance between \( \tau_{\text{max}} \) and \( \tau_{\text{min}} \) measured in units of shear stress. The amplitude as represented in Fig. 5.6 is an average amplitude as it represents an average value of all the individual amplitudes over the test run for a given consolidation and shear cell velocity.
Fig. 5.6: Change in average amplitude as a function of shear cell velocity

The ratio of the amplitude to $\tau_{\text{max}}$ is argued in this research to give an indication of the extent of stick-slip (refer to Section 5.4). From the graph presented in Fig. 5.7 it is observed that for a given consolidation the ratio decreases with an increase to the shear velocity. In addition, it can be argued that there is a general decrease in the ratio at a given shear cell velocity with an increase in normal stress. This is however not strictly the case for the intermediate consolidations.
Fig. 5.7: Percentage ratio of amplitude of stick-slip to $\tau_{\text{max}}$ as a function of shear cell velocity

At its highest point the ratio is close to 50% for the open lid study. In such cases $\tau_{\text{min}}$ has also been recorded to fall to zero. On the other hand, at 28 kN/m$^2$ and 19.55 mm/min, the ratio is at its lowest value of $\sim$ 1% (Fig 5.7).

Consider a powder bulk as represented by two concentric blocks in Fig. 5.8. A force $F$ at a constant velocity is applied to the top half causing the entire bulk to deform by a distance $x$ in the direction of the applied force. Under this condition, the shear stress acting within the powder is $F/a$, where $a$ is the cross-sectional area. The corresponding shear strain is given by $x/b$, where $b$ is the thickness of the bulk. The ratio of the shear stress to the shear strain as described is $G$. $G$ is a measure of the rigidity (or elasticity) of a system and is technically referred to as the shear modulus.

$G$ can be rewritten as; $G = Fb/xa$. The rigidity of the powder bulk can also be described by $k$ which is expressed as the same force $F$ yielding the same deflection $x$ as given by $k = F/x$. $k$ can therefore be defined as the elastic constant. In conclusion,
$G$ and $k$ can be related by the expression $G = k (b/a)$; where $a$ and $b$ are considered constants.

The initial slope prior to shear corresponds to the shear modulus of the system as illustrated in Fig. 5.8 (also refer to Section 5.3 for examples). It is important to note that $G$ is constant with displacement as shown by the extent of linearity of the slope.

There is some evidence from Fig. 5.9 below to suggest a general increase in rigidity (shear modulus) with increase in consolidation. This is however influenced by the velocity. Apart from the maximum consolidation of 28 kN/m$^2$, all other conditions showed maximum rigidity at very low velocities followed by stepwise reduction to a level value with further increase to the velocity.

A graph showing the range of shear moduli as a function of normal stress is presented in Appendix J. As shown above the shear modulus is related to the elastic constant $k$. The corresponding graphs showing the change in $k$ with velocity are presented in Appendix K. Individual graphs shown in Fig. 5.10(a-d) reveal the sharpest drop
occurring under the higher consolidation of 12.5 kN/m². Counter to observed trends, the lowest shear modulus under 28 kN/m² was observed at the lower velocities investigated (Fig. 10d). Further increase to the velocity increased the rigidity of the powder bulk to a maximum value followed by a sharp drop to low values similar to those observed at low velocities.

Further analysis of the rigidity investigated from Fig. 5.10(a-d) reveal comparable rigidity under 4 kN/m² and 28 kN/m² at velocities less than or equal to 1 mm/min. Under this condition the powder is recorded to be at its highest shear modulus under 12.5 kN/m². The rigidity of the powder is comparable with further increase in the velocity to ~ 2 mm/min for all consolidation conditions (except "open lid" consolidation). As mentioned, the rigidity of the powder reaches a maximum under 28 kN/m² at ~ 5 mm/min. All other conditions maintain similar levels of rigidity with the increase in velocity from 2 – 5 mm/min (approx.). It is interesting to note that the maximum rigidity reached under 28 kN/m² is lower than the rigidity observed under low velocities (< 1 mm/min) for 12.5 kN/m² consolidation.

Fig. 5.9: Shear Modulus (log) as a function of shear cell velocity under all consolidations investigated.
Chapter 5: Experimental Evidence from Stick-Slip Study

**Fig 5.10a:** Shear Modulus as a function of shear cell velocity under "open lid" condition

**Fig 5.10b:** Shear Modulus as a function of shear cell velocity under 4 kN/m² consolidation
Chapter 5: Experimental Evidence from Stick-Slip Study

Fig 5.10c: Shear Modulus as a function of shear cell velocity under 12 kN/m$^2$ consolidation

Fig 5.10d: Shear Modulus as a function of shear cell velocity under 28 kN/m$^2$ consolidation
Chapter 5: Experimental Evidence from Stick-Slip Study

As mentioned before, it is argued in this study that stick-slip is manifested in the form of frictional instability. Fig. 5.11 is a graph of the coefficient of friction under static conditions. The static coefficient of friction $\mu_s$ is taken as the ratio of $\tau_{max}$ to the consolidation stress acting on the powder.

Values of the static coefficient of friction are comparable under all consolidations above the open lid condition, and for velocities above $1 \text{ mm/min}$. Below this velocity (1 mm/min) and for conditions under $12.5 \text{ kN/m}^2$ and $28 \text{ kN/m}^2$ consolidation, the static coefficient of friction is observed to start from a lower value and then increase. The evidence provided in Fig 5.11 suggests a sharp increase in the static coefficient of friction under $12.5 \text{ kN/m}^2$ above the asymptotic value before falling sharply. Graphs showing the change in static coefficient of friction with velocity under each consolidation tested are presented in Appendix L.
5.12: Dynamic coefficient of friction $\mu_d$ as a function of shear velocity

The dynamic coefficient of friction $\mu_d$ (referred [31] previously as kinetic coefficient of friction $\mu_k$) is taken as the ratio of $\tau_{min}$ to the consolidation stress acting on the powder. In all cases $\mu_d$ increases with velocity as shown in Fig. 5.12 above. Under 28 kN/m$^2$ $\mu_d$ appears to increase to a maximum constant value with further increase in velocity above 5 mm/min. Graphs showing the change in the dynamic coefficient of friction as a function of shear velocity under each consolidation load tested are presented in Appendix M.
5.3 ANALYSIS OF EXPERIMENTAL TRACES FROM SHEAR CELL STUDIES

Some experiments performed under a given normal load and drive velocity showed some unique traces. In some cases the wave patterns are not observed elsewhere in the study. In this section the author presents the observations from the trace output of starch powder. A scanned trace of each condition is included in support of the observations reported. The chart speed for all conditions has been set to 60 cm/min.

5.3.1 0.075 kN/m² consolidation (open lid)

Under this condition and 3 V motor setting, a series of experiments conducted provided drive velocities ranging between 0.62 – 0.98 mm/min. The traces show that the powder reaches and maintains relative stability at critical shear stress before the first slips occurs. It was observed that in some cases the powder slipped to zero shear stress (i.e. \( \tau_{\text{min}} = 0 \) N/m²). A close examination of the failure pattern would suggest that in some cases stick-slip occurred in pairs (although separated by a notable time period of apparent stable flow), followed by a longer steady state phase at the critical shear stress and then a pair of slip and stick would follow. Furthermore, the trace shows that the powder experiences a noticeable period of zero shear stress after the first slip before the second half of stick-slip occur. The second set of stick-slip does not display a prolonged zero shear stress following slip.

The sequence of flow patterns and observations mentioned above are shown in Fig. 5.13a-g below. They show three separate experiments marked 1, 2 and 3 starting on trace marks 40, 60 and 80 respectively; with each experiment running from right to the left of the trace. It will be observed from the traces that the observations described above are not necessarily repeated at the same time across the experimental runs.
Fig. 5.13a: The trace captures the initial stick peaking at a steady constant value \( \tau_{\text{max}} \) as identified in the experiment marked “2”.

Fig. 5.13b: The next sequence of flow patterns for these runs shows stick-slip failure for the runs marked 3 and 1. Observe that both runs maintain \( \tau_{\text{min}} = 0 \) at the end of slip.
Fig. 5.13c: The next sequences shows a fast reoccurring stick slip for the run marked “1” with steady failure maintained for the other two runs.

Fig. 5.13d: Next sequence showing stick-slip occurring for run marked “2” with stable failure maintained for long periods for the other two runs.
Fig. 5.13e: This next sequence shows the two responses to slip observed where the run marked "1" shows prolonged $\tau_{\text{min}} = 0$ followed by a second slip, and runs marked "2" and "3" begin stick immediately following slip.

Fig. 5.13f: Next sequence showing a good example of prolonged stable failure exhibited by all examples.
Figure 5.13g: In this next sequence, experiment “1” shows a clear example of a “pair” of stick-slip occurring.

As the motor speed is increased to 6 V velocity readings ranged between 2.34 and 2.47 mm/min. The traces (Fig. 5.14a&b) showed some similarities to the condition described above, however, the first of the “pair” of slips maintains a shorter time at $\tau_{min} = 0$ compared to the slower condition above. As the experiment progresses, the duration at $\tau_{min} = 0$ is reduced and the second of the “pair” of stick-slip maintains a $\tau_{min}$ value above zero. Note that the two examples in these sequences start at trace marks 20 & 40.
Chapter 5: Experimental Evidence from Stick-Slip Study

Fig. 5.14a: Start of a sequence of failure patterns highlighting reduced duration at 
\[ \tau_{\text{min}} = 0 \] compared with Fig. 5.13.

Fig. 5.14b: Trace represents the last portion in this series (not in sequence with Fig. 5.14a) which highlights measurable shear stress for the second of the "pair" of stick-slip (i.e. \( \tau_{\text{min}} \) is not equal to zero).
Further increase to the motor velocity at 12 V provided velocities in the range of 5.96 – 6.18 mm/min. The powder displayed similar symptoms to the condition under 6 V with increasing reduction to the time spent at zero shear stress following the first slip (Fig. 5.15a&b). As experienced with conditions under the slower drive velocities above, this reduced as the experiment progressed.

At 24 V the motor provided velocities between 13.2 – 15.5 mm/min. At these velocities the powder showed significant reduction to the amplitude of the second of the pair of recurring slips with no other unique features observed (Fig. 5.16).
Fig 5.15b: Next sequence showing “pairs” of stick-slip occurring in between periods of stable failure. The second of the “pair” of slips can be seen to be clearly above \( r_{\text{min}} = 0 \).

Fig 5.16: Trace showing the failure pattern at maximum velocities tested. Observe the reduction in the amplitude of the second of the “pair” of stick-slips.
5.3.2 4 kN/m² consolidation
Under this condition the waveform displayed a unique elongated slip followed by a prolonged stick midway through each experiment for all drive velocities as displayed in Fig. 5.17a&b. This observation was not repeated under any other consolidation tested throughout the entire study.

Fig. 5.17a: Trace highlighting the observed elongated slip starting at X and prolonged stick ending at Y.
5.3.3 12.5 kN/m² consolidation

At 3 V the motor produced velocities in the range of 0.5 – 0.84 mm/min. The combination of the consolidation and drive velocity resulted in irregular failure patterns. Fig. 5.18a - d show examples of two such failures. The failure pattern marked “2” displays the classic saw-toothed stick-slip as shown in Fig. 5.18a & b, however following a closer examination of both figures, it is possible to see that the amplitude of the slip phase in Fig 5.18b is almost three times the amplitude in the previous sequence. The final sequence shown in Fig. 5.18c shows a dramatic reduction in slip amplitude that is almost undetected.
Chapter 5: Experimental Evidence from Stick-Slip Study

The experiment marked "1" shows a grossly irregular stick-slip failure. In the first sequence (Fig. 5.18a) the powder is observed to slip uncharacteristically. The apparent slip is argued to be likened unto a gradual yield rather than the characteristic slip occurring over a short period of time. As the experiment progresses the powder is observed to maintain stable flow for a noticeable period before repeating the uncharacteristic slip (described here as a "gradual yield") as shown in Fig. 5.18d.

Although both experiments are performed under identical normal stress level and similar velocities, it is clear to see the unique failure patterns both exhibit. Although the reason for the inconsistency is not investigated, it is proposed that the failure pattern is sensitive to a (or combination of) parameter(s) that is (are) likely to include the drive velocity.

Fig. 5.18a: Failure pattern at the start of two experiments showing two different failure patterns.
Fig 5.18b: Trace showing next sequence highlighting irregular failure.

Fig. 5.18c: Trace showing a section of the irregular failure patterns observed.
As the drive velocity is increased to approximately 3.4 mm/min, the failure pattern becomes more regular. Fig. 5.19a shows the first slip observed under these conditions. From the trace slip occurs at a mark of 65 and 50 for experiments marked “2” and “1” respectively. However, as the failure progresses, there is evidence to suggest that the initial slip is observed to occur at a shear stress lower than \( \tau_{\text{max}} \). Fig. 5.19b shows \( \tau_{\text{max}} \) is observed to occur at higher values; trace marks of 72 and 52 for experiments marked “2” and “1” respectively.

This observation is further enhanced with increased drive velocity as shown in Fig. 5.20a & b and Fig. 5.21. In Fig. 5.20a the drive velocity is approximately 5-6 mm/min. The initial slip occurs at trace marks ~ 44 and 54 for experiments marked “1” and “2” respectively. The next sequence shown in Fig 5.20b shows slip occurring at higher trace marks of 49 and 68.

Fig. 5.21 shows additional evidence of this observation occurring at the maximum velocities tested, 22 – 23.5 mm/min under the same consolidation.
Chapter 5: Experimental Evidence from Stick-Slip Study

Fig. 5.19a: Trace showing initial slip at shear stress lower than $\tau_{\text{max}}$ (3.4 mm/min)

Fig. 5.19b: Trace showing next sequence of slip occurring at higher shear stress, $\tau_{\text{max}}$ (3.4 mm/min)
Chapter 5: Experimental Evidence from Stick-Slip Study

**Fig. 5.20a:** Trace showing initial slip at shear stress lower than $\tau_{\text{max}} (5 - 6 \text{ mm/min})$.

**Fig. 5.20b:** Trace showing next sequence of slip occurring at higher shear stress, $\tau_{\text{max}} (5 - 6 \text{ mm/min})$. 
Fig. 5.21: Trace showing slip occurring before the powder reaches $\tau_{\text{max}}$ when shear is carried out at maximum drive velocities of 22 – 23.5 mm/min.

5.3.4 28 kN/m² consolidation

It is possible to generalise that the early part of failure showed some signs of stable flow prior to sudden reduction in amplitude as shear progressed. The observed reduction in amplitude occurring for all velocities tested under this consolidation gives evidence of the influence of increased consolidation on stick-slip failure.

Some examples of the failure sequence under different drive velocities are included below in figures 5.22 – 5.24.
Chapter 5: Experimental Evidence from Stick-Slip Study

Fig. 5.22a: Trace highlighting stable flow prior to initial slip (2.5 – 3 mm/min).

Fig. 5.22b: Next sequence highlighting significant reduction in amplitude (2.5 – 3 mm/min)
Chapter 5: Experimental Evidence from Stick-Slip Study

Fig 5.22c: Trace showing failure as shear progressed highlighting an increase in the frequency of stick-slip and with reduced amplitudes (2.5 – 3 mm/min).

Under 12 V (7.5 – 7.7 mm/min) the powder showed some sign of what could be described as step-wise slip rather than a single larger drop to $\tau_{\text{min}}$. As the velocity is increased further under 24 V (16.4 – 19.6 mm/min) the waveform commences with 2 - 4 large amplitude slips relative to the smaller and regular higher frequency amplitudes that follow. The regular stick-slip that follows is atypical of classic stick-slip but instead resembles noise-like patterns in a system based on the comparative ratio of amplitude to $\tau_{\text{max}}$, (see figures 5.23a – d).
Chapter 5: Experimental Evidence from Stick-Slip Study

Fig. 5.23a: Trace highlighting stable flow prior to initial slip (7.6 mm/min).

Fig. 5.23b: Next sequence highlighting significant reduction in amplitude (7.6 mm/min)
Chapter 5: Experimental Evidence from Stick-Slip Study

Fig 5.23c: Trace showing failure as shear progressed highlighting an increase in the frequency of stick-slip and with reduced amplitudes (7.6 mm/min).

Fig 5.24: Trace capturing the development of the failure pattern as shear progressed highlighting an increase in the frequency of stick-slip and with reduced amplitudes (16.4 mm/min).
5.4 DISCUSSION

One of the questions arising from this study is how to control the onset of, or eliminate stick-slip. In this study on starch powders stick-slip occurred under all conditions investigated. The normal stress acting on the powder has been identified as a key parameter influencing stick-slip. The velocity of the shear cell during shear has also been identified as a contributor to stick-slip; however both parameters could not eliminate stick-slip over the range investigated.

In this study, stick-slip has been proposed to rely on frictional instability of the shear layer or zone. This manifests itself as the difference between the static and dynamic coefficients of friction. Stable sliding, as experienced with both maltodextrin and HVP powders, occurs as a result of an unidentifiable difference in both sets of coefficients of friction. It is argued that the extent or “measure” of stick slip termed \( Q \), can be quantified by the ratio of kinetic to static coefficients of friction which tends to an upper value of 1 indicating stable flow.

The extent of stick-slip as described in this study is useful to monitor the effect of normal stress and velocity on stick-slip. It is also useful in predicting the possible effect of changes in operating (or experimental) conditions to the flow of powders that typically exhibit stick-slip such as starch.

The extent of stick-slip \( Q \) is presented in the following chapter. It is possible to use the dynamic model (see Chapter 6) to calculate theoretical values of \( Q \) for starch powder and plot a graph of measured and calculated values of \( Q \) under all conditions of normal stress and velocity are shown in Fig. 5.25. Measured values of \( Q \) under open lid and 12.5 kN/m\(^2\) consolidation show a marked increase (i.e. tendency towards stable flow by this definition) with increase in velocity. The most “stable” flow (based on measured values) occurred under the maximum consolidation stress of 28 kN/m\(^2\) with a value of 0.934 recorded.
The consolidation stress and drive velocity have a similar effect on \( \Omega \) using calculated values derived from the dynamic model (see Chapter 6). For a given velocity the difference across the range of consolidation stresses is small compared to the measured values. In the context of this study, the most stable flow occurred under 28 kN/m\(^2\) consolidation and drive velocity of 19.5 mm/min indicated by \( \Omega = 0.97 \).

**Fig 5.25: Comparison of the extent of stick-slip, \( \Omega \), over the range of data for measured and calculated values.**

The results presented in this chapter give sufficient evidence that the extent of stick-slip is dependent on the normal stress acting on the powder and the velocity at which shear occurs. The maximum shear stress \( \tau_{\text{max}} \) was observed to generally increase with normal stress. The maximum shear stress is equivalent to the static friction force experienced at the peak of the stick phase. Static friction force \( F_s \) is directly proportional to the force acting perpendicular to the direction of the friction force as studied in planar contacts [52]. In this study the force acting perpendicular to the friction force is predominantly the force exerted by the consolidation load. Therefore, \( F_s \) is expected to increase with an increase in the consolidation load thereby having the same effect on \( \tau_{\text{max}} \).
The mechanism of friction force acting between slip-planes can be applicable to bulk powder. Furthermore, considering the powder bulk; an increase in the consolidation would increase compaction. Typically this would increase the number of contacts and the contact area between each contacting particle. Adhesion forces would be increased with increased contacting surface area. The contact strength is therefore expected to increase, exhibited by an increase in static friction force.

Generally, \( \tau_{max} \) was observed to decrease with increase in shear cell velocity. At lower velocities the contact time is increased [31] facilitating increased adhesion forces. In the work presented by Schulze [16] the contact area is reported to be dependent on the duration of contact. Therefore, reducing the contact time would cause a reduction to the contact area which would reduce the adhesion force between particles. However investigations under the top two consolidations appear to be anomalies to this trend. If the lowest value of \( \tau_{max} \) value is to be ignored, it can be seen that \( \tau_{max} \) maintains a more constant value showing minimal change with velocity compared to the other conditions (see figures 5.2 & 5.3a – d). It is argued that the adhesion force provided by the high level of compaction (i.e. high contact area) outweighs the change in contact time provided by the change in velocity until the velocity approaches maximum level, at which point the contact time is sufficiently reduced causing a reduction to \( \tau_{max} \).

\( \tau_{min} \) was found to increase with normal stress in a similar way as \( \tau_{max} \) increases with normal stress as previously described. This suggests that the shear stress required to maintain stable sliding increases with normal stress which would agree with studies performed on limestone. Refer to Chapters 3 & 4 and also work published by Akers [14].

The shear stress at the end of each slip has been taken to be \( \tau_{min} \). The difference between \( \tau_{max} \) and \( \tau_{min} \) can be assumed to relate to the amount of “stored elastic energy” dissipated during slip. Data from the study show a slight increase in \( \tau_{min} \) with velocity. The subtle increase in \( \tau_{min} \) and decrease in \( \tau_{max} \) with velocity, for a given consolidation, gives a net reduction in the ‘amount’ of stored elastic energy dissipated (according to this assumed definition). Following the same argument, this suggests a
reduction in the propensity to store elastic energy with increasing velocity. Again, an increase in the velocity is linked with a reduction in net adhesion force due to reduced contact time.

The same effect described above extends to the observed reduction in amplitude for a given load with increase in velocity (the amplitude is the difference between \( \tau_{\text{max}} \) and \( \tau_{\text{min}} \)). The observed increase in amplitude with normal stress supports the argument of increased stored elastic energy with increase in normal stress.

In their work evaluating the transition from stable sliding to stick-slip failure, Bouissou et al. [31] concluded that the normal pressure is the main parameter that influences the transition between stable sliding and stick-slip motion. It is argued that any graphic evidence of a transition from stick-slip motion to stable sliding would require a reduction of the amplitude to an undetectable minimum. At 28 kN/m\(^2\) consolidation, the significant reduction in amplitude suggests grounds for a possible transition to stable sliding as the difference between \( \tau_{\text{max}} \) and \( \tau_{\text{min}} \) is significantly reduced. The amplitude levels fall to values comparable to conditions under 4 kN/m\(^2\) consolidation.

Absolute values of the amplitude are useful; however, one of the outcomes of this study is the emphasis on the ratio of the amplitude to maximum shear stress. It is argued, based on results and trace evaluation that this contributes to a more complete analysis of the extent of stick-slip. Generally the ratio decreases with normal stress (except for the intermediate normal stress conditions). From the results, it is argued that stick-slip failure for the powders studied can be reduced by increasing the normal stress and shear velocities to high levels such that \( \tau_{\text{min}} \) is comparable to \( \tau_{\text{max}} \).

Perhaps expected, the observed frequency increases with increase in velocity. The observed frequency however shows two distinct bands with comparative values for the open lid (0.075 kN/m\(^2\)) and 12.5 kN/m\(^2\) consolidation, followed by increased but comparative values for the 4 kN/m\(^2\) and 28 kN/m\(^2\) consolidation (see Fig. 5.1 and Appendix F). The difference between the lower and upper band is pronounced at velocities above 10 mm/min (see Fig. 5.1). The low frequency values observed under
12.5 kN/m$^2$ are unexpected. There is no theoretical evidence presented to suggest an explanation for this observation, however it is important to note that the powder displayed irregular failure patterns under 12.5 kN/m$^2$ consolidation as discussed in Section 5.3.3.

If the frequency of oscillation is not taken into account, conditions where $\tau_{\text{min}}$ progressively becomes closer to or equals to $\tau_{\text{max}}$ would indicate tending towards stable sliding (though such conditions were not reached during this study). This served as the basis for laying stronger emphasis on the ratio between amplitude and $\tau_{\text{max}}$ rather than the absolute values of amplitude. It is predicted that further increase in the normal stress and shear velocity would minimise the ratio tending further towards apparent stable sliding. At such stages the static coefficient of friction would be almost equal to the kinetic coefficient of friction.

From the study the static and dynamic coefficients of friction show some evidence of the influence of normal stress. The static coefficient of friction is lowest under open-lid consolidation and increases to comparative values under all other consolidations. The dynamic coefficient of friction does not show a regular pattern of the influence of normal stress for the middle consolidations (4 kN/m$^2$ & 12.5 kN/m$^2$). However, the effect of increasing the velocity produced a decrease in the static coefficient of friction and an increase in the dynamic coefficient of friction. This is not in entire agreement with studies conducted by Bouissou et al. [30] who reported reduction in static and dynamic coefficients of friction with increased velocity and normal stress. It is important to recognise that the work carried out by Bouissou et al. [31] was performed on shearing across two surfaces coated with powder material. The nature of their work allowed for a larger range of normal stress used in their study which is atypical of the shear cell instrument used in this study. In addition shearing along surfaces would isolate any effect of particle rearrangement experienced within the bulk powder.

The rigidity of the system increases with consolidation due to an increase in adhesion force with increased compaction. As mentioned previously, the shear modulus gives an account of the rigidity of the system (see figures 5.9, 5.10a – d & Appendix J, K).
Chapter 5: Experimental Evidence from Stick-Slip Study

Under the lower three consolidation loads the shear moduli were observed to decrease gradually to a near constant value, with a sharper drop exhibited under 12.5 kN/m² (the highest consolidation within this group). At 28 kN/m² the shear moduli were observed to increase moderately with velocity at the very low velocities investigated. Under this relatively high consolidation the powder exhibits high adhesion force. At low velocities it is proposed that there is a gradual rupture of the bonds at a rate equivalent to the shear rate acting on the powder. A slight increase to the velocity increases the shear rate, however it allows for an insufficient response time for the same rate of bond rupture ("tear"). The imbalance in response to an increased shear rate is marked with an increase in shear modulus.

The argument presented here is supported by observations presented in Section 5.3 describing inconsistent amplitudes and signs of slips (indicative of "bond rupture" as argued) occurring before the powder reaches critical shear stress \( \tau_{\text{max}} \) under low velocities tested for maximum consolidation – 28 kN/m².

The shear modulus of a powder depends upon the interparticle forces acting which can be described by the critical shear stress. Due to the consolidation technique employed, it is assumed that there is no particle re-ordering before failure. Results presented in this chapter show that the shear modulus is influenced by the drive velocity. At a given velocity, the rate of shear is sufficient to cause rupture of the bonds at an increased rate. At this point the shear moduli reach a peak value characterised by the maximum consolidation. Further increase to the velocity would cause a reduction in the rate of rupture of the bonds depicted by a drop in shear moduli measured. This argument elucidates the observation presented for consolidation under 12.5 kN/m² (Section 5.3) where irregular stick phases were observed at low velocities but became more regular with further increase to the velocity. All other consolidations are at this stage exhibited by the drop in shear moduli with increase in velocity where it is argued that the adhesion strength is low enough to accommodate a response to faster bond rupture with increase in velocity. It is further argued that equilibrium is reached for the effect of the rate of shear, depicted by the velocity of the shear cell, to rate of bond rupture. This is noted to be above 5 mm/min for the lower consolidations where the values of shear moduli appear
constant. The same is predicted to occur under maximum consolidation if the velocity could be increased further beyond the limit of the instruments employed.

A general review of the trends of the results presented in Section 5.2 shows that conditions under 12.5 kN/m$^2$ tend to fall outside of the observed trends. The cause of this is not known, however it may relate to the possible onset of transition from low consolidation (less than 4 kN/m$^2$) to higher consolidation as investigated in this study.
5.5 CONCLUSION

The stick-slip study conducted shows evidence that the extent of stick-slip can be affected by an increase in the level of consolidation and the rate of shear. The maximum shear stress ($\tau_{\text{max}}$) observed was found to increase with normal stress. $\tau_{\text{max}}$ has been described to reflect the static friction force ($F_s$) prior to failure. $\tau_{\text{max}}$ was also found to decrease with velocity.

The shear stress at the end of slip ($\tau_{\text{min}}$) was found to increase with normal stress and increase with velocity. This value reached a plateau under maximum normal stress of 28 kN/m$^2$.

$\tau_{\text{max}} - \tau_{\text{min}}$ has been presented as a reflection of the amount of stored elastic energy dissipated during slip. This value decreased with velocity therefore the propensity to store elastic energy has been concluded to decrease with velocity.

The observed frequency values increased with velocity for all consolidations investigated. The frequency values however showed two distinct bands with the lower band showing comparable values under open lid (0.075 kN/m$^2$) and 12.5 kN/m$^2$ consolidations, and the upper band showing larger frequency values under 4 kN/m$^2$ and 28 kN/m$^2$ consolidation.

There were comparable values of static coefficient of friction ($\mu_s$) with increase in normal stress above open-lid consolidation. The highest values of $\mu_s$ were recorded under open lid consolidation. Increase in velocity caused a decrease in $\mu_s$ for a given normal stress level. There is inconclusive evidence to identify the effect of normal stress on the dynamic coefficient of friction ($\mu_d$), however $\mu_d$ increased with velocity for a given normal stress.

The rigidity of the powders characterised by the shear modulus increased with normal stress. The influence of velocity on rigidity followed two unique trends. For consolidations below the maximum normal stress, the rigidity decreased with
velocity. Tests carried out using 28 kN/m² showed that the powder was observed to be more rigid with increase in velocity up to ~ 7 mm/min. This level of rigidity is maintained between ~ 7 - 16 mm/min. Further increase to the velocity is observed to render the powder more elastic (i.e. elastic constant decrease). The observations regarding the rigidity of the powder have been attributed to the rate of bond rupture.

Experiments carried out in this study were unable to eliminate stick-slip. From the study conducted, it is suggested that stick-slip may be eliminated at low consolidation and shear velocity, or at high consolidation and high velocity. In addition it is suggested that the occurrence of stick-slip is likely to be material dependent (such as with starch) and may prove more difficult to eliminate for some powders than others.
CHAPTER 6: MATHEMATICAL MODEL OF STICK-SLIP

6.1 INTRODUCTION

The observations presented in this study have added to the understanding of stick-slip failure and the controlling factors. A further approach in the understanding was to apply a mathematical model to the failure pattern based on the physical properties and knowledge of the controlling factors. Presented in this chapter is the logical development of the concept of stick-slip from a "simple model" to a workable mathematical [dynamic] model that represents the distinct phases of stick and slip.

The simple model is based on the illustration of a powder block as presented in Chapter 5 and can be used to calculate the duration of the stick phase. Further developments were made by considering the concentric (stick) and eccentric (slip) phases of a spring-pulley system. From this, mathematical expressions describing stick-slip (i.e. dynamic model) are presented.

A description of how both concepts represent conditions of powders within the shear cell is presented. The dynamic model relies on experimental input data which have been identified in this chapter in order to generate output data necessary for comparison with experimental observations. In addition, the assumptions and limitations of the dynamic model are presented in order to identify its effectiveness.

6.2 SIMPLE MODEL

In the previous chapter an illustration of the powder bulk was used to describe the relationship between the shear modulus, $G$ and the elastic constant, $k$. Consider the same block presented in Fig. 6.1 below with a thickness $b$ and shear modulus $G$. In order to simulate conditions experienced in the shear cell, assume the block shears along a shear plane at height $d$. It is further assumed that the block is elastic and the
base remains stationary, while the top half is subjected to a constant velocity $v$ resulting from a force $F$ acting over area $a$.

![Diagram of bulk powder](image)

**Fig. 6.1: Schematic of bulk powder used to illustrate the relationship between $k$ and $G$.**

Let us assume that the block will “yield” at a yield stress of $\tau_y$. If the system is taken to be stress-free at time $t = 0$ at which point a constant force $F$ is applied, the shear strain $\gamma$ in the block can be calculated to be the distance moved by the block in the direction of the force $(vt)$ divided by the thickness of the block ($b$). Therefore:

$$\gamma = \frac{vt}{b}$$

The shear modulus is defined as the shear stress associated with a unit measure of shear strain. From this definition the shear stress $\tau$ acting on the block can be represented as:

$$\tau = G\frac{t}{b}$$
At a given time \( t \), the stress applied reaches a critical value equal to the yield stress \( \tau_y \) and the block yields. This can be represented as:

\[
\tau_y = Gv \frac{t}{b}
\]

Rearranging, an expression for the time taken for “yield” to occur becomes:

\[
t = \frac{\tau_y b}{Gv}
\]

From the expressions above, the “yield” stress of the block represents the stress value causing sudden slip. The time taken for “yield” to occur from an initial condition of zero shear stress represents the duration of stick \( (t) \). The above expressions are useful in that they present a simple illustration of the stick phase up to the initial slip. If the “yield” stress \( \tau_y \) is assumed to be \( \tau_{\max} \) as described in Chapter 5, it follows that in the case where the shear modulus and yield stress of a powder are known, the above expressions can be applied to calculate the duration of the stick phase. The duration of the stick phase is an important parameter in describing stick-slip as experienced by powders.

The expression however is limited to describing conditions from an initial condition of zero shear stress up to the initial “yield” without describing the slip phase. This presents a barrier in describing the events leading to a repeated cycle of stick-slip phases. A revised model would be needed to fully describe the individual phases of the stick-slip cycle. This is considered in the following section.

In terms of modelling stick-slip, the expression here assumes that the powder will slip at \( \tau_y \) which is not always the case. It is possible for the powder to maintain steady flow for a period of time before slip occurs and still be described to yield. Consequently, the calculated time taken for initial slip is based on the assumption that the powder slips at \( T = \tau_y b / Gv \), which is not always the case.
6.3 DYNAMIC MODEL

6.3.1 Development of Model

In order to represent stick-slip as it occurs in bulk powders, consider a spring-pulley system as illustrated in Fig. 6.2 below [53]. The mass, \( M \), is driven by the movement of the motor \( A \), which compresses the spring. Movement of the mass occurs once the force exerted from the compressed spring is greater than the friction force, \( F \), which serves to oppose motion.

![Schematic of pulley driven system](image)

**Fig. 6.2: Schematic of pulley driven system**

However, according to Halling [53], a detailed study of the sequence of events causing motion reveals the onset and possible continuation of stick-slip motion. Experimental evidence provided in **Chapter 5** show that static friction force (and critical shear stress) varies with drive velocity. Therefore, assuming that the friction, \( F \), varies with velocity, if the motor is driven at a constant velocity such as to compress the spring, movement of the mass is opposed by friction between the base of the mass and the surface. The increasing drive force provided by the spring is matched by the growing friction force which tends to an upper limit, \( F_s \) (as described in **Section 5.3**) i.e. static friction.
Once the mass is set in motion, the friction falls rapidly to the dynamic friction value, $F_d$ (see Section 5.3) such that there is an unbalanced force $F_s - F_d$ causing a sudden acceleration. The velocity of $M$ increases until the drive force falls to the value $F_d$. It follows that if the velocity of the mass is greater than the motor speed, the spring force continues to fall and the mass decelerates. Eventually, the mass comes to rest and the cycle of events is repeated.

The time taken for the friction force to climb to $F_s$ from zero represents the stick phase, immediately followed by the slip phase until the mass comes to a stop.

The analogy of the spring-pulley system has a resemblance to the shearing application of the Jenike-type shear cell. It follows the argument presented in this study which describes stick-slip as frictional instability that is dependent on the material’s (elastic) ability to store the energy caused by the driving force before yielding or failure. Halling [53] captures this with a simplified expression as documented below. The material’s physical property describing its ability to store the energy caused by the driving force is described by the spring constant, conventionally denoted by $k$. The material’s subsequent reaction to failure is captured by the frictional resistance measured as described by $F$.

As the motor is driven, (assumed to be at a constant velocity), such as to move the mass, the spring compresses. In the case of the shear cell, the motor is driven at a constant velocity such as to shear the powder bulk. However, rather than an immediate shear it can be assumed that there is some elastic compression of the powder bulk.

The force associated with the stored elastic energy within the powder bulk grows as the motor drives the shear cell. Slip occurs at a force equal to the static friction force of the slip plane.

The above description presents a conceptual approach to modelling stick-slip failure using the spring-pulley system. From this, a refined mathematical model can be constructed as presented below:
By manipulation of the expression presented by Halling, [53] a force balance can be written describing the motion in Fig 6.2 as follows:

$$M \frac{d^2x}{dt^2} + k(x - vt) = -F$$

Where; $v$ is the drive (motor) velocity
$t$ is the time taken
$k$ is the spring constant
$x$ is the displacement of the mass $M$
$F$ is the frictional resistance by the mass.

Let us assume that the mass rests on a rough surface and is driven at a low and constant velocity $v$. (It is important to note that all other notations are in accordance with those given in Chapter 5). The force in the spring during the compressive stage is assumed to be linear with time and eventually reaches a maximum value equal to the static frictional force $F_s$, given by:

$$F_s = \mu_s Mg$$

Where; $\mu_s$ as defined in Chapter 5 is the coefficient of static friction.

At this point slip occurs and the mass moves. It is important to note that the mass is not accelerated if $\mu_s = \mu_d$ (where $\mu_d$ as defined in Chapter 5 is the dynamic coefficient of friction), whereby the force in the spring will remain constant at $\mu_s Mg$. However this is not the case in stick-slip and $\mu_d < \mu_s$ is readily assumed. If $\mu_d < \mu_s$ the force in the spring will be $kx$ and the frictional force $\mu Mg$. A force balance can be written for the motion of the block such as:

$$-M \frac{d^2x}{dt^2} = kx - \mu Mg$$

[1]
The force balance above also represents the forces acting in the shear cell as shown in Fig. 6.3 below:

![Fig 6.3: Schematic of shear cell setup highlighting forces in action](image)

**Fig 6.3:** Schematic of shear cell setup highlighting forces in action

The force balance during slip gives (assuming the drive velocity is negligible compared to the slip velocity):

\[
M \frac{d^2x}{dt^2} + kx = \mu Mg
\]

Where; \(x\) is the displacement of the block.

Rearranging gives the same expression as equation [1]. The solution to this second order differential equation has the following standard form as given by Halling [53]:

\[
x = \frac{\mu Mg}{k} + A \sin(\omega t) + B \cos(\omega t)
\]  

[2]

Where; \(\omega = \sqrt{\frac{k}{M}}\), and

[3]

\(A\) and \(B\) are constants.

Using a time of \(t = 0\) as a boundary condition for the start of slip, it follows that:
Friction force at \( t = 0 \) is \( F_s \);

\[ F_s = kx \]

Also,

\[ F_s = \mu_s Mg \]

Therefore,

\[ x = \frac{\mu_s Mg}{k} \]  \[\text{[4]}\]

Assuming that the velocity of the block prior to slip is zero (i.e. the block is momentarily stationary), it follows that at \( t = 0 \):

\[ \frac{dx}{dt} = 0 \]  \[\text{[5]}\]

Furthermore it follows that, at \( t = 0 \) and substituting \( x \) in [4] into [2] we get the expression;

\[ \frac{\mu_s Mg}{k} = \frac{\mu_s Mg}{k} + A\sin(0) + B\cos(0) \]

Therefore,

\[ B = \frac{\mu_s Mg}{k} - \frac{\mu_s Mg}{k} \]

\[ B = \frac{(\mu_s - \mu_d)Mg}{k} \]  \[\text{[6]}\]

Similarly, using a time of \( t = 0 \) as a boundary condition for the end of slip, it follows that:

Friction force at \( t_{\text{end of slip}} = 0 \) is \( F_d \).
Chapter 6: Mathematical Model of Stick-Slip Study

\[ F_d = k\dot{x} \]

Also,
\[ F_d = \mu_d Mg \]

Therefore,
\[ x = \frac{\mu_d Mg}{k} \]

[7]

Assuming that the mass comes to rest at the end of slip the velocity of the block prior to stick is zero, at \( t = 0 \) (i.e. \( t_{(end\ of\ slip)} \)) it follows that:

\[ \frac{dx}{dt} = 0 \]

[8]

Furthermore it follows that, at \( t_{(end\ of\ slip)} = 0 \) and substituting \( x \) in [7] into [2] we get the expression;

\[ \frac{\mu_d Mg}{k} = \frac{\mu_d Mg}{k} + A\sin(\omega t) + B\cos(\omega t) \]

Therefore,

\[ -A\sin(\omega t) = B\cos(\omega t) \]

Therefore;

\[ -A = B \left[ \frac{\cos(\omega t_{(end\ of\ slip)})}{\sin(\omega t_{(end\ of\ slip)})} \right] \]

[9]

Applying the boundary condition \( t_{(end\ of\ slip)} = 0 \) to Equation [9] gives:

\[ A = 0 \]

[10]

(as \( \sin(0) = 0 \)).
Therefore using [6] and [10] in [2], an expression for the distance moved by the mass can be written:

\[ x = \frac{\mu_s Mg}{k} + \left[ \frac{(\mu_s - \mu_d) Mg}{k} \right] \cos(\sigma t) \]  \[11\]

Using \( F = kx \), Equation [11] can be rearranged to give the friction force:

\[ F = \mu_s Mg + (\mu_s - \mu_d) Mg \cos(\sigma t) \]  \[12\]

Differentiating Equation [11], the velocity becomes:

\[ \frac{dx}{dt} = -\left[ \frac{(\mu_s - \mu_d) Mg \sigma}{k} \right] \sin(\sigma t) \]  \[13\]

According to the second boundary condition, slip will stop when the velocity is reduced to zero i.e. when \( \sigma t = \pi \). It follows that for \( \sigma t = \pi \), \( \cos(\sigma t) = -1 \).

Therefore the distance travelled is:

\[ x = \frac{\mu_s Mg}{k} - \frac{(\mu_s - \mu_d) Mg}{k} \]  \[14\]

\[ x = \frac{(2 \mu_s - \mu_d) Mg}{k} \]

The total distance travelled during slip becomes the datum distance [4] minus the distance reached at the end of slip [14] above. Thus;

Slip distance \( x_s \) is:

\[ x_s = \frac{\mu_s Mg}{k} - \frac{(2 \mu_s - \mu_d) Mg}{k} \]  \[15\]

\[ x_s = \frac{2(\mu_s - \mu_d) Mg}{k} \]
Rearranging [14] we obtain the force at the end of slip:

\[ F_{\text{endofslip}} = (2\mu_d - \mu_s)Mg \]  

[16]

It is assumed that static friction is re-engaged at the end of slip i.e. no reverse slip occurs. The block will then again move forward at a velocity \( v \) until \( x = \frac{\mu_s Mg}{k} \).

In summary, the force in the spring grows linearly according to \( F = kx \) to a maximum value \( \mu_s Mg \) (stick phase). The force falls from this value to \( (2\mu_d - \mu_s)Mg \) over a time period of \( \frac{\pi}{\omega} \) representing the slip phase. Following this the force increases to maximum force \( \mu_s Mg \) over the time interval \( \frac{(2\mu_s - 2\mu_d)Mg}{kv} \).

The force acting on the block undergoing stick-slip motion is as a function of time represented in Fig. 6.4 below:

**Fig. 6.4 Sketch of the model in operation**
6.3.2 Evaluation of Dynamic Model

The effectiveness of the dynamic model can be determined by direct comparison with experimental data, i.e. plotting a theoretical trace and compare with experimental trace. Using the expressions presented above, the following steps can be taken to obtain data to plot a theoretical trace as follows.

1) Measure $F_{\text{max}}$ from trace and calculate $\mu_s$.
2) Use calculated value of $\mu_s$ above and measured value of $F_{\text{min}}$ to calculate $\mu_d$.
3) Calculate elastic constant $k$ ($\Delta$force on load cell / $\Delta$distance moved by base) as described in Fig. 6.5.
4) Calculate the velocity $v$ and $\omega$ from measured parameters (i.e. mass, elastic constant, distance travelled by base and time taken to cover distance travelled by base).
5) Measure the slip and stick duration.
6) Calculate the slip and stick duration.

(Fig. 6.5 below shows an example of how to measure parameters from a given trace. Expressions for calculated parameters have been presented in the development of the dynamic model and some shown in Fig. 6.4 above).
Chapter 6: Mathematical Model of Stick-Slip Study

The dynamic model presented in this chapter has been applied to a total of 46 experimental runs and tested using results from “Run 44” as an example. The data collected and calculated values are summarised in the Table 6.1 below.

It is possible to identify a perfect match for measured and calculated values of $F_{\text{max}}$, $F_{\text{min}}$ and $\mu_s$ as these parameters are obtained by manipulation of the same values or/and mathematical expressions. Calculated values of $\mu_k$ however are derived by an expression unique to the dynamic model. In the example shown in Table 6.1 the measured and calculated values of $\mu_k$ are 0.68 and 0.72 respectively. Both figures are comparable and reasonable as they are less than 1 and also less than $\mu_s$, which is the same value in both cases as already mentioned.
## Chapter 6: Mathematical Model of Stick-Slip Study

<table>
<thead>
<tr>
<th>Experimental Conditions</th>
<th>Measured Parameters from Trace</th>
<th>Calculated Parameters from Dynamic Model</th>
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</thead>
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<tr>
<td>Consolidation Load, 7.5 kg</td>
<td>Critical Consolidation Shear Stress, $\tau_{\text{max}}$, 21.6 kN/m²</td>
<td>Coefficient of static friction, $\mu_s$, calculated 0.76</td>
</tr>
<tr>
<td>Consolidation Stress, 28.3 kN/m²</td>
<td>Friction force, $F_{\text{max}}$, measured 56 N</td>
<td>Coefficient of dynamic friction, $\mu_d$, calculated 0.72</td>
</tr>
<tr>
<td>Velocity, $v$, 7.55 mm/min</td>
<td>Coefficient of static friction – defined as $\tau_{\text{max}}$/consolidation stress, $\mu_s$, measured 0.76</td>
<td>“Extent of stick-slip”, $\Omega$, $\mu_d/\mu_s$ 0.95</td>
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<td>Thickness of powder, $b$, 22 mm</td>
<td>Shear Stress at end of slip, $\tau_{\text{min}}$, 19.3 kN/m²</td>
<td>Duration of stick, $t_{\text{stick}}$, 1.56 secs</td>
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<td>Friction force at end of slip, $F_{\text{min}}$, 50.2 N</td>
<td>Duration of slip, $t_{\text{slip}}$, 2.9 secs</td>
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<td></td>
<td>Coefficient of dynamic friction – defined as $\tau_{\text{min}}$/consolidation stress, $\mu_s$, measured 0.68</td>
<td>Friction force, $F_{\text{max}}$, 56 N</td>
</tr>
<tr>
<td></td>
<td>Elastic constant, $k$, 29.3 kN/m</td>
<td>Friction force, $F_{\text{min}}$, 50.2 N</td>
</tr>
</tbody>
</table>
Chapter 6: Mathematical Model of Stick-Slip Study

Table 6.1: An example of the application of the model ("Run 44")

<table>
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<th>Parameter</th>
<th>Value</th>
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<tbody>
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<td>Shear Strain, $\gamma$</td>
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<tr>
<td>Shear Modulus, $G$</td>
<td>249 kN/m²</td>
</tr>
<tr>
<td>Angular Velocity, $\omega$</td>
<td>1.09 radians</td>
</tr>
<tr>
<td>Duration of stick, $t_{\text{stick-measured}}$</td>
<td>0.8 secs</td>
</tr>
<tr>
<td>Duration of slip, $t_{\text{slip-measured}}$</td>
<td>0.2 secs</td>
</tr>
</tbody>
</table>

The duration of stick in the above example is measured at 0.8 seconds and calculated to be 1.56 seconds (see also Fig 6.6d). In the context of evaluating the model, it is argued that the calculated values represent a good estimate of the measured duration of stick as evident across the entire range of data collected (see Fig 6.6a-d & Appendix N). Both the measured and calculated values show a decrease in the duration of the stick phase with increase in shear cell velocity, and an increase in the duration of the stick phase with increase in consolidation stress.
Comparison of the Duration of Stick under Open Lid (0.075 kN/m$^2$) consolidation.

Fig 6.6a: Comparison of the duration of stick under open lid (0.075 kN/m$^2$) consolidation.

Comparison of the Duration of Stick under 4 kN/m$^2$ Consolidation

Fig 6.6b: Comparison of the duration of stick under open lid (4 kN/m$^2$) consolidation.
Fig 6.6c: Comparison of the duration of stick under open lid (12.5 kN/m$^2$) consolidation.

Fig 6.6d: Comparison of the duration of stick under open lid (28 kN/m$^2$) consolidation.
Counter to this, the measured and calculated durations of slip are 0.2 and 2.9 seconds respectively highlighting a noticeable disparity. Furthermore, Fig 6.7a-d (see also Appendix O) show a clear but consistent disparity between measured and calculated values of the duration of slip. The measured values show a weak response with increase in velocity and normal stress while the calculated values show an increase with increase in normal stress.

The reason for the disparity between measured and calculated values is not clear. The weak relationship between measured values with velocity over the range of normal stresses may suggest a limitation in the response provided by the instrument.

**Fig 6.7a: Comparison of the duration of slip under open lid (0.075 kN/m²) consolidation.**
Chapter 6: Mathematical Model of Stick-Slip Study

Comparison of the Duration of Slip under 4 kN/m² consolidation

Fig 6.7b: Comparison of the duration of slip under open lid (4 kN/m²) consolidation.

Comparison of the Duration of Slip under 12.5 kN/m² consolidation

Fig 6.7c: Comparison of the duration of slip under open lid (12.5 kN/m²) consolidation.
Chapter 6: Mathematical Model of Stick-Slip Study

Fig 6.7d: Comparison of the duration of slip under open lid (28 kN/m²) consolidation.

The measured dynamic coefficients of friction $\mu_d$ show a tendency to increase slightly with velocity and also increase with normal stress apart from the 4 kN/m² and 12.5 kN/m² consolidations which are reversed.
Comparison of Measured and Calculated values of Kinetic Coefficients of Friction

The calculated dynamic coefficients of friction $\mu_d$ (shown in Fig. 6.8 above) show no obvious change with velocity under a given normal stress but showed an increase with normal stress apart from the 4kN/m$^2$ and 12.5kN/m$^2$ consolidations which are more evenly matched. The individual graphs per given consolidation load are included in Appendix P.

Using both sets of data (measured and calculated) as described in this section, it is possible to plot the actual and theoretical trace of “Run 44” as shown in Fig. 6.9 below. The time taken for the powder to initially yield is calculated using the expression from the “simple model” and therefore readily assumes a linear change in friction force with time until the point of slip i.e. the powder will slip as the shear stress immediately reaches the critical shear stress. Graphs of the time to initial yield are presented in Appendix Q. For the purpose of a graphical comparison, the measured duration of slip (Fig. 6.9 below) has been used instead of the calculated value, due to the huge disparity in measured and calculated slip times. In addition, the graphs such as Fig. 6.9 showing a comparison of actual and theoretical trace have
been drawn by drawing straight lines between each measured or calculated stick and slip points.

![Graph showing an example of an actual trace alongside a theoretic trace using the dynamic model.](image)

**Fig. 6.9:** Graph showing an example of an actual trace alongside a theoretic trace using the dynamic model.

The graph above (Fig 6.9) represents a useful method to evaluate the dynamic model. In this example, it is clear that the duration of stick is calculated to be longer than the measured duration from the trace. Despite this discrepancy, it is argued that across the range of experimental conditions tested, the dynamic model has shown to give a valuable account of the failure pattern of a powder under a given condition.

In the example above, the original stick-slip failure pattern is presented in Fig 6.10 below. Note that the orientation of the failure pattern is laterally inverted and the numbering on the ordinate axis are in arbitrary units.

It is possible to identify three “phases” as shown by the sequence of failure in Fig 6.10a, b & c respectively. The initial phase (Fig. 6.10a) shows classic saw-toothed stick-slip failure which develops into periods of stable failure at $F_{\text{max}}$ (Fig. 6.10b) where slip would have been expected to occur. As shear progresses, stick-slip is
observed to occur more frequently with a significant reduction in amplitude (Fig. 6.10c).

A comparison of Fig 6.9 and Fig 6.10 highlights a limitation of the dynamic model as it is not robust enough to capture the unique failures observed. The graph of the dynamic model as applied in Fig. 6.9 is effective in describing continuous failure (Fig 6.10c) where stick-slip is assumed to be established and continuous.

---

**Fig. 6.10a:** Start of sequence showing initial failure characterised by classic saw-toothed stick-slip.

**Fig. 6.10b:** Next sequence showing apparent stable failure characterised by plateaus before slip occurs (also refer to Section 5.3).
Fig. 6.10c: Final sequence showing rapid saw-toothed stick slip with noticeable reduced amplitude.

Further evidence of the application of the dynamic model above is presented for other experimental trials under different consolidation stresses below. The complete set of graphs showing measured and calculated traces including the initial slip phase are presented in Appendix R.
Chapter 6: Mathematical Model of Stick-Slip Study

Fig. 6.11: Graph showing an example of an actual trace alongside a theoretic trace using the dynamic model. A scan of the experimental trace is included and discussed in Fig. 5.21 marked “2”.

Fig. 6.12: Graph showing an example of an actual trace alongside a theoretic trace using the dynamic model. A scan of the experimental trace is included and discussed in Fig. 5.17b marked “3”.

167
Fig. 6.13: Graph showing an example of an actual trace alongside a theoretic trace using the dynamic model. A scan of the experimental trace is included and discussed in Fig. 5.16 marked “3”

Although it is argued that the model describes the key features of stick-slip failure, however the graphs above assume a linear shear/force response with time between each stick and slip points. It is argued that this is a reasonable assumption for the majority of experimental conditions. As mentioned previously in this section, the failure pattern prior to initial slip may not be perfectly linear but exhibits asymptotic failure pattern just before the initial slip. It is possible to plot the gradual change in friction force during the slip phase by applying the expression $F = \mu_s Mg + (\mu_s - \mu_d)Mg\cos(\omega t)$ as given above. This expression applied to calculated values of coefficients of friction gives a theoretical duration of slip as summarised in Table 6.1 above. The graph of the slip phase is presented in Fig. 6.14 below:
As mentioned in this section (and perhaps expected), the dynamic model is limited in capturing some of the unique features of stick-slip observed as described above and in Section 5.3. Some of these unique features include an elongated slip followed by an extended stick phase halfway through failure, and changes in the amplitude and frequency as exhibited by Runs 44 (Fig 6.10).

It is argued however, that the model serves as a valuable tool in characterising the parameters that govern the individual stages of stick-slip failure. For example, the dynamic model shows the direct inverse relationship of drive velocity on the duration of stick. The duration of slip, however, according to the dynamic model is influenced largely by the consolidation stress. Furthermore, the expressions developed from the dynamic model highlight the significant contributions of the normal stress (or load), as the term $M$ or $Mg$ appears describing $F_{\text{max}}$, $F_{\text{min}}$, $\tau_{\text{slip}}$ and $\tau_{\text{stick}}$; where $M$ and $g$ represent the consolidation mass and force per unit mass due to gravity respectively.

In conclusion, the work presented in developing the dynamic model contributes largely to understanding the various stages of stick-slip failure and the impact of the normal stress and drive velocity. A spreadsheet showing all the data used is included in Appendix S.
CHAPTER 7: RESEARCH CONCLUSION AND FUTURE WORK

Based on the study of the literature reported in Chapter 2 the cohesion of the powders investigated was expected to all increase with increase in relative humidity. Shear cell studies investigating the influence of adsorbed moisture on cohesion revealed an increase in cohesion with humidity for one of the three powders – HVP. The increase in cohesion is reported to be as a direct response to the HVP powder exhibiting a high affinity for water. HVP powders displayed signs of over-consolidation during the preshear which is argued to be as a result of interlocking.

Maltodextrin powders showed no change in cohesion with relative humidity however it is important to note that the powders felt more difficult to stir under dry conditions. As with the HVP powder, the effect of interlocking is argued. Furthermore, the possible effect of lubrication from moisture is removed under dry conditions thereby making it difficult to stir.

Starch powders showed inconclusive response to changes in the relative humidity although it is reported to become increasingly difficult to stir with increase in humidity. It is argued that the effect of relative humidity on cohesion is greatly reduced due to internal adsorption of the available water causing a reduced effect to the formation of liquid bridges.

The conflicting evidence of the values of cohesion for starch and maltodextrin, and the difficulty experienced in stirring is linked with the complexity of food powders. In addition it is concluded that the shear cell used is not sensitive enough for cohesion measurement for both powders under the humidity conditions tested.

Maltodextrin powders displayed stick-slip under dry conditions while starch powders displayed stick-slip under all humidity conditions tested. Stick-slip study carried out on starch revealed that stick-slip is governed largely by the level of consolidation and drive velocity. The development of the dynamic model served as a useful tool in mapping the
changes to material and physical properties constituting stick-slip as the levels of consolidation and velocity are varied. As part of the model, the parameter $\Omega$, is presented in this study as a useful measure in describing the extent of stick-slip. This is useful in evaluating the effect of normal stress and velocity investigated.

Results from the stick-slip study showed some inconsistent trends for the intermediate consolidation levels of $4 \text{kN/m}^2$ and $12.5 \text{kN/m}^2$. As a recommendation for future work, it is possible to further investigate stick-slip failure with smaller increments to the level of consolidation (e.g. $4 \text{kN/m}^2$, $6 \text{kN/m}^2$, $8 \text{kN/m}^2$, $10 \text{kN/m}^2$ & $12 \text{kN/m}^2$). Furthermore, it is possible to investigating beyond the maximum consolidation and velocities used in this study and observe the effect on the extent of stick-slip, $\Omega$.

The discrepancy between the calculated duration of slip using the dynamic model compared to the actual duration of slip presents an avenue for future work. This could require advancement to the existing model to accommodate this observed discrepancy.

There is scope to advance the current study by investigating the mechanical properties of the individual particles as well as the powder bulk. An example of such is a compressive test using a conventional instrument such as the Instron.

It would be of interest to repeat the stick-slip study using other materials and relate solid material mechanical properties to stick-slip failure.

Finally, a parallel study repeating the work done in this study (and incorporating the proposed ideas for future work above) using different type of shear tester remains a good candidate for future study.
REFERENCES


48) Barbosa-Canovas G.V., Ortega-Rives E., Juliano P. and Yan H. Food Powders (2005), Plenum Publishers, New York, USA.


LIST OF APPENDICES

Appendix A - Material Manufacturing Procedure

Appendix B - Extra SEM photographs

Appendix C - Calibration of Shear Cell

Appendix D - Graphs of Individual Yield Loci

Appendix E - Graph of Shear Cell Velocity as a function of Motor Setting

Appendix F - Graphs of Effect of Shear Cell Velocity on Observed Stick-Slip Frequency

Appendix G - Graphs of Effect of Shear Velocity on $F_{max}$

Appendix H - Graphs of Effect of Shear Velocity on $F_{min}$

Appendix I - Graphs of Effect of Shear Cell Speed on Average Amplitude

Appendix J - Graph of Shear Modulus (G) as a function of Normal Stress (over a range of Velocities)

Appendix K - Graphs of Elastic Constant (k) as a function of Shear Cell

Appendix L - Graphs of Static Coefficient of Friction

Appendix M - Graph of Dynamic Coefficient of Friction
<table>
<thead>
<tr>
<th>Appendix N</th>
<th>Graph of Duration of Stick – Measured and Calculated</th>
</tr>
</thead>
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<tr>
<td>Appendix O</td>
<td>Graph of Duration of Slip – Measured and Calculated</td>
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<tr>
<td>Appendix P</td>
<td>Graphs of Dynamic Coefficient of Friction – Measured and Calculated</td>
</tr>
<tr>
<td>Appendix Q</td>
<td>Graphs of Yield Time – Measured and Calculated</td>
</tr>
<tr>
<td>Appendix R</td>
<td>Graphs of (i) Stick-Slip - Measured and Calculated values, and (ii) corresponding graphs showing the Slip Phase using the Dynamic Model</td>
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<tr>
<td>Appendix S</td>
<td>Spreadsheet of Stick-Slip Study</td>
</tr>
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</table>
APPENDIX A

MATERIAL MANUFACTURING PROCEDURE
MATERIAL MANUFACTURING PROCEDURE

MALTODEXTRIN

Maltodextrin is produced from the hydrolysis of cornstarch. The industrial procedure for the production of maltodextrin powder is presented in Fig. A1 below. When molecules of starch are hydrolysed, dextrins, maltose, and finally glucose are formed according to the reaction below [37]. The reaction is catalysed by enzyme or acid plus heat:

\[
\text{Starch} + \text{Water} \xrightarrow{\text{Enzymes or Acid plus Heat}} \text{Dextrin} + \text{Maltose} + \text{Glucose}
\]

Fig. A1: Process steps for the production of maltodextrin powders.
HVP

The manufacturing procedure for HVP has not been extracted from literature, but instead has been obtained from Centura (Fig. A2). Protein stock (raw material) undergoes a hydrolysis reaction. The hydrolysed product is neutralised and filtered. The filtered liquor is evaporated and stored.

\[ \text{Protein Stock} \]

1. Hydrolysis Reaction
2. Neutralisation
3. Filtration
4. Evaporation

\[ \text{Liquid Storage} \]

- Spray Dry
  - Sieving
  - End Product (Package)

- Vacuum Drying
  - Milling
  - Sieving
  - End Product (Package)

\( \text{Fig. A2: Process steps for the production of hydrolysed vegetable protein powders by – (a) spray drying and (b) vacuum drying methods.} \)
As mentioned, there are two methods of production — spray and vacuum drying. However the primary treatment procedures as outlined above are the same. In spray drying, the stored liquor is spray dried, sieved and packed. The other method is to vacuum dry the stored liquor, mill the powder, and sieve before packing.
APPENDIX B

EXTRA SEM PHOTOGRAPHS
EXTRA SEM PHOTOGRAPHS

STARCH
STARCH
MALTODEXTRIN
MALTODEXTRIN
HVP

HVP1.4

10u
APPENDIX C

CALIBRATION OF SHEAR CELL
CALIBRATION DATA

Keywords to note:

**Actual Mass (g):** Represents the mass of weight as measured using the balance.

**Measured Mass (g):** Represents the force measured by the digital display unit expressed in grams.

**Actual Force (N):** Represents the calculated force from the actual mass.

**Measured force (N):** Represents the calculated force from the measured mass.

Each mass (or force) correspond to particular position on the graph as recorded by the chart recorder. These points will be used as reference points for the calibration.

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<th>Actual Force (N)</th>
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**Force = ma,** where \( m \) is the mass measured in kg, and \( a \) is the acceleration due to gravity.

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<tr>
<th>Diameter of lid:</th>
<th>5.75E-02 m</th>
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<tr>
<td>Area of lid:</td>
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Calibration Curve (Dial 1)

\[ y = 0.0996x - 0.0085 \]
CALIBRATION DATA

Keywords to note:

**Actual Mass (g):** Represents the mass of weight as measured using the balance.

**Measured Mass (g):** Represents the force measured by the digital display unit expressed in grams.

**Actual Force (N):** Represents the calculated force from the actual mass.

**Measured force (N):** Represents the calculated force from the measured mass.

Each mass (or force) corresponds to a particular position on the graph as recorded by the chart recorder. These points will be used as reference points for the calibration.

**DIAL NUMBER:** 2

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Force = ma, where m is the mass measured in kg, and a is the acceleration due to gravity.

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<th>5.75E-02</th>
<th>m</th>
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<td>Area of lid:</td>
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<td>m²</td>
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</table>
Calibration (Dial 2)

\[ y = 0.1983x \]

Actual Force (N) vs. Position of graph paper
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Calibration of Chart Trace - Dial 5

\[ y = 0.0003x^3 - 0.0183x^2 + 0.7401x \]
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Calibration of Chart Recorder - Dial 10

\[ y = 0.0028x^3 - 0.0828x^2 + 1.5318x \]
APPENDIX D

GRAPHS OF INDIVIDUAL YIELD LOCI
Yield locus for starch powder plotting mid-point data; 0%RH
### Shear Tests Consolidation - 3 kPa

Consolidation Load: 1049 g
Consolidation Stress: 396.3 N/m²

Normal Load (2) Preshear Weight Lid + Top Ring Weight of Powder Total Normal Stress (.g) (Nm²)

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<th>Height</th>
<th>Normal Load</th>
<th>Lid-Top Ring Weight</th>
<th>Total Normal Stress</th>
<th>Average Normal Stress</th>
<th>Average Shear Stress</th>
<th>Highest Value Shear Stress</th>
<th>Lowest Value Shear Stress</th>
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Twists: 10
Humidity: 0%

Average Shear Average Average Lowest Value Lowest Value Highest Value Highest Value Normal Stress Preshear Stress Shear Stress Preshear Stress Shear Stress Preshear Stress Shear Stress (Nm²) (Nm²) (Nm²) (Nm²) (Nm²) (Nm²) (Nm²) (Nm²)
Yield Locus for starch powder using mid points; 25 - 30 %RH
### Sample: Starch

**Shear Tests Consolidation - 3 kPa**

**Date:** 18th July 2002

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<th>Average Shear Stress (N/m²)</th>
<th>Average Shear Stress (N/m²)</th>
<th>Lowest Value Shear Stress (N/m²)</th>
<th>Lowest Value Shear Stress (N/m²)</th>
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Yield locus for starch powder plotting MIDPOINTS; 84 %RH
**Sample: Starch**

**Shear Tests Consolidation - 3 kPa**

**Consolidation Load:** 1049 g  
**Consolidation Stress:** 3063.06991 N/m²  
**Date:** 29th July '03 (March '03)

**Preshear Load:** 699 g  
**Preshear Stress:** 3022.738069 N/m²  
**Twist:** 10  
**Humidity:** 84 %RH

<table>
<thead>
<tr>
<th>Weight Load (g)</th>
<th>Lid + Top Ring Weight of Powder (Top Ring) g</th>
<th>Total (g)</th>
<th>Shear Normal Force (N)</th>
<th>Shear Normal Stress (N/m²)</th>
<th>Shear Normal Stress (chart units)</th>
<th>Pre shear Normal Stress (N/m²)</th>
<th>Pre shear Normal Stress (chart units)</th>
<th>Shear Stress (N/m²)</th>
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<th>Average Pre shear Stress (N/m²)</th>
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Yield Locus - Maltodextrin @ 1.5 kPa Consolidation (Low Preshear), 0 %RH.
### Shear Tests:

**Sample:** Maltodextrin  
**Consolidation Load:** 448.6 g  
**Preshear Load:** 299.3 g  
**Twist:** 5

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**Avg. Mass (top ring):** 14.6

**Shear Tests Consolidation ~ 1.5 kPa**

**Date:** 28th Oct. '02

**Humidity:** 0%
Yield Locus - Maltodextrin @ 1.5 kPa Consolidation (low preshear), 25 - 30 %RH.
60

80

100

119.6

(g)
159.5

5

1
2
3
4

1
2
3
4
5

1
2
3
4
5

I
2
3
4
5

1
2
3
4
5

8l.l
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81.1
81.1
81.1

81.1
8l.l
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81.1

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16.2

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14.7
17.2

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15.4
14.1
15.7
16.3

16.3
14.8
15.8
16.2
15.2

15.4
15.2
17.3
16.8
13.4

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257.9
257.4
254

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156.2
155.7
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157.3

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175.8
178.3

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197.4

217
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216.5
216.9
215.9

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972.4444182
959.5993871

(N/m2)

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1.543113

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1.724598
1.7491 23

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1.936494

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594.2715889

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673.6085461

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742.3672424
737.4559069
743.5006275
745.7673977

14.75
15
15.5
15
13.5

14.75
14.25
14.25
16
16.25

15
17
15.5
14.75
15

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14.75
1126.4242

15 1145.516136
14.75
1126.4242
14.75
1126.4242
16 1221.883878
17 1298.251621

(chart units) (N/m2)

Shear
Preshear
Normal Stress Stress

1496.113671 N/rn2

Preshear
Stress

Date: 2-18th Oct '02
299.2 g

2.12877 819.8152244
2.114055 814.1482989
2.123865 817.9262492
2.127789 819.4374294
2.117979
815.659479

2.51136
2.509398
2.529999
2.525094
2.49174

(N)

Shear
Normal Force

Normal Load (2)
Lid + Top Ring
Weight of Powder Total
(g)
(Top Ring)

Consolidation Stress:

Weight

Preshear Load:
Preshear Stress:
(Normal)

448.6 g

Shear Tests Consolidation- 3 kPa

1694.788524 N/rn2

Consolidation Load:

Sa mole: Maltodextrin

Shear
Stress

14-30%

6.75
7
7.5
6.75
6.25

7.75
7.75
7.75
8.25
8

8.5
8
9.5
8.25
8.75

9.75
9.5
9
8.5
8

11
10
10.25
12.5
12

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477.29839

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591 .8500036
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610.9419392

649.1258104
610.9419392
725.4935528
630.0338748
668.217746

744.5854884
725.4935528
687.3096816
649.1258104
610.9419392

840.0451664
763.677424
782.7693596
954.59678
916.4129088

(chart units) (N/m2)

Shear
Stress

Humidty:

Twists: 5

591 8537007

669.9061548

742.7450374

817.3973362

967.9864368

(N/m2)

Average Shear
Normal Stress

1126.4242

1153.15291

1179.88162

1134.060975

523.1190355

603.305165

656.7625847

683.4912945

851.5003278

(N/m2)

(N/m2)

1183.700007

Average
Shear Stress

Average
Preshear Stress

2214.66453

2291 .032272

2672.870984

2405.583886

1374.61936

(N/m2)

Lowest Value
Preshear Stress

973 6887156

1107.33265

1317.343556

1603.72259

763.677424

(N/m2)

Lowest Value
Shear Stress

2520.1355

2558.31937

2749.23873

2672.87098

154644678

(N/m2)

Highest Value
Preshear Stress

1069.148394

1221.883878

1489.170977

1794.641946

1813.733882

(N/m2)

Highest Value
Shear Stress


Yield Locus - HVPI @ 1.5 kPa Consolidation, 0 %RH (Low Preshear).
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Yield Locus - HVP1 @ 1.5 kPa Consolidation, Ambient, 25 - 30 %RH.
### Sample: HVPl

**Shear Tests Consolidation – 1.5 kPa**

**Date:** Nov. '02

**Twists:** 15

**Consolidation Load:** 648.6 g

**Pre shear Load:** 299.3 g

**Consolidation Stress:** 2450.378592 N/m²

**Pre shear Stress:** 1508.913367 N/m²

**Humidity:** 14–30%

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<th>Pre shear Stress (chart units) (N/m²)</th>
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APPENDIX E

GRAPH OF SHEAR CELL VELOCITY AS A FUNCTION OF MOTOR SETTING
Shear Cell Velocity as a function of Motor Setting

Voltage Setting (V) vs. Shear Cell Velocity (mm/min)

- • 0.075 kN/m² (Open Lid)
- ▲ 4 kN/m²
- ○ 12.5 kN/m²
- × 28 kN/m²
APPENDIX F

GRAPHS OF EFFECT OF SHEAR CELL VELOCITY ON OBSERVED STICK-SLIP FREQUENCY
Effect of Shear Cell Velocity on Observed Stick-Slip Frequency

- 0.075 kN/m² (Open Lid)
- 4 kN/m²
Effect of Shear Cell Velocity on Observed Stick-Slip Frequency

Shear cell velocity (mm/min)

Observed Frequency - # cycles per second (Hz)

Effect of Shear Cell Velocity on Observed Stick-Slip Frequency

Shear cell velocity (mm/min)

Observed Frequency - # cycles per second (Hz)
APPENDIX G

GRAPHS OF EFFECT OF SHEAR VELOCITY ON $F_{\text{max}}$
Effect of Shear Cell Velocity on $F_{\text{max}}$

![Graph showing the effect of shear cell velocity on $F_{\text{max}}$. The graph includes data points for different stress levels: 0.075 kN/m$^2$ (Open Lid), 4 kN/m$^2$, 12.5 kN/m$^2$, and 28 kN/m$^2$. The x-axis represents shear cell velocity (mm/min), and the y-axis represents $F_{\text{max}}$ (N). The data points are color-coded for each stress level.](image-url)
Effect of Shear Cell Velocity on $F_{\text{max}}$

![Graph 1: Effect of Shear Cell Velocity on $F_{\text{max}}$](image1)

- 0.075 kN/m² (Open Lid)

![Graph 2: Effect of Shear Cell Velocity on $F_{\text{max}}$](image2)

- 4 kN/m²

AG2
Effect of Shear Cell Velocity on $F_{\text{max}}$

- $F_{\text{max}}$ (N) vs. Shear Cell Velocity (mm/min)

**Top Graph:**
- Shear Cell Velocity (mm/min) ranging from 0 to 25
- $F_{\text{max}}$ ranging from 0 to 45

- Data points for 12.5 kN/m²

**Bottom Graph:**
- Shear Cell Velocity (mm/min) ranging from 0 to 25
- $F_{\text{max}}$ ranging from 0 to 70

- Data points for 28 kN/m²

AG3
APPENDIX H

GRAPHS OF EFFECT OF SHEAR VELOCITY ON $F_{min}$
Effect of Shear Cell Velocity on $F_{\text{min}}$

![Graph showing the effect of shear cell velocity on $F_{\text{min}}$. The graph includes data points for different stress levels: 0.075 kN/m² (Open Lid), 4 kN/m², 12.5 kN/m², and 28 kN/m².](image-url)
Effect of Shear Cell Velocity on $F_{\min}$

Effect of Shear Cell Velocity on $F_{\min}$
Effect of Shear Cell Velocity on $F_{\text{min}}$

Effect of Shear Cell Velocity on $F_{\text{ave}}$
APPENDIX I

GRAPHS OF EFFECT OF SHEAR CELL SPEED ON AVERAGE AMPLITUDE
Effect of Shear Cell Speed on Average Amplitude

Effect of Shear Cell Speed on Average Amplitude
APPENDIX J

GRAPH OF SHEAR MODULUS (G) AS A FUNCTION OF NORMAL STRESS (OVER A RANGE OF VELOCITIES)
Shear Modulus (G) variation with Normal Stress (over a range of velocities)
APPENDIX K

GRAPHS OF ELASTIC CONSTANT (k) AS A FUNCTION OF SHEAR CELL
Elastic Constant, k (log scale) as a function of Shear Cell Velocity and Normal Stress

Shear Cell Velocity (mm/min)

Elastic Constant, k (N/m) - log scale

- 0.075 kN/m² (Open Lid)
- 4 kN/m²
- 12.5 kN/m²
- 28 kN/m²
Change in Elastic Constant, $k$ under 0.075 kN/m$^2$ (Open Lid) as a function of velocity

Change in Elastic Constant under 4 kN/m$^2$ as a function of Shear Cell Velocity
Change in Elastic Constant, $k$ under 12.5 kN/m$^2$ as a function of Shear Cell Velocity

![Graph 1: Change in Elastic Constant, $k$ under 12.5 kN/m$^2$ as a function of Shear Cell Velocity](image1)

Change in Elastic Constant, $k$ under 28 kN/m$^2$ as a function of Shear Cell Velocity

![Graph 2: Change in Elastic Constant, $k$ under 28 kN/m$^2$ as a function of Shear Cell Velocity](image2)

AK3
APPENDIX L

GRAPHS OF STATIC COEFFICIENT OF FRICTION
Change in Static Coefficient of Friction with Shear Cell Velocity

Change in Static Coefficient of Friction with Shear Cell Velocity

0.75 kN/m²

4 kN/m²
Change in Static Coefficient of Friction with Shear Cell Velocity

- 1.4
- 1.2
- 1.0
- 0.8
- 0.6
- 0.4
- 0.2

Shear Cell Velocity (mm/min)

Change in Static Coefficient of Friction with Shear Cell Velocity

- 0.9
- 0.8
- 0.7
- 0.6
- 0.5
- 0.4
- 0.3
- 0.2
- 0.1

Shear Cell Velocity (mm/min)
APPENDIX M

GRAPH OF DYNAMIC COEFFICIENT OF FRICTION
Dynamic Coefficient of Friction under 0.075 kN/m$^2$ as a function of Shear Cell Velocity

Dynamic Coefficient of Friction under 4 kN/m$^2$ as a function of Shear Cell Velocity
Dynamic Coefficient of Friction under 12.5 kN/m$^2$ as a function of Shear Cell Velocity

![Graph showing dynamic coefficient of friction under 12.5 kN/m$^2$](image1)

Dynamic Coefficient of Friction under 28 kN/m$^2$ as a function of Shear Cell Velocity

![Graph showing dynamic coefficient of friction under 28 kN/m$^2$](image2)
APPENDIX N

GRAPH OF DURATION OF STICK – MEASURED AND CALCULATED
Comparison of the Duration of Stick - measured and calculated - log scale

- 0.075 kN/m² - Open Lid (measured)
- 4 kN/m² (measured)
- 12.5 kN/m² (measured)
- 28 kN/m² (measured)
- 0.075 kN/m² - Open Lid (Calculated)
- 4 kN/m² (Calculated)
+ 12.5 kN/m² (measured)
- 28 kN/m² (calculated)
APPENDIX O

GRAPH OF DURATION OF SLIP – MEASURED AND CALCULATED
Comparison of the Duration of Slip - measured and calculated
APPENDIX P

GRAPHS OF DYNAMIC COEFFICIENT OF FRICTION – MEASURED AND CALCULATED
Comparison of Measured and Calculated values of Dynamic Coefficients of Friction

![Graph showing comparison of measured and calculated values of dynamic coefficients of friction. The x-axis represents velocity (mm/min), ranging from 0 to 25. The y-axis represents dynamic coefficient of friction, ranging from 0 to 0.9. Different symbols and colors indicate various friction values and their sources (measured or calculated).]
APPENDIX Q

GRAPHS OF YIELD TIME – MEASURED AND CALCULATED
Comparison of Time to Initial Slip - measured and calculated using simple model

- Open Lid - 0.075 kN/m² - measured
- 4 kN/m² - measured
- 12.5 kN/m² - measured
- 28 kN/m² - measured
- Open Lid - 0.075 kN/m² - calculated
- 4 kN/m² - calculated
- 12.5 kN/m² - calculated
- 28 kN/m² - calculated

Time to yield (seconds)

Velocity (mm/min)
Comparison of Time to Initial Yield under Open Lid - 0.075 kN/m² consolidation

Comparison of Time to Initial Yield under 4 kN/m² consolidation
Comparison of Time to Initial Yield under 12.5 kN/m² consolidation

Comparison of Time to Initial Yield under 28 kN/m² consolidation

AQ3
APPENDIX R

GRAPHS OF (i) STICK-SLIP – MEASURED AND CALCULATED VALUES, AND (ii) CORRESPONDING GRAPHS SHOWING THE SLIP PHASE USING THE DYNAMIC MODEL
Actual and Theoretical Trace (Run 1: Open Lid - 0.075 kN/m² consolidation and 0.62 mm/min)

Frictional Instability - Run 1
Actual and Theoretical Trace (Run 2: Open Lid - 0.075 kN/m² consolidation and 0.77 mm/min)

Friction Instability - Run 2
Actual and Theoretical Trace (Run 3: Open Lid - 0.075 kN/m² consolidation and 0.98 mm/min)

Frictional Instability - Run 3
Actual and Theoretical Trace (Run 4: Open Lid - 0.075 kN/m² consolidation and 0.78 mm/min)

Frictional Instability - Run 4
Actual and Theoretical Trace (Run 5: Open Lid - 0.075 kN/m² consolidation and 0.83 mm/min)

Frictional Instability - Run 5
Actual and Theoretical Trace (Run 6: Open Lid - 0.075 kN/m² consolidation and 2.4 mm/min)

Frictional Instability - Run 6
Actual and Theoretical Trace (Run 7: Open Lid - 0.075 kN/m² consolidation and 2.5 mm/min)

Frictional Instability - Run 7
Actual and Theoretical Trace (Run 8: Open Lid - 0.075 kN/m² consolidation and 2.4 mm/min)

Frictional Instability - Run 8
Actual and Theoretical Trace (Run 9: Open Lid - 0.075 kN/m² consolidation and 6 mm/min)

Frictional Instability - Run 9
Actual and Theoretical Trace (Run 10: Open Lid - 0.075 kN/m² consolidation and 6.2 mm/min)

Frictional Instability - Run 10
Actual and Theoretical Trace (Run 11: Open Lid - 0.075 kN/m² consolidation and 6.07 mm/min)

Frictional Instability - Run 11
Actual and Theoretical Trace (Run 12: Open Lid - 0.075 kN/m² consolidation and 13.2 mm/min)

Frictional Instability - Run 12
Actual and Theoretical Trace (Run 13: Open Lid - 0.075 kN/m² consolidation and 15.5 mm/min)

Frictional Instability - Run 13
Actual and Theoretical Trace (Run 15: 4 kN/m² consolidation and 1.0 mm/min)

Frictional Instability - Run 15
Actual and Theoretical Trace (Run 16: 4 kN/m² consolidation and 1.2 mm/min)

Frictional Instability - Run 16
Actual and Theoretical Trace (Run 17: 4 kN/m² consolidation and 1.1 mm/min)

Frictional Instability - Run 17
Actual and Theoretical Trace (Run 18: 4 kN/m² consolidation and 3.1 mm/min)

Frictional Instability - Run 18
Actual and Theoretical Trace (Run 19: 4 kN/m$^2$ consolidation and 2.8 mm/min)

Frictional Instability - Run 19
Actual and Theoretical Trace (Run 20: 4 kN/m$^2$ consolidation and 2.7 mm/min)

Frictional Instability - Run 20

AR19
Actual and Theoretical Trace (Run 21: 4 kN/m² consolidation and 7.3 mm/min)

Frictional Instability - Run 21
Actual and Theoretical Trace (Run 22: 4 kN/m² consolidation and 7.3 mm/min)

Frictional Instability - Run 22
Actual and Theoretical Trace (Run 23: 4 kN/m² consolidation and 8 mm/min)

Frictional Instability - Run 23
Actual and Theoretical Trace (Run 24: 4 kN/m² consolidation and 16.4 mm/min)

Frictional Instability - Run 24
Actual and Theoretical Trace (Run 25: 4 kN/m² consolidation and 16.8 mm/min)

Frictional Instability - Run 25
Actual and Theoretical Trace (Run 27: 12.5 kN/m³ consolidation and 0.5 mm/min)

Frictional Instability - Run 27
Actual and Theoretical Trace (Run 28: 12.5 kN/m\(^2\) consolidation and 0.7 mm/min)

Frictional Instability - Run 28
Actual and Theoretical Trace (Run 29: 12.5 kN/m² consolidation and 0.84 mm/min)

Frictional Instability - Run 29
Actual and Theoretical Trace (Run 30: 12.5 kN/m$^2$ consolidation and 0.8 mm/min)

Frictional Instability - Run 30
Actual and Theoretical Trace (Run 31: 12.5 kN/m² consolidation and 3.4 mm/min)

Frictional Instability - Run 31
Actual and Theoretical Trace (Run 32: 12.5 kN/m² consolidation and 3.4 mm/min)

Frictional Instability - Run 32
Actual and Theoretical Trace (Run 33: 12.5 kN/m² consolidation and 6.2 mm/min)

Frictional Instability - Run 33
Actual and Theoretical Trace (Run 34: 12.5 kN/m² consolidation and 5.7 mm/min)

Frictional Instability - Run 34
Actual and Theoretical Trace (Run 35: 12.5 kN/m² consolidation and 23 mm/min)

Frictional Instability - Run 35
Actual and Theoretical Trace (Run 37: 28 kN/m² consolidation and 0.96 mm/min)

Frictional Instability - Run 37
Actual and Theoretical Trace (Run 38: 28 kN/m² consolidation and 0.9 mm/min)

Frictional Instability - Run 38
Actual and Theoretical Trace (Run 39: 28kN/m² consolidation and 1.1 mm/min)

Frictional Instability - Run 39
Actual and Theoretical Trace (Run 41: 28 kN/m² consolidation and 7.7 mm/min)

Frictional Instability - Run 41
Actual and Theoretical Trace (Run 42: 28 kN/m² consolidation and 2.5 mm/min)

Frictional Instability - Run 42
Actual and Theoretical Trace (Run 43: 28 kN/m² consolidation and 7.7 mm/min)

Frictional Instability - Run 43
Actual and Theoretical Trace (Run 45: 28 kN/m² consolidation and 19.6 mm/min)

Frictional Instability - Run 45
Actual and Theoretical Trace (Run 46: 28 kN/m² consolidation and 16.4 mm/min)

Friction Instability - Run 46
APPENDIX S

SPREADSHEET OF STICK-SLIP STUDY
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