

Experimental and Discrete Element Modelling Investigation of Flow Behaviour of Cohesive Powders under Varying Consolidation States

by

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Abstract

Granular materials find use in many industrial processes, such as mixing, coating, granulation, milling, transport, and discharge. For these processes to work efficiently and without interruptions or stoppages, it is necessary to evaluate the flowability of the powders for the design and operation of the bulk solids handling equipment. Flowability of powders is generally evaluated using the methodology proposed by Jenike, which is often referred to as the shear testing methodology. The shear testing methodology involves first shearing a powder sample under a defined normal stress to a steady or critical shear consolidation state characterized by constant shear stress and bulk density, followed by measuring the strength of the powder by shearing the powder sample until failure under a lower normal stress. However, there are many handling scenarios in industry in which the powders might not be subjected to flow regimes leading to critical shearing states, such as in dosing, powder feeders for tableting, and small-scale silos/hoppers for storage and discharge. In such scenarios where the internal shearing deformation of the powders is limited during flow, the powders may not achieve the critical consolidation conditions and instead be only partially consolidated, it is plausible that the Jenike method of flowability assessment may not apply. This study considers the possibility of powders to be partially consolidated and investigates the impact of consolidation state on powder flowability. A flowability characterization procedure to test powder specimens under varying consolidation states was developed using the Schulze Ring Shear Tester (RST). Flowability of two cohesive powders at steady and partially consolidated states were characterized using RST, and flat-bottom model silo experiments were conducted to study the influence of consolidation in powder flow. The RST results showed that the extent of shear deformation and the resultant state of consolidation impacted the yield loci and the strength of the powders, and it marginally impacted the flow function and thus the flowability, the latter finding was also confirmed by the silo experimentation results. This study shows that the powder flowability evaluated at critical consolidation is a good approximation of the flowability at partially consolidated states.

To study the particle level phenomena underlying the bulk behaviour observed during shearing and flow, this research further investigated the powder flow in the shear tests using the discrete element method (DEM). As it is computationally prohibitive to simulate powder flow problems at the real particle scale for the micron-sized particles, a meso-scale or coarse-graining approach was adopted to scale up the particle size to reduce the number of particles in the system and to simplify the particle shape representation. Prior coarse-graining studies have not been able to reproduce the experimental flow function which is obtained by conducting the shear test at varying levels of pre-shear normal stress, requiring stress history dependent/sensitive behaviour of the modelled particle system. In this thesis, the meso-scale cohesive DEM model of the shear testing was developed using the EEPA contact model to reproduce the full flow function and to study the particle level phenomena. The DEM model was calibrated using the experimental data. A fractional Design of Experiments

(DOE) study was utilised to explore the parametric space and a Multi Objective Genetic Algorithm (MOGA) was used to minimize the values of the objective functions in the calibration of the model parameters. The calibrated DEM model obtained by the adopted meso-scale and calibration approach is able to reproduce the stress history dependent flow behaviour for the critical and partial consolidation states, and the full flow function for the critical state. The likely underlying factors causing the partially consolidated powder to have lower failure strength than the critically consolidated powder were also investigated using the upscaled DEM model. The bulk density, velocity and bulk stresses were evaluated from the discrete particle data and were used to study the particle level phenomena such as shear strain evolution, and principal plane orientation in the localized zones in the powder bed. The coarse-graining technique was found to be a useful modelling tool for conserving or reproducing the key characteristics of the original system and for studying the particle level phenomena whilst decreasing the computational costs.

Lay Summary of Thesis

Powders are widely used in many industries such as pharmaceuticals, food processing, and chemicals. For these industries to run smoothly, powders must flow reliably through the various processing, handling, and storage equipment. The evaluation of powder flowability is therefore important to ensure the reliability and consistency of the industrial operations. Flowability of powders is generally evaluated using the methodology proposed by Jenike, which is often referred to as the shear testing methodology. The shear testing methodology involves first shearing a powder sample under a defined normal stress to a steady or critical shear consolidation state characterized by constant shear stress and bulk density, followed by measuring the strength of the powder by shearing the powder sample until failure under a lower normal stress.

In many industrial conditions, powders may not be critically consolidated but instead are only partially consolidated before flow initiation, hence it is not clear if the laboratory measurement of flowability under critical consolidation is still applicable to partially consolidated powders. This research studied the effect of varying consolidation levels on powder flowability, experiments were carried out on specialized equipment which allowed for testing powder flow under different levels of consolidation.

The results show that although partially consolidated powders have a lower breakage strength than critically consolidated powders, their overall ability to flow is marginally influenced by the consolidation level. This suggests that the standard Jenike testing method can provide a reasonable estimate of powder flow even if it is partially consolidated. Computer simulations were used to reproduce the experimental flow behaviour and to study the observed experimental behaviour of partially consolidated powders exhibiting lower breakage strength. The simulations successfully reproduced the experimental flow behaviour at different consolidation states and were subsequently used to examine the likely causes of the lower breakage strength exhibited by partially consolidated powders.

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List of Symbols

a	Contact patch radius	m
a_n	Scalar anisotropy	
a_o	Contact radius of the contact when $f_{JKR}^n = 0$	m
a_s	Contact area due to the presence of surface energies of the two contacting bodies	m ²
$\mathbf{a}_t$	Particle translational acceleration at time-step t	m/s ²
A	Area of compression/shearing lid	m ²
A_1	Area of plug	m ²
A_D	Cross sectional area of the lid	m ²
Bo_g	Granular Bond number	
c	Cohesion	Pa
c_n	Normal damping coefficient	N s/m
c'_n	Normal damping coefficient of CG particle	N s/m
c_t	Tangential damping coefficient	N s/m
c_{eq}	Equivalent damping coefficient of original particle system	N s/m
C	Damping coefficient	N s/m
C_b	Beverloo dimensionless coefficient	
C_c	Critical damping coefficient	N s/m
COR_{p-g}	Particle-geometry coefficient of restitution	
COR_{p-p}	Particle-particle coefficient of restitution	
d	Diameter of particle	m
d_{10}	Particle diameter at 10 th percentile	m
d_{50}	Particle diameter at 50 th percentile	m
d_{90}	Particle diameter at 90 th percentile	m
d_c	Diameter of the coarse-grained particle	m
d_{crit}	Critical hopper outlet diameter	m
d_o	Diameter of the original particle	m
D_h	Orifice hydraulic diameter	m
D_0	Plug diameter	m
e	Coefficient of restitution	
$e_{C,PP}$	Restitution coefficient of the CG particle	
$e_{O,PP}$	Restitution coefficient of the original particle	
E	Young's modulus or modulus of elasticity	Pa
E^*	Equivalent Young's modulus of particles i and j in contact	Pa
$\tilde{E}$	Adjusted Young's modulus	Pa
$\tilde{E}'$	Adjusted Young's modulus of the up-scaled particle	Pa
E_i	Young's modulus of particle i	Pa
E_j	Young's modulus of particle j	Pa
$\mathbf{g}$	Acceleration due to gravity	m/s ²
G	Modulus of rigidity	Pa
G^*	Equivalent modulus of rigidity of particles i and j in contact	Pa
G_i	Modulus of rigidity of particle i	Pa
G_j	Modulus of rigidity of particle j	Pa
h	Lifting height of cylinder	m
h_a	Arch effective thickness	m
H_{bed}	Bed height	m
$H(\theta_c)$	Arch thickness parameter	

i	Power value for EEPA mode selection	
$\mathbf{f}_i$	Sum of all the forces acting on the particle i	N
$\mathbf{f}_i^c$	Contact force acting on particle i at the c^{th} contact point	N
f_c^t	Limiting tangential friction force	N
f_o	Constant pull-off force	N
f_{min}	Maximum adhesive force	N
ff	Hopper flow factor	
ff_c	Ratio of major principal stress σ_1 and the unconfined yield strength σ_c	
$\mathbf{f}_{nCG}$	Normal contact force of CG particle	N
$\mathbf{f}_{tCG}$	Tangential contact force of CG particle	N
$\mathbf{f}_n$	Particle contact force in normal direction	N
$\mathbf{f}_t$	Particle contact force in tangential direction	N
f^n	Particle contact force magnitude in normal direction	N
f^t	Particle contact force magnitude in tangential direction	N
f_s^n	Normal spring force	N
f_d^n	Normal damping force	N
$\mathbf{f}_s^t$	Tangential spring force	N
$\mathbf{f}_d^t$	Tangential damping force	N
$\mathbf{f}_{s,t}^t$	Tangential contact force at the current time-step t	N
$\mathbf{f}_{s,t-1}^t$	Sum of all tangential force increments at the previous time-steps	N
$f_{s,t}^n$	Contact normal force at the current time-step t	N
$f_{s,t-1}^n$	Sum of all normal force increments at the previous time-steps	N
$\Delta \mathbf{f}^t$	Increment in the tangential force	N
f_{JKR}^n	JKR normal contact force	N
$\mathbf{f}_{JKR}^t$	JKR tangential contact force	N
f_o^{JKR}	Pull-off force defined as the force required to separate the two particles in contact	N
f_{hys}^n	Hysteretic spring normal force (Luding model)	N
f_{hys}	Hysteretic spring normal force (EEPA model)	N
$\mathbf{F}_{Is}$	Inter-particle contact force of scaled particle	N
$\mathbf{F}_{Io}$	Inter-particle contact force of original particle	N
$\mathbf{F}_{Bs}$	Body forces of scaled particle	N
$\mathbf{F}_{Bo}$	Body forces of original particle	N
F^c	c^{th} total contact force	N
$F_{cohesive}$	Particle cohesive force	N
F_{ij}	Fabric tensor	
F_{ij}^d	Deviatoric part of fabric tensor	
F_a	Lifting force	N
I	3 rd order identity matrix	
I_i	Moment of inertia of particle i	kg m ²
k_σ	Ratio between the major principal stress and applied normal stress	
k	Particle stiffness coefficient	N/m
k_b	Beverloo dimensionless coefficient	
$k_{n,r}$	Stiffness of original particle in the system	N/m
$k_{n,s}$	Stiffness of upscaled particle in the system	N/m
k'_n	Stiffness coefficient of CG particle	N/m
k_{eq}	Equivalent stiffness of original particle system	N/m

k_1	Loading branch stiffness	N/m
k_2	Unloading branch stiffness	N/m
k_2^{max}	Maximum unloading branch stiffness	N/m
k_{adh}	Adhesive branch stiffness	N/m
$k_{C,PP}$	Stiffness of the CG particle	N/m
$k_{O,PP}$	Stiffness of the original particle	N/m
k_{lid}	Stiffness of lid	N/m
k_n	Normal spring stiffness	N/m
k_t	Tangential spring stiffness	N/m
k'_t	Tangential stiffness of up-scaled particle	N/m
k_{series}	Equivalent stiffness of the springs of the original particle system	N/m
k_{spring}	Spring stiffness	N/m
l	Coarse-graining ratio	
$\mathbf{l}_i^c$	Vector from the centre of the particle i to the contact point	m
L	Instantaneous powder bed thickness	m
m_h	Parameter describing hopper shape	
m	Particle mass	kg
m^*	Equivalent mass of particles i and j in contact	kg
m_{bed}	Mass of powder bed	kg
m_i	Mass of the particle i	kg
m_j	Mass of the particle j	kg
m_s	Mass of scaled particle	kg
m_o	Mass of original particle	kg
m_p	Mass of particle	kg
m_{lid}	Mass of lid	kg
M_D	Torque experienced by the shearing lid during shearing	N m
$\mathbf{M}_{Is}$	Contact torque of scaled particle	N m
$\mathbf{M}_{Io}$	Contact torque of original particle	N m
$\mathbf{n}$	Unit vector in normal direction of particle contact	m
$\hat{n}_{ij}$	Unit normal vector connecting the center of CG particles	m
n_{pc}	Number of particles in contact with the particle p	
N	Normal load	kg
N_c	Total number of particle contacts	
N_{eff}	Normal load effectively transferred to the bottom	kg
N_r	Number of original particles in the system	
N_s	Number of particles in the upscaled system	
N_{tran}	Transition load	kg
O	Objective function	
P	Number of input variables	
$\mathbf{q}_i^c$	Torque acting at the particle contacts other than due to a tangential force	N m
$\mathbf{r}_i$	Position of the particle i	m
$\mathbf{r}_j$	Position of the particle j	m
r_o	Outer diameter of the lid	m
r_{in}	Inner diameter of the lid	m
r_m	Lever arm of the force	m
r_{min}	Smallest size particle in granular system	m
r_{rad}	Radial distance of the powder element	m
$\mathbf{r}_t$	Particle translational position at time-step t	m
$\mathbf{r}_{t+\Delta t}$	Particle translational position at time-step $t + \Delta t$	m

R	Particle radius	m
R'	Scaled system particle radius	m
R^*	Equivalent radius of particles i and j in contact	m
$R^{*'} $	Effective radius of up-scaled particle	m
R_i	Radius of particle i	m
R_j	Radius of particle j	m
R_r	Radius of original particle in the system	m
R_s	Radius of upscaled particle in the system	m
R_m	Mean particle radius	m
s	Shear index	
S^c	Vector connecting the particle centre with the c^{th} contact point	m
t	Time	s
t_c	Collision duration	s
$\mathbf{t}$	Unit vector in tangential direction of particle contact	m
T	Tensile strength	Pa
$\mathbf{T}_i$	Total torque acting on the particle i	N m
T_{cric}	Critical Rayleigh time-step	s
T_R	Rayleigh time step	s
$U_{Hertz}(\delta)$	Strain energy stored in a Hertzian contact	J
$U_{KE}(v)$	Energy during the collision/impact	J
$U_{spring}(\delta)$	Deformation energy of the linear spring	J
v	Relative velocity	m/s
$\mathbf{v}_n$	Particle velocity in normal direction	m/s
$\mathbf{v}_t$	Particle velocity in tangential direction	m/s
$\mathbf{v}_{rel}^t$	Relative velocity in the tangential direction	m/s
$\mathbf{v}_t^{CG}$	Translational (tangential direction) velocity of the CG particles	m/s
$\mathbf{v}_{rel}^n$	Relative velocity in the normal direction	m/s
$\mathbf{v}_t$	Particle translational velocity at time-step t	m/s
$\mathbf{v}_{t+\Delta t}$	Particle translational velocity at time-step $t + \Delta t$	m/s
$\mathbf{v}_{nCG}$	Translational velocity of CG particles in the normal directions	m/s
$\mathbf{v}_{tCG}$	Translational velocity of CG particles in the tangential directions	m/s
V_{bed}	Volume of powder bed	m ³
V_p	Volume of the particle p	m ³
V_s	Volume of multi-sphere scaled-up DEM particle	m ³
V_r	Volume of original particle in the system	m ³
V_s	Volume of upscaled particle in the system	m ³
w	Work of adhesion per unit contact area	J/m ²
w'	Work of adhesion per unit contact area of the up-scaled particle	J/m ²
W	Average mass discharge rate through the hopper orifice	kg/s
W_g	Weight of particle	N
α_t	Particle angular acceleration at time-step t	rad/s ²
β	Damping constant	
$\beta(l)$	Coarse graining factor	
β^*	Angle between the normal to the wall and the principal direction at the hopper wall	rad
β_n	Normal dashpot coefficient	N s/m
β_t	Tangential dashpot coefficient	N s/m
β_{DD}	Angle of repose in draw down experiment	rad
γ	Shear strain	
γ_n	Damping parameter	

γ'_n	Damping parameter of up-scaled particle	
γ_t	Tangential spring stiffness	N/m
γ^*	Contact surface energy	J
γ_o	Viscous damping coefficient	N s/m
γ_1	Surface energy of particle i	J
γ_2	Surface energy of particle j	J
γ_{12}	Interface energy of particles i and j in contact	J
γ_n	Shear strain on x plane	
γ_{yx}	Shear strain on yx plane	
δ	Particle normal overlap	m
δ_{CG}	Normal overlap of the contacting CG particles	m
δ_{eff}	Effective angle of friction	rad
δ_{max}	Maximum particle overlap	m
δ_{max}^*	Plastic flow limit overlap	m
δ_{min}	Overlap at f_{min}	m
δ_n	Normal overlap	m
δ_o	Plastic deformation (Luding model)	m
δ_p	plastic overlap (EEPA model)	m
δ_s	Particle overlap of scaled particle	m
δ_{series}	Equivalent stiffness of particles	N/m
δ_o	Particle overlap of original particle	m
δ_{tCG}	Tangential overlap of the contacting CG particles	m
Δf^n	Change in the normal force	N
Δt	Time-step	s
$\Delta \gamma$	Adhesion energy	J
$\Delta \delta_n$	Normal displacement increment	m
$\Delta \delta_t$	Tangential displacement increment	m
ε	Normal strain	
ε_n	Normal strain on x plane	
ε_r	Porosity of rice grains	
ε_s	Porosity of the multi-sphere scaled-up DEM rice particle	
ε_{xx}	Normal strain on x plane	
$\varepsilon_{x,pre}$	Ratio of the compression in the powder bed during pre-shearing to original bed height	
ε_{yy}	Normal strain on y plane	
ζ	Damping ratio	
η	Damping coefficient	N s/m
θ	Angle of inclination of stress plane	rad
θ_{ang}	Instantaneous or incremental angular displacement	rad
θ_c	Inclination of the hopper wall with the vertical	rad
θ_t	Particle angular position at time-step t	rad
$\theta_{t+\Delta t}$	Particle angular position at time-step $t + \Delta t$	rad
θ_t	Twisting angle	rad
κ	Fraction of the steady state shear stress	
λ	Scale factor	
λ_p	Plasticity ratio	
μ	Coefficient of static friction	
$\mu_{C,PP}$	Sliding friction coefficient of the CG particles	
$\mu_{O,PP}$	Sliding friction coefficient of the original particles	
$\mu_{r\ p-g}$	Particle-geometry coefficient of rolling friction	

$\mu_{r\ p-p}$	Particle-particle coefficient of rolling friction	
$\mu_{s\ p-g}$	Particle-geometry coefficient of static friction	
$\mu_{s\ p-p}$	Particle-particle coefficient of static friction	
ν	Poisson's ratio	
ν_i	Poisson's ratio of particle i	
ν_j	Poisson's ratio of particle j	
ξ	Tangential spring length or deformation	m
Π	Dimensionless number	
ρ_b	Bulk density	kg/m ³
$\rho_{b,crit}$	Critical bulk density	kg/m ³
ρ_s	Solids density	kg/m ³
ρ'_s	Solid density of the up-scaled particle	kg/m ³
ρ_s^r	Density of original particle in the system	kg/m ³
ρ_s^S	Density of upscaled particle in the system	kg/m ³
σ	Normal stress	Pa
σ_1	Major principal stress	Pa
σ'_1	Stress required to support a stable arch	Pa
$\sigma_{1,crit}$	Critical consolidation stress	Pa
σ_{1silo}	Consolidation stress at the hopper outlet in the silo experiment	Pa
σ_2	Minor principal stress	Pa
σ_c	Unconfined yield strength	Pa
σ_{cJ}	Jenike yield strength	Pa
σ_{cM}	Miccio yield strength	Pa
$\sigma_{c,crit}$	Critical unconfined yield strength	Pa
σ_n	Normal stress on plane	Pa
σ_{pre}	Pre-shear normal stress	Pa
σ_{sh}	Normal stress at incipient shearing	Pa
σ_{tran}	Transition stress	Pa
σ_w	Wall normal stress	Pa
σ_x	Normal stress on x plane	Pa
σ_y	Normal stress on y plane	Pa
τ	Shear stress	Pa
τ'	Partial consolidated shear stress	Pa
τ_n	Shear stress on plane	Pa
τ_{pre}	Pre-shear shear stress	Pa
τ_{sh}	Failure shear stress at shear to failure	Pa
τ_w	Wall shear stress	Pa
τ_{yx}	Shear stress on yx plane	Pa
φ_e	Effective angle of internal friction	rad
φ_f	Dimensionless plasticity depth	
φ_{lin}	Slope of the linearised yield locus	rad
φ_x	Wall friction angle	rad
Φ_{DD}	Shear angle in draw down experiment	rad
ψ	Principal strain and stress angle	rad
ω	Angular velocity	rad/s
ω^{CG}	Angular velocity of the CG particles	rad/s
ω_i	Angular velocity of the particle i	rad/s
ω_n	Natural frequency	rad/s
ω_o	Rotational velocity of original particle	rad/s
ω_s	Rotational velocity of scaled particle	rad/s

ω_t	Particle angular velocity at time-step t	rad/s
$\omega_{t+\Delta t}$	Particle angular velocity at time-step $t + \Delta t$	rad/s
ω_{tr}	Trough angular velocity	rad/s

Abbreviations

3-D	Three dimensional
AdHys	Adhesive hysteretic rolling resistance
AOR	Angle of repose
CED	Cohesion energy density
CFD	Computational fluid dynamics
CG	Coarse-grained
DEM	Discrete element method
DOE	Design of experiments
DYS	Dynamic yield strength
EEPA	Edinburgh Elasto-Plastic Adhesive contact
EPSD2	Elastic-plastic spring-dashpot
JKR	Johnson Kendall Roberts
MELS	Modified Extensible Lattice Sequence
MEPA	Macro Elasto-Plastic Adhesion model
MOGA	Multi-Objective Genetic Algorithm
OFAT	One factor at a time
PSD	Particle size distribution
SED	Surface energy density
SEM	Scanning electron microscopy
SF	Static friction
SJKR	Simplified Johnson Kendall Roberts
SUP	Scaled-Up Particle model

This thesis is dedicated to my loving Grandparents

Smt. Shakuntla Sharma & Sh. Om Datt Sharma

and

Smt. Prem Lata Sharma & Sh. Rishi Kesh Sharma

For their endless love, belief, encouragement, and invaluable teachings throughout my life

Chapter 1 Introduction

1.1 Background

Granular materials can be defined as a collection of discrete or independent solid particles (Nedderman, 1992), where the particle size can range from the nanometer to the centimeter scale (Yu, 2005). The granular materials encompass a wide variety of materials, ranging from coarse materials extracted during mining operations to powders composed of fine individual particles in the pharmaceutical sector. Granular materials are ubiquitous in many industries across diverse sectors, such as pharmaceuticals, food processing, and chemicals, and are therefore used in a variety of industrial processes, including granulation, milling, mixing, conveying, storage, and discharge. For ensuring smooth and efficient operation of these processes, it is imperative to assess the flowability of powders to design and properly operate the bulk solids handling equipment. Powder flowability can be generally defined as the ability of the powders to flow. Powder flow can be defined as the relative movement of the powder particles amongst other particles or against the surface of the container (Garg et al., 2022). Powder flow properties can be characterized using various tests/methods, such as angle of repose, bulk density ratios, powder rheometry tests, and shear testing, among others (Barletta et al., 2019). Barletta et al. (2019) classified the methods available to determine the flow properties into three broad categories; static methods, dynamic methods, and defined consolidation methods. The static methods measure the properties of the powder at the end of the sample preparation process, where the process involves powder flow. Examples of such methods include measurement of the static angle of repose and determining the bulk density ratios, such as Hausner ratio. The dynamic methods measure the properties of the powder which are attained during the dynamic flow conditions during a forced or imposed flow, for example, by measuring the time taken for a powder to flow through an orifice or determination of the flow energy of the powder by the FT4 powder rheometer. The defined consolidation methods involve the consolidation and failure steps. In these flow methods, quasi-static deformations or very slow flow conditions are established, to correspond to the flow initiation conditions in powder handling and storage equipment. The consolidation step includes consolidating the powder sample under defined stress conditions; the failure step follows the consolidation step, in which the strength of the powder or its resistance to flow is measured. For design and subsequent operation of the powder handling equipment, the powder's flowability is generally evaluated using the methodology proposed by Jenike (1964), which is often referred to as shear testing, in which the flow properties are evaluated at defined consolidation states.

The Jenike shear testing methodology used to design reliable mass flow silos assumes the powder to be at the steady state during flow and therefore the two-stage shear testing method where the sample is first critically consolidated followed by measurement of its strength, is considered representative of the flow conditions encountered in silo discharge. In shallow containers and other situations where the internal shear

of the powder is limited during flow, the powder may not achieve steady state conditions. There are many handling scenarios in industry in which such a situation may occur, such as in dosing, intermittent powder flow, filling of powders in pharmaceutical and food industries, small-scale silos/hoppers for storage and discharge, powder feeders for tableting, and powder spreading process used in additive manufacturing techniques. Handling scenarios where the powder might not achieve the steady state deformation conditions may be classified as a partially consolidated state or partially consolidated flow. In such flow conditions the flow has not yet fully developed, and therefore, shear stress and bulk density at flow are not constant with time.

To study the particle level phenomena underlying the bulk behaviour observed during shearing and flow, the powder flow can be investigated using the discrete element method (DEM) introduced by Cundall and Strack (1979). The DEM simulations help to model granular flow problems at the particle scale. The granular material in DEM is represented as discrete or distinct elements or particles; the particle contacts are detected and the contact forces acting during the particle interactions are determined by utilizing suitable contact models. Using Newton's laws of motion, the accelerations, velocities and the positions of the particles are thus determined after accounting the net force acting on each individual particle. Although, DEM enables modelling the particle interactions at the real particle scale, it is still prohibitively expensive to model granular systems having average size of particles below 200 μm , as a larger number of particles in the system leads to a higher number of detected contacts, leading to a greater number of calculations for computing the forces. The computational resources available make modelling the particles at this real scale to be time consuming and prohibitively expensive. In addition, real particles often do not have a consistent repeatable shape which can be modelled in DEM, moreover, the surface roughness and asperities are hard to model. Therefore, to model granular systems having particles in the micron range, the mesoscopic scale approach is often used where each DEM particle is a representation of an intermediate length scale between the actual particle size and the bulk scale. A coarse-graining or meso-scale approach scales up the particle size and simplifies the particle shape to represent the collection of smaller particles which helps reduce the number of particles in the system enabling modelling fine particle systems. This helps in studying the particle level phenomena at the mesoscopic scale, whilst conserving or reproducing the key characteristics of the original system and lowering computational costs.

1.2 Research scope and objectives

The research carried out in this PhD thesis aims to develop a new understanding of the effect of consolidation state on the flow properties of cohesive powders and to develop a systematic flow characterization method for partially consolidated powders, thereby enabling study of powder flow behaviour over a wide range of consolidation states ranging from critical to partial consolidation encountered in bulk solids handling. Further, this study aims to develop and calibrate a meso-scale cohesive DEM model of shear testing and employ it to study the microscopic phenomena underlying the macroscopic stress history dependent flow behaviour observed in the experiments.

The objectives of this thesis are as follows:

- To study the influence of consolidation on the flowability of cohesive powders, and to develop a method to evaluate flowability of partially consolidated powders using shear testing.
- To study the powder flow in model silo experiments to investigate the influence of consolidation on flowability.
- To develop a meso-scale cohesive DEM model to reproduce the experimental powder flow behaviour and employ it to study the underlying microscopic phenomena.

1.3 Structure of the thesis

The thesis aims at presenting and discussing the work to fulfil the objectives and is divided into six chapters, a brief description of the contents of these chapters is outlined below.

Chapter 1 presents the brief background of the research carried out in the thesis, together with the research scope and the objectives. A brief overview of the content of the constituent thesis chapters is also given.

Chapter 2 reviews the literature relevant to the work carried out in this thesis. This chapter first focuses on the Jenike shear testing methodology and the relevance of the steady state, followed by the discussion on the relevance, importance, and need of testing powders at partial consolidation. The second part of this chapter briefly discusses the fundamentals of DEM followed by reviewing the contact models for modelling the particle interactions. The chapter then reviews the literature addressing the DEM modeling of shear testing and the need for developing a model able to predict the full stress history dependent flow behaviour.

Chapter 3 presents the flowability characterization of powders under varying consolidation states, the use of coaxiality hypothesis in determining the stress state at partially consolidated states is discussed. The chapter then presents and discusses the effect of consolidation state on the yield strength and flow function of two cohesive powders, with the experiments carried out on the shear cell. The chapter then presents the

experimental setup and testing methodology of the model silo experiments carried out to study and verify the effect of consolidation state on the powder flowability.

Chapter 4 presents the meso-scale cohesive DEM model development of shear testing using shear cell experiments. The simulation setup details are discussed, followed by discussion on the model parameters used for the parametric sensitivity study and model calibration. The chapter then presents the identification of the sensitive model parameters and their role in development of the DEM model, followed by the calibration of the model. The calibrated model parameters are then used to simulate the full shear test at varying consolidation loads with the effectiveness of the model checked by comparison with the corresponding experiments. The validation study of the calibrated model carried out by simulating partial consolidation state is then presented.

Chapter 5 presents the upscaling of the calibrated shear cell cohesive DEM model with the effectiveness of upscaling verified with the comparison with the calibrated model. The upscaled model is utilized to investigate the likely underlying factors behind the partially consolidated powder having lower failure strength than the critically consolidated powder. The model is then used to study particle level phenomena such as shear strain evolution, and principal plane orientation by evaluating the bulk density, velocity, and bulk stresses from the discrete particle data.

Chapter 6 summarizes the main conclusions drawn from this doctoral work and puts forward recommendations for future research.

Chapter 2 Literature Review

2.1 Introduction

This chapter reviews the relevant literature necessary to understand the background of the work carried out in this thesis. The literature review presented in this chapter aims to identify knowledge gaps in the current literature and justifies the need to address them. The chapter is divided into two parts; the first part addresses the research pertaining to the experimental flowability characterization of powders, specifically discussing and reviewing the Jenike shear testing methodology. The second part of this chapter addresses the research carried out to model powder systems using the discrete element modeling (DEM) methodology.

2.2 Review of Jenike shear testing methodology

2.2.1 Introduction to Jenike shear testing methodology

The Jenike shear testing methodology evaluates the yield strength of the powder after consolidating the powder to a critical state. Shear testing, based on principles as first established by Jenike, (1964) and later elucidated by Schulze (2008), approaches powder flow testing with a two-stage procedure, in which the first stage is used to consolidate the powder, and the second stage to measure its strength. Powder consolidation in the first measurement stage (pre-shear stage) is carried out by shearing the powder under defined normal stress until it reaches the steady state (critical state) condition characterized by constant powder volume and shear stress values. After stopping the pre-shear, the powder strength is measured in the second stage (shear stage), in which the powder is sheared under lower normal stress. The Jenike shear testing methodology gives a numerical definition to powder flowability and defines a method to measure it. This methodology has been widely used to measure the flowability of powders, as it has been successfully utilized to design silos and hoppers to ensure adequate flow of powders with varied flowability and for a variety of industrial applications. The shear testing methodology forms the broad basis of testing in many commercial testers such as the Schulze Ring Shear Tester (Schulze and Wittmaier, 2003), the Brookfield Powder Flow Tester (Berry et al., 2015), the Anton Paar Shear Cell (Zinatlou Ajabshir et al., 2024), and the Freeman FT4 Rheometer Shear Cell (Stavrou et al., 2020). The shear test and the resultant flow function are often used to characterize the flowability of powders. The Schulze Ring Shear Tester (Barletta et al., 2019; Schulze, 2008) is one of the most widely used equipment for conducting shear testing experiments to determine the powder flow function and quantify the powder's flowability.

The powder flow from the silo or hopper outlet can be broadly classified as mass flow or funnel flow. During mass flow, all the powder particles are in motion or are mobilized such that the complete bulk of the powder is in motion. On the contrary, during funnel flow, stagnant zones near the silo wall are formed such that only a central core or portion of the powder bulk is in motion. Flow obstructions such as arching and ratholing can lead to flow stoppages and therefore are not desirable, arching can be defined as the development of a stable

arch or bridge across the silo/hopper outlet which stops the powder flow. Ratholing is the development of permanent stagnant zones near the silo walls where the powder does not flow or is blocked even after the powder from the central flowing core/channel has emptied, therefore the silo is not emptied completely. Jenike's seminal work (Jenike, 1964) on powder flowability and silo design provides a criterion for designing arch free mass flow silos and rathole free funnel flow silos. Jenike's approach for designing silos is based on the assumption of a radial stress field in the hopper which means that the major principal stress in the converging hopper part of the silo is proportional to the distance from the virtual hopper apex. The major principal stress is a function of the hopper inclination angle, effective angle of internal friction and the wall friction angle. To represent the stress state in the hopper section, Jenike derived partial differential equations considering equilibrium of an infinitesimal volume element in the hopper. A solution for these equations only exists for specific combinations of the hopper inclination, effective internal friction and wall friction angles, meaning the mass flow will only take place at these specific parametric combinations. If no solution exists for the equations, it means that the wall friction along the hopper wall is not mobilized leading to powder not moving along the hopper wall, a stagnant zone will be formed leading to funnel flow. Thus, depending on the internal friction and wall friction properties, silos can be designed to ensure mass flow or funnel flow by varying the hopper inclination. Further, critical outlet dimensions of the hopper can be determined to either ensure arch free flow in mass flow silos and arch and rathole free flow in funnel flow silos.

Jenike designed the shear testing procedure to replicate in the experiment the assumptions he made about powder flow to design mass flow hoppers, that is, hoppers in which the powder is fully mobilized throughout the volume. It was hypothesized that under steady flow in a converging hopper, the powder is continuously deforming under shear. Although the local stress experienced by the flowing powder changes as the material crosses or passes through changing cross-sections of the hopper, the powder can be considered to be at the critical state in every position. Furthermore, according to Jenike, a stable arch forms stopping the flow when the maximum bearing stress in the powder arch equals the unconfined yield strength of the powder. Assuming that the powder has gained its strength while flowing in the hopper, the relevant major principal stress for consolidation is the one experienced at the stable arch position during critical state flow. Therefore, it is the strength that would be measured in the shear tester if the major principal stress in the pre-shear phase matches with the one occurring in the hopper at the arch position. Since in the design procedure the arch position is not known a priori, to find the right major principal stress and powder strength, the Jenike design procedure uses the experimental function that correlates the powder strength with the major principal stress occurring in the powder in the pre-shear stage, which is called the flow function and is a key representation of powder flowability relevant to mass flow hopper design. The flow function is the result of two independent theoretical analyses of the stress distribution in a static arch and in a flowing hopper. To solve the design problem, the powder flow function is combined with the hopper flow factor, another function that relates the maximum stress on the static arch to the major principal stress during powder flow.

Jenike's shear testing methodology provides a robust method to design mass flow and funnel flow silos; the powder properties derived from shear testing are used for this purpose. The following section discusses the shear testing methodology proposed by Jenike to determine the yield locus, the effective angle of internal friction, and the powder flow function necessary for the design of silos. The subsequent section after the next section would discuss the design of mass flow silos by using the properties determined from shear testing.

2.2.2 Jenike shear testing methodology and relevance to silo design

The powder sample being tested is first brought to a base state (steady state) where it is assumed to be relieved of its previous stress history, and therefore, it can be considered a reproducible state. This step is referred to as the pre-shear stage, as shown in Figure 2-1. The powder sample is subjected to a constant pre-shear normal stress during shearing. The sample is sheared until constant shear stress and bulk density are achieved. This state of the powder is called the "steady state", and the sample is "critically consolidated". The coordinates of the pre-shear point on the normal stress σ – shear stress τ plane represent the pre-shear normal stress, σ_{pre} , and the corresponding shear stress, τ_{pre} , at the steady state. After the critical consolidation, the sample is relieved from shearing by bringing the shear stress down to zero. Then, the sample is subjected to a normal stress σ_{sh} lower than the pre-shear normal stress and sheared until "incipient flow/failure", where the shear stress τ_{sh} reaches a maximum (peak value) and then decreases. The normal and shear stress values at the incipient flow point are recorded and used as coordinates for the shear point on the $\sigma - \tau$ plane. This point is one of the curve's points representing the shear stress that, under the assigned normal stress, can be applied to the powder to obtain an internal shear. Such a curve is named "the yield locus". This procedure of critically consolidating the sample and shearing until failure under normal stresses lower than the pre-shear normal stress is repeated to obtain additional shear points of the yield locus. The Mohr circle (Parry, 2004) representing the state of stress at steady state is used to calculate the major principal stress, σ_1 , during consolidation. The Mohr circle representing the state of stress in an unconfined material (the minor principal stress is equal to zero) is used to calculate the unconfined yield strength, σ_c . The stresses σ_1 and σ_c would be the normal consolidation stress and the strength measured in a uniaxial compression test. The yield locus is unique to the applied pre-shear normal stress, and several yield loci are plotted to obtain different values of (σ_1, σ_c) by changing the applied pre-shear normal stresses. The (σ_1, σ_c) values are then plotted on the $\sigma_1 - \sigma_c$ plane to obtain the flow function curve, this curve exhibits a positive slope, meaning that the unconfined yield strength increases with increasing consolidation stress. The function relating the unconfined yield strength to the major principal stress occurring during consolidation is the flow function, $\sigma_c = f(\sigma_1)$.

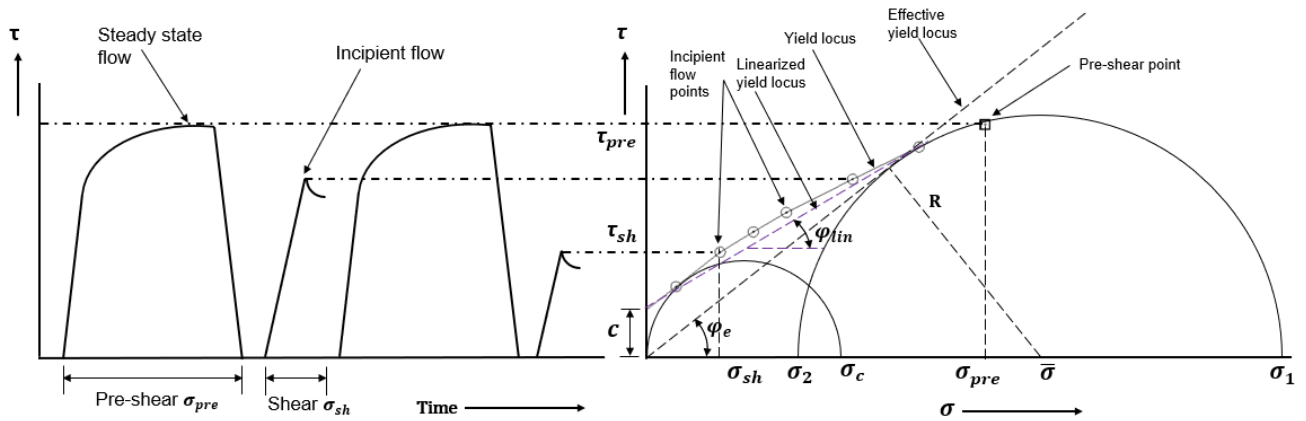


Figure 2-1: Shear test to determine yield loci and powder flowability (adapted from Schulze (2008)).

The stresses σ_1 and σ_c and their relevance can be understood by the uniaxial compression test. In the uniaxial compression test as described in Figure 2-2, the powder sample is filled into a hollow cylinder with cross-sectional area A , assuming that internal walls of the cylinder are frictionless. The powder sample is loaded or compressed with a vertical stress σ_1 known as the consolidation stress or major principal stress. After consolidating the powder specimen and relieving the applied consolidation stress, the hollow cylinder is removed. Then the consolidated specimen is loaded with increasing vertical stress, till the specimen breaks or fails. This stress at failure σ_c is called unconfined yield strength or compressive strength. The bulk solid specimen fails or yields at a particular stress σ_c , which is dependent on the prior stress history of the powder which in turn depends on the consolidating stress σ_1 . Usually, greater consolidation stress σ_1 leads to greater unconfined yield strength σ_c . The powder consolidation affects also the bulk density of the material or the particle packing in the specimen. Assuming coaxiality in the major principal stress direction during consolidation and yield in the shear testing, as well as a steady state consolidation in the uniaxial compression test, the $\sigma_1 - \sigma_c$ values obtained from the shear test are analogous to the corresponding values obtained from the uniaxial compression.

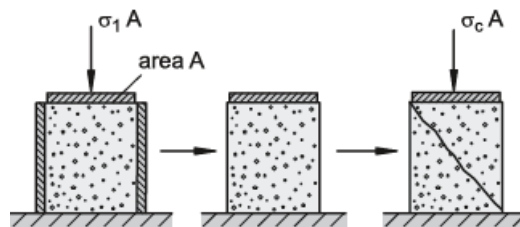


Figure 2-2: Uniaxial compression test (Schulze, 2008).

As discussed above, the Jenike methodology to gauge the powder flowability assumes that the powder achieves the steady state in the hopper/silo at the time of flow, and therefore the pre-shear stage is relevant to this flow condition. The Mohr circles at steady state flow at changing consolidation stress can be considered together, and their envelope, well represented by a line passing through the origin of the $\sigma - \tau$ plane, can be referred to as the “effective yield locus” (Jenike, 1964, 1961). This envelope of the steady-state Mohr circles

is relevant to the calculation of the stress distribution in the hopper during discharge, assuming that the material is everywhere at steady state during discharge. The slope angle of the effective yield locus, δ_{eff} , is referred to as “effective angle of friction”. The ratio of the major and minor principal stresses at steady state flow can be calculated using δ_{eff} as $\frac{\sigma_1}{\sigma_2} = \frac{1 + \sin \delta_{eff}}{1 - \sin \delta_{eff}}$, Jenike (1964, 1961) reported that this ratio and the resulting δ_{eff} values vary slightly with the consolidation stress for a given bulk solid operating under constant temperature and moisture content. Multiple texts relating to bulk powder technology (Barletta et al., 2019; Roberts, 2005; Schulze, 2008) consider the effective yield locus at individual consolidation stresses and thus calculate the effective angle of internal friction φ_e at varying σ_1 , the line passing through the origin of the $\sigma - \tau$ plane and tangent to the Mohr circle at steady state is defined as the effective yield locus, and this definition of effective yield locus is adopted in this thesis. As the major principal stress σ_1 during flow can vary; therefore, the effective angle of internal friction can be determined at the varying σ_1 values and used in silo design, φ_e can be considered as a quantification of the internal friction at steady state flow. Powder discharge from the hopper is assumed to take place at steady state, therefore, in practical design applications, the results of the flow at steady state are used to design the silo and calculate the hopper inclination angle used to determine the transition between mass and funnel flow. For this purpose, calculation of the hopper stresses and the mass flow/ funnel flow design procedures requires also the determination of the wall friction angle. The wall friction angle is determined through a dedicated test on the shear tester by allowing shearing of powder samples over the silo wall coupons. The wall friction test is conducted by moving the powder material relative to the wall material. The wall friction coefficient is defined as the ratio of the wall shear stress τ_w required to move the powder against the wall material with the applied wall normal stress σ_w . The wall friction angle is calculated by the arctangent of the wall friction coefficient.

The size of an arch-free silo opening is designed assuming the powder to be critically consolidated during the time of flow, and therefore the powder properties measured in the shear stage of the two-stage powder flow test are considered relevant to this. The powder’s unconfined yield strength, considered a function of the major principal stress during consolidation, is the powder property derived from shear tests and used in the design procedure along with the effective angle of internal friction and the flow function.

2.2.3 Silo design

The flowability tests conducted on the Schulze RST help obtain the flow function, internal friction and bulk density of the powder sample, together with the wall friction angle these parameters can be used to design the silos. The silo can be designed according to Jenike’s design methodology for ensuring mass flow such that the minimum outlet size and slope of the hopper section are determined.

Jenike (1961) hypothesized that the stresses in the hopper section of the mass flow silo are influenced by the distance from the virtual hopper vertex such that a radial stress field exists within the hopper section of the

silo. In the radial stress field, the stress intensity is a function of the radial coordinate. The stress state is dependent on the effective angle of internal friction φ_e , the wall friction angle φ_x and the slope or inclination of the hopper wall with the vertical θ_c . Jenike (1964) provided diagrams with mass flow boundaries separating the mass flow and funnel flow regions from which hopper inclination required for ensuring mass flow at particular values of φ_e and φ_x can be determined. Mass flow occurs at specific combinations of φ_e , φ_x and θ_c , requiring adequate slope and smoothness of the hopper wall to mobilize wall friction, else stagnant zones close to the wall will form and funnel flow would occur. Roberts (2005) provided Equation (2-1) to determine the hopper inclination at the bounds or limits between the mass and funnel flow.

$$\theta_c = \frac{\pi}{2} - \frac{1}{2} \cos^{-1} \left(\frac{1 - \sin \varphi_e}{2 \sin \varphi_e} \right) - \frac{1}{2} \left[\varphi_x + \sin^{-1} \left(\frac{\sin \varphi_x}{\sin \varphi_e} \right) \right] \quad (2-1)$$

Where, $\frac{1}{2} \left[\varphi_x + \sin^{-1} \left(\frac{\sin \varphi_x}{\sin \varphi_e} \right) \right]$ can be defined as β^* , that at the wall is the angle between the normal to the wall and the principal direction.

For designing conical hoppers, it is advised to reduce θ_c by 3° from the limiting value at mass flow/ funnel flow boundaries as a safety factor to ensure mass flow (Roberts, 2005). The next step of silo design involves determining the minimum dimension of the hopper outlet to avoid formation of a cohesive arch.

Formation of a cohesive arch in the hopper results in a force being transferred to the hopper walls. This force is a result of the weight of the bulk solid in the arch and results in a stress σ'_1 which is the stress acting in the arch and is required to support this arch. Jenike (1961) idealized the hopper arch as a self-supporting dome and assumed the mass above the dome does not transmit load to it (Schulze, 2008), resulting in no stress between the arch and the material above. Jenike (1964) defined a parameter called hopper flow factor ff which is defined as a ratio between the major principal stress σ_1 and the stress acting in the arch σ'_1 with $ff = \frac{\sigma_1}{\sigma'_1}$. The flow factor is a function of θ_c , φ_x and φ_e with it describing the stress conditions in the hopper during flow. Jenike (1964) provided diagrams of the flow factors with the mass flow boundaries for ensuring no arching flow condition for the conical and wedge hoppers for the varying values of θ_c , φ_x and φ_e . For the flow factor to be calculated more conveniently, Arnold et al. (1982) and Arnold and McLean (1976) provided equations which find mention in Roberts (2005). The flow factor ff can be determined from Equation (2-2). X and Y can be determined from Equation (2-3) and Equation (2-4) respectively. The arch thickness parameter $H(\theta_c)$ can be determined from Equation (2-5), $m_h = 1$ for axi-symmetric flow channels, therefore for conical hoppers, $m_h = 1$ to calculate X, Y and $H(\theta_c)$.

$$ff = \frac{Y (1 + \sin \varphi_e) H(\theta_c)}{2(X - 1) \sin \theta_c} \quad (2-2)$$

$$X = \frac{2^{m_h} \sin \varphi_e}{1 - \sin \varphi_e} \left[\frac{\sin(2\beta^* + \theta_c)}{\sin \theta_c} + 1 \right] \quad (2-3)$$

$$Y = \frac{[2(1 - \cos(\beta^* + \theta_c))]^{m_h} (\beta^* + \theta_c)^{1-m_h} \sin \theta_c + \sin \beta^* \sin^{1+m_h}(\beta^* + \theta_c)}{(1 - \sin \varphi_e) \sin^{2+m_h}(\beta^* + \theta_c)} \quad (2-4)$$

$$H(\theta_c) = \left[\frac{(130 + \theta_c)}{65} \right]^{m_h} \left[\frac{(200 + \theta_c)}{200} \right]^{1-m_h} \quad (2-5)$$

For uninterrupted flow in the hopper, the formation of the arch should be avoided which can stop the flow. The arch will be stable if the unconfined yield strength σ_c of the powder is more than the stress σ'_1 in the arch. For conical hoppers there exists a critical hopper outlet diameter d_{crit} where $\sigma_c = \sigma'_1$, a diameter larger than this d_{crit} value will lead to the major stress σ'_1 in the arch to be more than σ_c and thus arching to be avoided. The steps followed for silo design to ensure mass flow are listed as follows:

1. Calculation of hopper inclination angle θ_c for mass flow (θ_c value is reduced by 3° for use in next step)
2. Calculation of ff from θ_c , φ_x and φ_e .
3. The line of the major stress in the arch $\sigma'_1 = \frac{\sigma_1}{ff}$ is plotted along with the flow function curve. The principal stress value σ_1 where these two curves intersect is used to calculate φ_x and φ_e , these values are used for the next iteration to calculate ff .
4. Step 3 is iteratively repeated until the σ_1 and ff values stabilize, and there is no significant change in these values with further iterations.
5. This stabilized σ_1 value is called the critical consolidation stress $\sigma_{1,crit}$, σ'_1 value at $\sigma_{1,crit}$ is calculated and called the critical unconfined yield strength $\sigma_{c,crit}$. The critical bulk density $\rho_{b,crit}$ is calculated at $\sigma_{1,crit}$ to calculate the critical hopper outlet diameter d_{crit} (Equation (2-6)) to ensure mass flow.

$$d_{crit} = H(\theta_c) \frac{\sigma_{c,crit}}{\rho_{b,crit} * g} \quad (2-6)$$

2.2.4 Flow function as a tool to gauge flowability

The yield strength σ_c of the powder can be impacted by the moisture and temperature it is subjected to during flow, and its storage time in the silo or bulk solid equipment before flow. If a powder flows continuously or instantaneously from a silo, the temperature and humidity can be assumed to be constant and as it is a continuous flow, therefore the storage time or consolidation time of the powder is zero. Under such conditions, the yield locus and the yield strength become a function of the major principal stress during consolidation σ_1 such that $\sigma_c = f(\sigma_1)$. The relation $\sigma_c = f(\sigma_1)$ is called the instantaneous flow function or just the flow function of the powder, flowability of the powder can be characterized by this function. The ratio ff_c of major principal stress σ_1 and the unconfined yield strength σ_c can be used to numerically characterize and quantify the flowability. Jenike (1964) classified the flowability or flow behaviour into different regions based on ff_c , which was later extended by Thomas and Schubert (1979) to yield the classification as listed in Table 2-1.

Table 2-1: Powder flow classification into different regions based on ff_c .

$ff_c < 1$	Not flowing
$1 < ff_c < 2$	Very cohesive
$2 < ff_c < 4$	Cohesive
$4 < ff_c < 10$	Easy-flowing
$10 < ff_c$	Free-flowing

This classification helps to compare the flowability of a powder under varying consolidation stress σ_1 and to compare the flowability of two powders at a particular consolidation stress σ_1 . The flow function is obtained by the shear test as discussed earlier by computing the unconfined yield strength σ_c at the varying consolidation stresses σ_1 . As seen in Figure 2-3, the powder B transitions from the very cohesive to cohesive region as the major principal stress increases, powder A has a poorer flowability than powder B as its unconfined yield strength is higher at comparable major principal stresses.

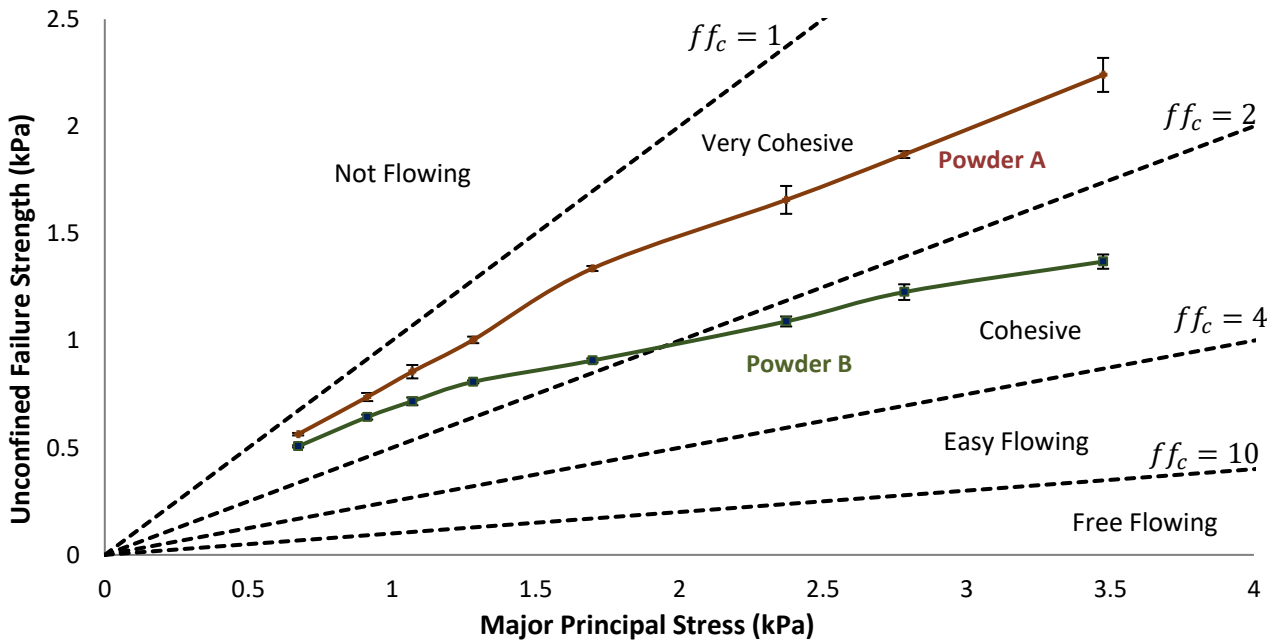


Figure 2-3: Lines of constant flowability dividing the $\sigma_1 - \sigma_c$ plane into different regions, and flow function curves of powders A & B.

Jenike's assumption of the powder attaining steady state during flow is widely accepted as robust since the bulk solid undergoes considerable deformation and shearing in an industrial silo to ensure the attainment of steady state. Shearing and travel distances in industrial silos and hoppers are of the order of the equipment size and ensure the attainment of steady state. The hopper section of the silo converges towards the outlet; this causes the powder to be compressed in the horizontal direction and to be dilating in the vertical direction due to the gravity enabled flow in downward direction. The larger stresses acting in the hopper are thus horizontal causing the major principal stress to be oriented horizontally along the axis of the hopper (Schulze, 2008). Once the flow from the hopper fully develops and stabilizes, a radial stress field is developed in the

lower part of the hopper in which the local stress at a point in the hopper is assumed to be proportional to the distance of that point from the hopper apex. When the powder is being emptied, the stresses near the hopper outlet are independent of the fill level of the silo and from the stresses in the upper part of the hopper. The powder deformation during flow from the hopper thus corresponds approximately to the steady state flow, which is defined as the flow at constant stresses and a constant bulk density (caused by the powder compression in the horizontal direction and the dilation in vertical direction in the hopper).

The Jenike shear testing methodology used to design arch free mass flow silos assumes the powder to be at the steady state during flow and therefore the two-stage shear testing procedure where the sample is first critically consolidated followed by measurement of its strength is considered relevant for silo design. In shallow containers and other situations where the internal shear of the powder is limited during flow, the powder may not achieve steady state conditions. There are many handling scenarios in the industry in which such a situation may occur, such as in dosing, filling of powders in pharmaceutical and food industries, small-scale silos/hoppers for storage and discharge, powder feeders for tableting and powder spreading process in layer-by-layer powder bed fusion process used in additive manufacturing techniques. Handling scenarios where the powder might not achieve the steady state deformation conditions may be classified as a partially consolidated state or partially consolidated flow, in such flow conditions the flow has not yet fully developed and, therefore, shear stress and bulk density at flow are not constant with time.

2.2.5 Discussion on critical, over-consolidation, and partial consolidation

Steady state powder flow is defined as a state of fully developed flow in the hopper section of the silo where the powder mass is compressed in the horizontal direction and is dilated in the vertical direction, as a result the horizontal stress is larger than the vertical stress (Schulze, 2008). The deformation of the bulk solid while flowing in the hopper approximately corresponds to steady-state flow which is defined as flow at constant stresses and constant bulk density. The shear stress and the normal stress at steady-state can be considered to be remaining fixed during the steady-state flow, along with the deformation being isochoric (taking place at constant volume) due to which bulk density at steady state flow remains constant. The shear stress and the bulk density of the bulk solid at the steady state depends on the normal stress the sample is subjected to, meaning a specific normal stress for a powder should produce repeatable combination of shear stress and bulk density at the steady state.

In the hopper section of the silo considering the bulk solid element at a particular cross section or at a point, the powder is subjected to a major principal consolidation stress. The bulk solid develops a strength which is a function of the consolidation stress. Steady state is described as a state at a cross section in the hopper where the bulk density is constant and the shear and normal stresses are constant during the flow. In the shear testing apparatus, such as Jenike or Schulze ring shear experiments the major principal stress applied to the bulk solid in the apparatus is considered to be similar to that acting in the hopper at a particular point

or cross section. The design methodology is based on the critical condition required for the material to flow at a critical cross section. The pre-shear step forces the sample to be in a reproducible state with almost constant voidage, relevant to the condition at a cross section of the hopper. In shear testing, the powder is subjected to the consolidation stress that it faces at the hopper cross section, the strength or the stress that is required for the material to flow is calculated in the shear experiment. The yield locus of the material is the representation of the failure limit of the sample, with it showing the relation between the shear stress required for the material to flow whilst it being subjected to a normal stress. The yield locus is unique to a particular consolidation stress as the stress history influences the failure strength measured at the incipient flow points. The incipient failure points are determined relevant to the pre-shearing condition and it is used to determine the major principal stress and unconfined yield strength. The major principal stress is determined by estimating the stress state by constructing a Mohr circle tangent to the yield locus and passing through the pre-shear point. The yield locus is taken to be terminating at the pre-shear point as the stresses in the sample cannot exceed those seen at critical consolidation. The unconfined yield strength is determined by a Mohr circle tangent to the yield locus and passing through the origin, as the arch is considered to be supported by only its own weight and the resulting bearing stress it experiences. It is assumed that the failure or crack propagates from the free surface of the solid, and that the sample does not experience any stresses apart from the self-weight and is unrestricted from the bottom. Therefore, the minor principal stress is assumed to be zero, necessitating the Mohr circle to pass through the origin, with the unconfined yield strength determined by the major principal stress of this Mohr circle. In the hopper at different cross-sections there would be different stresses and bulk density during flow as the cross-section changes in dimension, but for a particular cross section during flow the bulk density and stresses are constant with time, i.e., flow has fully developed and flowing at constant parameters. Strength of the bulk solid represented as the unconfined yield strength is a function of the major principal stress as the strength varies with compression and the ensuing particle friction and the number of contacts. The flow function curve is a locus of the unconfined and major principal stresses; the flow factor defined as the ratio of the consolidation stress and the arch bearing stress helps to determine how easily the material will flow or what are the chances of the material to arch.

The state of consolidation achieved by shearing an uncompacted or loose specimen to a constant shear stress and bulk density under a constant normal stress is called the critically consolidated or steady state. Consider now a sample which is critically consolidated by shearing under a normal stress σ . Now if the sample is sheared under a normal stress σ_{low} lower than the previous consolidation normal stress of σ , this sample would correspond to shearing of an over-consolidated sample. In an over-consolidated specimen, the particles have come closer to each other due to the prior consolidation under normal stress σ . Upon shearing of this sample under σ_{low} , powder flow would start only after the yield limit is attained, meaning that the shear stress is sufficient or large enough to cause relative motion between the particles which are adhering to each other. At the start of shearing, there is an increase of the shear stress which causes elastic deformation

of the sample. The consolidated bulk specimen can start to flow once the shear stress is large enough to cause plastic deformation or failure in the sample, which is defined as incipient flow where the particles can move relative to each other. If the over-consolidated sample is sheared to failure in a shear testing device such as the Jenike shear tester or the Schulze RST, the failure of the sample takes place in the shear zone, which is a thin planar area. Post failure, the particles in the shear zone region move relative to each other and the inter-particle distances increase. Therefore, in the shear zone, the sample dilates as the bulk density decreases, the shear resistance and the resulting shear stress decreases. If shearing of the sample is continued post failure, the powder in the shear zone will continue to dilate till the shear stress and bulk density become constant, corresponding to the steady state flow which would have been achieved if a loose or uncompacted specimen would have been sheared under the normal stress σ_{low} . The shear to failure step of the shear test corresponds to the shearing of the over-consolidated sample till failure. First, an uncompacted powder sample is critically consolidated under the pre-shear normal stress σ_{pre} characterized by attainment of steady state shear stress and bulk density. Then, the shear to failure is carried out under a decreased normal stress σ_{sh} , the sample is sheared till attainment of incipient flow or powder failure. As the sample was consolidated at a larger normal stress σ_{pre} , it is consolidated more than it would have been under the lower normal stress σ_{sh} , the powder sample is thus over-consolidated w.r.t σ_{sh} , which is the normal stress at shear.

Partially consolidated state can be defined as the condition where steady state is not reached. At a particular cross section of the hopper or at a particular point, the stresses and the bulk density are not constant with time, the flow has not fully developed. To reach steady state deformation at a particular cross section, a finite or critical amount of shear deformation is required for the particles to have a constant bulk density and to have a near constant number of contacts which is seen in the constant shear stress under a constant normal load. Partially consolidated state would be seen when the deformation in the sample has caused plastic deformation, but it is not enough to cause deformation at a constant bulk density and stresses, meaning the particle contacts haven't fully established and powder voidage is not constant. In the shear experiment, the shear stress during the shearing stage increases till it reaches a steady state value at which particle contacts have fully established and the particles have fully rearranged to result in a constant bulk density. The shear deformation of the powder in the partial consolidation state causes the shear stress to be still rising as the frictional contacts between the particles haven't been fully established or mobilized. The deformation at the partial consolidation state is a fraction of the deformation required for the steady state due to which the steady state has not been established. When the sample is sheared as in the case of the translational shear tester such as Jenike, it takes some finite time or shear strain to reach the steady state. During this time, the sample is flowing but not at a constant bulk density and shear stress. Partially consolidated state tries to capture this state of flow, i.e., when the powder particles are still rearranging and are still trying to establish fully stable contacts and the flow is not at a constant shear stress and bulk density.

In powder shear testing, the powder shear strain is the principal variable forcing/pushing the powder towards the steady state. However, it can be assumed that the level of partial consolidation reached by the powder in the initial transient of the pre-shear stage can be estimated by the fraction of the steady-state shear stress reached. The shear stress experienced by the powder particles at the time of flow for the partially consolidated state is a fraction of the shear stress experienced by the powder at the steady state. The flow at partial consolidation has not yet been fully developed to be at the steady state (isochoric deformation at constant stresses) but is instead flowing at a state where the shear stresses are a fraction of the steady state. Such flow conditions where the flow has not yet fully developed due to which shear stress and bulk density at flow are not constant with time but are instead varying may be found in situations such as intermittent flow of powder and other handling scenarios as described earlier.

The powder flow measurements at critical consolidation according to Jenike's shear testing methodology may not adequately describe the powder flow behaviour for partially consolidated powders. Accordingly, the need arises to characterize powder flow at under consolidated or partial consolidation state conditions to gauge the impact of the deformation state on powders' flowability. The present study focuses on the systematic characterization of powder flow properties of cohesive powders, considering the possibility of partial consolidation. The powder under the same normal stresses for the steady and partial consolidation states being subjected to different shear strain and resulting shear stresses at flow can cause the flowability of the powder at steady and partial consolidation states to vary. The shear stresses at partial consolidation state are lower, which can lead to different yield loci for the two states. The cohesion, yield strength and the flowability of the powder as a result can vary from their steady state counterparts. The effect of the partial consolidation state on the ensuing powder flow needs to be investigated.

2.3 Review of discrete element modelling (DEM)

2.3.1 Introduction to DEM

The granular materials are composed of distinct or discrete elements or particles, the interactions of these distinct elements with the other elements or particles and the walls of the storage/handling containers influence the bulk behaviour of the granular material. The distinct element method (more widely known as the discrete element method DEM) introduced by Cundall and Strack (1979) is an explicit numerical method used to describe the mechanical behaviour of the particles. The forces and displacements of an assembly of particles representing the granular material are determined by computing the contact forces and displacements of the individual constituent particles. DEM helps to simulate the granular flow problems at the particle scale by taking into account the physical and mechanical properties of the particles in the system and is a widely used computational tool in academia and industry to study the powder behaviour in various bulk handling processes and industries. The DEM particles are rigid, and their interactions can be modelled by the "hard sphere" or "soft sphere" approach. In the "hard sphere" approach, the particle contacts are

assumed to take place over short time durations with no particle overlap allowed during the contact. Energy loss during the particle collision is used to determine the particle motion. The “soft sphere” approach in contrast allows a small amount of particle overlap at the contact points, with this overlap analogous to the deformation that occurs at the particle contact points of real granular materials. This particle overlap is used to calculate the particle contact force which is further used to update the particle positions. The “soft sphere” approach is the most widely used DEM approach as it can be used to model a broader range of particle systems including dense particle flows.

I. DEM calculation sequence

O’Sullivan (2011) provided a schematic overview (Figure 2-4) of the sequence of calculations in a DEM simulation. The first step involves defining the system geometry, the particle properties such as size and shape, and the contact model to compute the contact forces during the particle interactions. The next steps include identifying the particle contacts, calculation of the contact forces and the subsequent calculation of the resultant net force acting on the particles. The particle accelerations are then determined and used to determine the particle velocities, displacement and rotations using Newton’s laws of motion. The particle positions are then updated, and the simulation moves forward by one time increment or one time-step. The total simulation time is divided into a finite number of time-steps, and the calculation sequence is followed at each time-step till the simulation is completed.

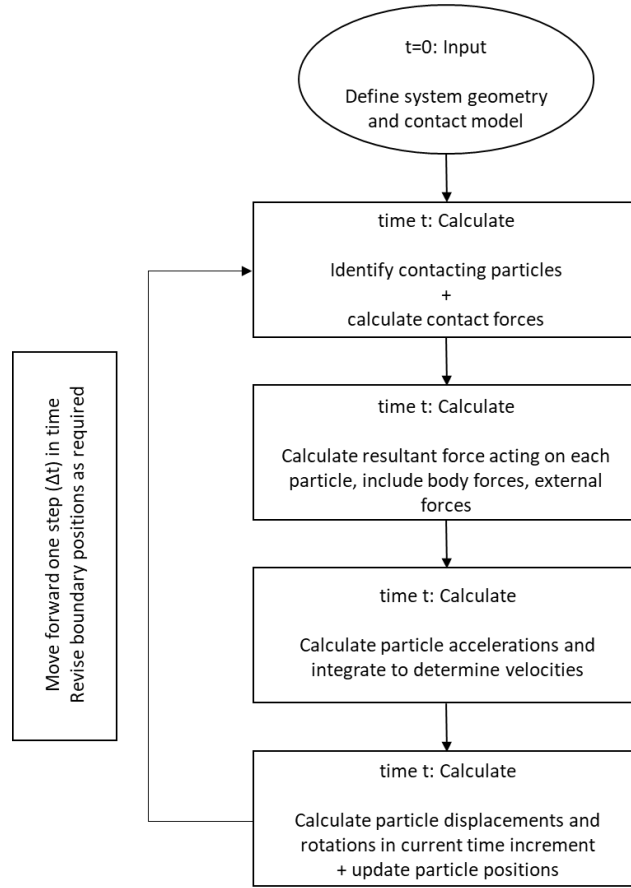


Figure 2-4: DEM calculation sequence (O’Sullivan, 2011).

II. Force, velocity and position determination/calculation

If all the forces acting on a single particle i are known, the change in position and velocity of the particle can be determined by integrating Newton’s equation of motion for the translational (Equation (2-7)) and rotational (Equation (2-8)) motion.

$$m_i \frac{d^2}{dt^2} \mathbf{r}_i = \mathbf{f}_i + m_i \mathbf{g} \quad (2-7)$$

where m_i is the mass of the particle i , t is time, $\mathbf{r}_i$ is the position of the particle, $\mathbf{f}_i$ is the sum of all the forces acting on the particle apart from gravity, $\mathbf{g}$ is the acceleration due to gravity.

The total force $\mathbf{f}_i$ acting on the particle i can be calculated as the summation of its contact forces with the other particles and walls as $\mathbf{f}_i = \sum_c \mathbf{f}_i^c$ (Luding, 2008a), where $\mathbf{f}_i^c$ is the contact force acting on particle i at the c^{th} contact point. This contact force can also include non-contact forces such as van der Waals forces, which in DEM models may act once the particles are in contact, or can be activated even if the particles are some distance apart.

$$I_i \frac{d}{dt} \boldsymbol{\omega}_i = \mathbf{T}_i \quad (2-8)$$

where I_i represents the moment of inertia of particle i , ω_i is the angular velocity of the particle and T_i is the total torque acting on the particle.

The total torque T_i on the particle can be calculated as $T_i = \sum_c (l_i^c \times f_i^c) + q_i^c$ (Luding, 2008a), the vector from the centre of the particle i to the contact point is represented by l_i^c , q_i^c presents the torque acting at the particle contacts other than due to a tangential force, such as rolling resistance.

Newton's equations of motions are used to determine the translational and rotational accelerations of the particles; these accelerations are numerically integrated over a time-step to then determine and update the particle velocities and positions. Several methods such as the Euler time integration method which is a first order time integration method, and second order time integration methods such as Position Verlet and Velocity Verlet are available for this numerical integration. The Euler time integration scheme is used in this study, and the particle velocities (Equations (2-9) – (2-10)) and positions (Equations (2-11) – (2-12)) can be determined from the following Equations.

$$\mathbf{v}_{t+\Delta t} = \mathbf{v}_t + \mathbf{a}_t \Delta t \quad (2-9)$$

$$\boldsymbol{\omega}_{t+\Delta t} = \boldsymbol{\omega}_t + \boldsymbol{\alpha}_t \Delta t \quad (2-10)$$

where $\mathbf{v}_{t+\Delta t}$ and $\boldsymbol{\omega}_{t+\Delta t}$ are the updated particle translational and angular velocity respectively, $\mathbf{v}_t$ and $\boldsymbol{\omega}_t$ are the particle translational and angular velocity respectively at the current time-step, $\mathbf{a}_t$ and $\boldsymbol{\alpha}_t$ are the particle translational and angular acceleration respectively at the current time-step and Δt is the time-step.

$$\mathbf{r}_{t+\Delta t} = \mathbf{r}_t + \mathbf{v}_t \Delta t \quad (2-11)$$

$$\boldsymbol{\theta}_{t+\Delta t} = \boldsymbol{\theta}_t + \boldsymbol{\omega}_t \Delta t \quad (2-12)$$

where $\mathbf{r}_{t+\Delta t}$ (Equation (2-11)) and $\boldsymbol{\theta}_{t+\Delta t}$ (Equation (2-12)) are the updated particle translational and angular position respectively, $\mathbf{r}_t$ and $\boldsymbol{\theta}_t$ are the particle translational and angular position respectively at the current time-step.

Krugger-Emden et al. (2008) demonstrated that the accuracy of DEM integration schemes depends significantly on time-step size, and that higher-order schemes such as position Verlet permit larger timesteps to be used. However, the study showed that the Euler time integration scheme can be used with decent levels of accuracy when sufficiently small time-steps are used, which allow for the particle collision to be resolved satisfactorily over several time-steps. Since the GPU solver of Altair EDEM is restricted to Euler time integration, a sufficiently small time-step was selected for the granular flow simulations whilst using this scheme.

III. Explicit time integration

The Euler time integration method is an explicit time integration method where the properties of the next time step are calculated by the properties at the current time step. The DEM simulation time is divided into

a number of timesteps, the time-step is the finite time increment between two consecutive iterations. The simulations in order to be stable and representative of the real phenomena require a sufficiently small value of the time step so that each particle collision takes place over a number of time steps. The motion of the particles in the granular assembly can be impacted by the disturbance waves, and the time step should be small enough to prevent this wave from propagating further than its immediate neighbouring particles. The Rayleigh wave travels close to the surface of the body; its propagation speed is used in determining the time step in the DEM simulations. The time taken for this wave to propagate through a solid is called the Rayleigh time step T_R , determined from Equation (2-13) (Zheng et al., 2023), the simulation time-step should be smaller than the time required for a Rayleigh wave to transverse or propagate through the smallest size particle r_{min} in the granular assembly. This is commonly referred to as the critical time-step T_{cric} , the time-step used in DEM simulations is a fraction of this critical time-step T_{cric} or T_R .

$$T_R = \frac{\pi r_{min} \sqrt{\frac{\rho_s}{G}}}{0.1631\nu + 0.8766} \quad (2-13)$$

where ρ_s is the solids density of the bulk material, G is the modulus of rigidity related to Young's modulus E by the relation $E = 2G(1 + \nu)$, ν is the Poisson's ratio of the bulk material.

Another approach to determine the value of the critical time-step is to consider the natural frequency of the equivalent spring-mass system (Malone and Xu, 2008). The DEM particle is considered as a simplified spring-mass system where the particle with mass m is connected to ground by a spring of stiffness k . The critical time step is thus determined as $T_{cric} = 2\sqrt{m/k}$ (Cundall and Strack, 1979). O'Sullivan and Bray (2004) recommended a critical time-step of $T_{cric} = 0.17\sqrt{m/k_{spring}}$, for three-dimensional DEM simulations by considering the interaction of particle with mass m at the contact point through a spring of stiffness k_{spring} . A time step of 7.5% of the Rayleigh time step was used in the simulations in this study as this time step provided time-step independent and stable results.

The interaction between two spherical particles can be represented as two overlapping spheres with overlap δ as seen in Figure 2-5. The overlap which represents the particle deformation at the contact point can be determined from Equation (2-14).

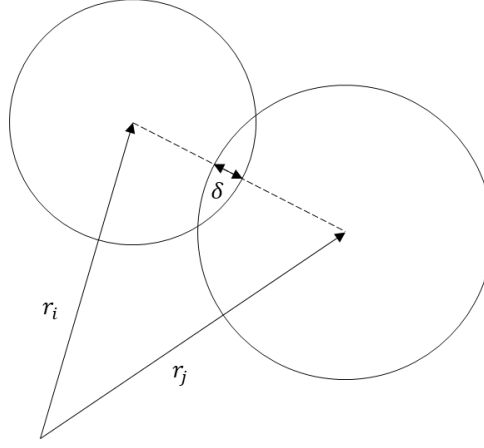


Figure 2-5: Particle contact overlap.

$$\delta = (R_i + R_j) - (\mathbf{r}_i - \mathbf{r}_j) \cdot \mathbf{n} \quad (2-14)$$

where R_i and R_j are the radii of the particles i and j in contact, $\mathbf{r}_i$ and $\mathbf{r}_j$ are the positions of the particles i and j in contact, $\mathbf{n}$ is the unit vector pointing from particle j to particle i calculated as $\mathbf{n} = \mathbf{n}_{ij} = (\mathbf{r}_i - \mathbf{r}_j) / |\mathbf{r}_i - \mathbf{r}_j|$. At the contact point c , the force on particle i from the particle j can be decomposed into normal and tangential parts as, $\mathbf{f}_i^c = \mathbf{f}_n + \mathbf{f}_t$, where the normal part, $\mathbf{f}_n = f^n \mathbf{n}$, tangential part, $\mathbf{f}_t = f^t \mathbf{t}$ and $\mathbf{n} \cdot \mathbf{t} = 0$.

2.3.2 DEM contact models

Since the introduction of the DEM method to model the interaction of particles in granular assemblies, several contact models have been developed to model these interactions. After the particle-particle and particle-wall contacts have been detected, the next step is the calculation of the forces where the contacts have been detected. The contact forces are calculated through the contact model selected by the user. Most DEM contact models use a combination of springs, dashpots and sliders to model the particle interactions at the contact points. As discussed earlier, the particles are allowed to overlap which is representative of their deformation, let's take a case of modelling the particle-particle interaction in the normal and tangential direction. The particle overlap as shown in Figure 2-5 represents the normal overlap, consider this overlap to be the compression of the spring between the two particles in the normal direction. A damper is also provided in the normal direction of the contact as seen in Figure 2-6 to dissipate some part of the particle contact/collision energy. The damper is used to represent the real phenomena of particle interactions where energy during the contact or collision is lost due to friction, sound, heat and other possible losses. Dampers are used to mimic this real-world phenomenon of energy dissipation and for the particles to reach equilibrium in the various granular processes which are being modelled. In the tangential direction, a frictional slider is provided additionally to a tangential spring and tangential damper. The tangential force in some DEM models is limited by the Coulombic friction (Drucker, 1954) limit; the slider can be used to initiate or represent the particle sliding or slip motion once the tangential force exceeds this Coulombic frictional limit. Additionally, the slider can be used to limit the tangential force to this frictional limit so that the particles do not breach

this limit and thus only stick and do not slide. The user selects appropriate contact models for computing the contact forces in the normal and tangential directions dependent on the granular problem being simulated. The following paragraphs will present some of the DEM contact models frequently used in literature, the equations and methodology for the force computation in both normal and tangential directions are discussed for these models.

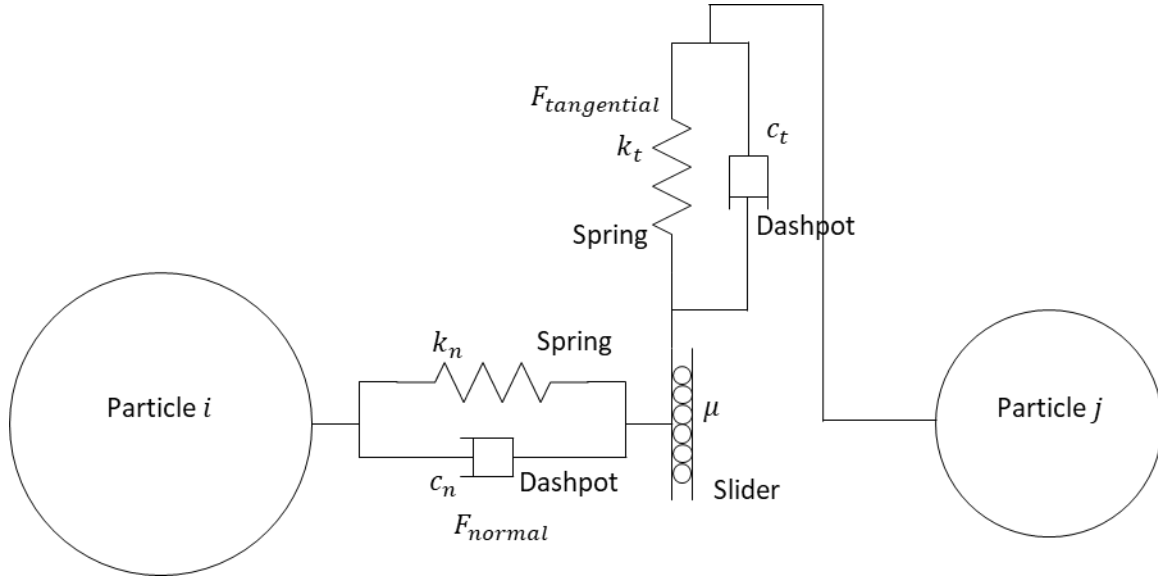


Figure 2-6: DEM particle contact model in normal and tangential direction.

I. Linear spring model

The linear spring model was introduced by Cundall and Strack (1979) to model the granular assemblies at the particle level. The linear spring model is an elastic contact model based on Hooke's law which has a linear spring and a parallel dashpot in the normal direction. The interactions in the tangential direction are modelled by a parallel linear spring-dashpot connected in series to a slider. The dashpots in the normal and tangential direction facilitate the energy dissipation. The total contact force at a particle contact is the summation of the normal and tangential forces, $\mathbf{f}_i^c = \mathbf{f}_n + \mathbf{f}_t$ ($\mathbf{f}_n = f^n \mathbf{n}$, $\mathbf{f}_t = f^t \mathbf{t}$ and $\mathbf{n} \cdot \mathbf{t} = 0$). The contact normal force f^n is summation of the normal spring force f_s^n , and the normal damping force, f_d^n , $f^n = f_s^n + f_d^n$. The force overlap relationship for the normal contact is expressed as Equation (2-15).

$$f_s^n = k_n \delta \quad (2-15)$$

where k_n and δ are the normal spring stiffness and normal overlap respectively.

The expression for the normal stiffness can be obtained by considering the strain energy stored in a Hertzian contact (Equation (2-16)) with the deformation energy of the linear spring (Equation (2-17)) at the same overlap. If the particle contact arises from a particle moving with a relative velocity v , it is assumed it transfers all its energy during the collision/impact $U_{KE}(v)$ to the Hertzian spring, equating this energy helps in obtaining the maximum particle overlap δ_{max} .

$$U_{Hertz}(\delta) = \int_0^\delta \frac{4}{3} E^* \sqrt{R^*} \delta^{3/2} = \frac{8}{15} E^* \sqrt{R^*} \delta^{5/2} \quad (2-16)$$

where E^* and R^* are the equivalent Young's modulus and equivalent radius respectively, determined by $\frac{1}{E^*} = \frac{(1-\nu_i^2)}{E_i} + \frac{(1-\nu_j^2)}{E_j}$ and $R^* = \frac{(R_i \times R_j)}{(R_i + R_j)}$. $E_i, E_j; \nu_i, \nu_j$; and R_i, R_j are the Young's modulus, Poisson's ratio and radii respectively of the particles i and j in contact.

$$U_{spring}(\delta) = \frac{1}{2} k_n \delta^2 \quad (2-17)$$

If $U_{Hertz}(\delta_{max}) = U_{spring}(\delta_{max})$, then, $k_n = \frac{16}{15} E^* \sqrt{R^*} \delta_{max}^{1/2}$, if the kinetic energy of the impact $U_{KE}(v) = \frac{1}{2} m^* v^2 = U_{Hertz}(\delta_{max})$, the expression for δ_{max} can be obtained as Equation (2-18).

$$\delta_{max} = \left(\frac{15}{16} \frac{m^* v^2}{E^* \sqrt{R^*}} \right)^{2/5} \quad (2-18)$$

where m^* is the equivalent mass calculated by $\frac{1}{m^*} = \frac{1}{m_i} + \frac{1}{m_j}$, m_i and m_j are mass of the particles i and j in contact respectively, v is the relative velocity or the velocity of impact.

In the original model proposed by Cundall and Strack (1979), the contact normal force at the current time-step $f_{s,t}^n$ is calculated by incrementally adding the change in the normal force $\Delta f^n = k_n \Delta \delta_n$ at that time-step to the sum of all force increments at the previous time-steps $f_{s,t-1}^n$ such that; $f_{s,t}^n = f_{s,t-1}^n + \Delta f^n$, $\Delta \delta_n$ is the normal displacement increment. The linear spring model provided by commercial DEM suites such as Altair EDEM and ANSYS Rocky, calculate the normal force by considering the instantaneous overlap at that time-step (Equation (2-15)).

The normal damping force f_d^n is determined by the expression $f_d^n = \beta_n v_{rel}^n$, where β_n is the normal dashpot coefficient and v_{rel}^n is the relative velocity in the normal direction.

Garg et al. (2012) used the expressions for the coefficient of restitution e and collision time provided by Silbert et al. (2001) to arrive at the Equation (2-19) for β_n .

$$\beta_n = \sqrt{\frac{4m^* k_n}{1 + \left(\frac{\pi}{\ln e}\right)^2}} \quad (2-19)$$

The contact tangential force f_t is summation of the tangential spring force f_s^t , and the tangential damping force, f_d^t , $f_t = f_s^t + f_d^t$. The force overlap relationship for the tangential contact force f_s^t is expressed in incremental terms to calculate the tangential contact force at the current time-step $f_{s,t}^t$ as per Equation (2-20)

$$f_{s,t}^t = f_{s,t-1}^t + \Delta f^t \quad (2-20)$$

where $f_{s,t-1}^t$ is the tangential force at the previous time-step, i.e., the sum of all force increments at the previous time-steps, Δf^t is the increment in the tangential force calculated as $\Delta f^t = k_t \Delta \delta_t$, k_t is the tangential spring stiffness, $\Delta \delta_t$ is the tangential displacement increment.

The tangential damping force f_d^t is determined similarly to the normal damping force by the expression $f_d^t = \beta_t v_{rel}^t$, where β_t (Equation (2-21)) is the tangential dashpot coefficient and v_{rel}^t is the relative velocity in the tangential direction. The tangential dashpot coefficient is calculated as

$$\beta_t = \sqrt{\frac{4m^*k_t}{1 + \left(\frac{\pi}{\ln e}\right)^2}} \quad (2-21)$$

The expression for the normal dashpot coefficient β_n (Equation (2-19)) follows from the linear spring-dashpot collision model of Garg et al. (2012). The tangential contact is modelled by an analogous linear spring-dashpot collision model with an additional slider, so that the tangential relative motion satisfies the same type of second-order ODE as the normal motion ($m^*\ddot{\delta} + \beta_n\dot{\delta} + k_n\delta = 0$), with stiffness k_t instead of k_n . Consequently, the same restitution-damping relation applies in the tangential direction. Assuming a similar coefficient of restitution e for both normal and tangential directions, the tangential dashpot coefficient β_t is obtained by an expression analogous to Equation (2-19), yielding Equation (2-21).

Cundall and Strack (1979) reported that the analytical results obtained by (Mindlin, 1949) for the ratio of tangential and normal stiffness, k_t/k_n for elastic bodies in contact with elliptical contact areas to be between 2/3 and 1, this ratio can thus be chosen accordingly.

The contact tangential force f_t is limited by the Coulombic-type friction law, if the tangential force exceeds the maximum limit set by the frictional slider, the value of f_t is determined as $\mu f^n t$, where μ represents the coefficient of static friction and $f_t \leq \mu f^n t$.

II. Hertz-Mindlin (no-slip) model

The normal force calculation in this model is based on the Hertzian contact theory (Hertz, 1881) where the force-displacement relationship is non-linear. The tangential force calculation is based on the works of Mindlin (1949) and Mindlin and Deresiewicz (1953), the tangential force model is an incremental force-displacement model for elastic spheres under frictional contact. The combination of these force-displacement models in the normal and tangential directions is referred to as the Hertz-Mindlin (no-slip) model. The expressions for determining or deriving the normal and tangential stiffness together with the theory to relate the coefficient of restitution with the damping coefficient are provided by Tsuji et al. (1992).

The contact normal force f^n is summation of the normal spring force f_s^n , and the normal damping force, f_d^n , $f^n = f_s^n + f_d^n$. The force overlap relationship for the normal contact is expressed as Equation (2-22)

$$f_s^n = \frac{4}{3} E^* \sqrt{R^*} \delta^{3/2} \quad (2-22)$$

where E^* and R^* are the equivalent Young's modulus and radius of the particles i and j in contact respectively as defined earlier, δ is the normal overlap.

The instantaneous normal stiffness $k_n(\delta)$ (Equation (2-23)) for the non-linear elastic normal contact dependent on the normal overlap can be determined by differentiating f_s^n w.r.t δ .

$$k_n(\delta) = \frac{df_s^n}{d\delta} = 2E^* \sqrt{R^*} \delta^{1/2} \quad (2-23)$$

The damping force for the normal direction f_d^n can be calculated from Equation (2-24)

$$f_d^n = 2 \sqrt{\frac{5}{6}} \beta \sqrt{m^* k_n} v_{rel}^n \quad (2-24)$$

where β is a damping constant calculated as, $\beta = \frac{\ln e}{\sqrt{(\ln e)^2 + \pi^2}}$ (Tsuji et al. 1993), e is the coefficient of restitution, m^* is the equivalent mass of the particles i and j in contact and v_{rel}^n is the relative velocity in the normal direction.

The contact tangential force f_t is summation of the tangential spring force f_s^t , and the tangential damping force, f_d^t , $f_t = f_s^t + f_d^t$. The force overlap relationship for the tangential contact force f_s^t is expressed in incremental terms to calculate the tangential contact force at the current time-step $f_{s,t}^t$ as Equation (2-25).

$$f_{s,t}^t = f_{s,t-1}^t + \Delta f^t \quad (2-25)$$

where $f_{s,t-1}^t$ is the tangential force at the previous time-step, i.e., the sum of all force increments at the previous time-steps, Δf^t is the increment in the tangential force calculated as $\Delta f^t = k_t \Delta \delta_t$, k_t is the tangential spring stiffness, $\Delta \delta_t$ is the tangential displacement increment which can be calculated as $\Delta \delta_t = v_{rel}^t \Delta t$, where v_{rel}^t and Δt are the relative velocity in the tangential direction and time step respectively.

The tangential stiffness k_t is calculated as $k_t = 8G^* \sqrt{R^*} \delta^{1/2}$, where G^* is the equivalent modulus of rigidity calculated as $\frac{1}{G^*} = \frac{(2-\nu_i)}{G_i} + \frac{(2-\nu_j)}{G_j}$. G_i , G_j and ν_i , ν_j are the modulus of rigidity and Poisson's ratio respectively of the particles i and j in contact.

The tangential damping force f_d^t can be determined with an expression similar to the normal damping force from Equation (2-26)

$$f_d^t = 2 \sqrt{\frac{5}{6}} \beta \sqrt{m^* k_t} v_{rel}^t \quad (2-26)$$

where v_{rel}^t is the relative velocity in the tangential direction.

The contact tangential force $\mathbf{f}_t$ is limited by the Coulombic-type friction law, if the tangential force exceeds the maximum limit set by the frictional slider, the value of $\mathbf{f}_t$ is determined as $\mu f_s^n \mathbf{t}$, where μ represents the coefficient of static friction and $\mathbf{f}_t \leq \mu f_s^n \mathbf{t}$. Chung (2006) suggested using the normal spring force f_s^n and not the total normal force f^n for calculating the friction limit.

The linear spring and Hertz-Mindlin contact models model the particle interaction in the normal direction without the consideration of cohesive forces. For modelling powders which are composed of fine particles, often times the cohesive forces such as the van der Waals forces acting at the particle scale significantly impact the flow behaviour of the bulk powder. Also, as seen from the study conducted by Johnson et al. (1971), adhesive forces develop between the particles due to the surface energy (originating from van der Waals forces). Therefore, to accurately model fine cohesive powders it is important to capture the effect of the particle level forces such as the van der Waals force on the adhesive force between the particles, the model discussed next tries to capture this effect.

III. Johnson-Kendall-Roberts (JKR) model

Johnson et al. (1971) studied the impact of surface energy on the elastic bodies in contact. They noted that at low loads, the area of contact between two spheres was significantly larger than the one predicted by Hertz. They noted that the contact area tended towards a constant value as the loads applied were reduced to zero, if the spheres were clean and dry it led to strong adhesion forces, at higher loads the results were found to be closely matching the Hertz theory. The observations suggested that attractive surface forces were acting between the solids, and their effect became increasingly significant as the loads tended towards zero. Johnson et al. (1971) derived relations to capture the impact of surface energy on the contact radius and adhesive force between the lightly loaded spherical surfaces, these equations are used in the calculation of normal contact force in the DEM model came to be known popularly as the Johnson-Kendall-Roberts (JKR) model.

The increased contact area a_s due to the presence of surface energies of the two contacting bodies can be determined by Equation (2-27).

$$a_s^3 = \frac{3R^*}{4E^*} \left[f_{JKR}^n + 3\pi\gamma^*R^* + \sqrt{f_{JKR}^n 6\pi\gamma^*R^* + (3\pi\gamma^*R^*)^2} \right] \quad (2-27)$$

where R^* and E^* are the equivalent radius and Young's modulus as defined earlier, γ^* is the contact surface energy calculated as $\gamma^* = \gamma_1 + \gamma_2 - \gamma_{12}$, γ_1 and γ_2 are the surface energies of particles i and j in contact respectively and γ_{12} is the interface energy of these particles. The normal force f_{JKR}^n at the contact is a function of contact radius a_s and is given by Equation (2-28) (Coetzee and Scheffler, 2023)

$$f_{JKR}^n = \frac{4E^*a_s^3}{3R^*} - \sqrt{8\pi\gamma^*E^*a_s^3} \quad (2-28)$$

The normal overlap δ_{JKR}^n or displacement can be related to the contact radius a_s by Equation (2-29) (Johnson, 1985).

$$\delta_{JKR}^n = \frac{a_s^2}{R^*} - \sqrt{\frac{2\pi\gamma^* a_s}{E^*}} \quad (2-29)$$

The contact normal force f^n is summation of the normal force f_{JKR}^n , and the normal damping force, f_d^n , $f^n = f_{JKR}^n + f_d^n$. The contact stiffness of the equivalent spring in the normal direction can be calculated as $k_n = 2E^* a_s$, this value of k_n can be used in Equation (2-24) to calculate the damping force f_d^n (Chen and Elliott, 2020). The contact stiffness of the equivalent spring in the tangential direction can be calculated as $k_t = 8G^* a_s$ (Marshall, 2009), where G^* is the equivalent modulus of rigidity calculated from $\frac{1}{G^*} = \frac{(2-\nu_i)}{G_i} + \frac{(2-\nu_j)}{G_j}$.

The contact tangential force f_t is summation of the tangential spring force f_{JKR}^t , and the tangential damping force, f_d^t , $f_t = f_{JKR}^t + f_d^t$. The force overlap relationship for the tangential contact force f_{JKR}^t is expressed in incremental terms to calculate the tangential contact force at the current time-step (Chen et al., 2022) in a similar way as the tangential force calculation in the Hertz-Mindlin model.

The contact radius a_o (Equation (2-30)) of the contact when $f_{JKR}^n = 0$ represents the force equilibrium where the force due to the Hertzian contact and the surface energy become equal.

$$a_o = \left(\frac{9\pi\gamma^* R^{*2}}{2E^*} \right)^{1/3} \quad (2-30)$$

The pull-off force f_o^{JKR} which is defined as the force required to separate the two particles in contact (Morrissey, 2013) can be determined from Equation (2-31).

$$f_o^{JKR} = -\frac{3}{2}\pi\gamma^* R^* \quad (2-31)$$

When two particles come into contact which can be defined as the particle overlap value to be zero, the value of the normal force drops to a fraction of the pull-off force which can be used to represent the van der Waals forces between the particles.

The models discussed above can be regarded as non-hysteretic contact models because loading and unloading in the normal direction takes place on the same branch. Depending on the contact law employed, the normal force-deformation relation may be linear (Linear-spring model), or nonlinear (Hertz-Mindlin, JKR model). In real particle contacts, however, particles undergo elastic and plastic deformations when they are pressed together, showing hysteretic behaviour (Jones, 2003). The plastic deformation causes increased contact areas during unloading, and a result increased cohesive behaviour. Therefore, it is imperative to include the elastic-plastic nature of the particle contacts along with the adhesive nature for simulating the

flow behaviour and other characteristics of a real cohesive powder. A model encapsulating the above-mentioned particle behaviour was developed by (Luding, 2008b, 2007, 2006) and is discussed further.

IV. Luding model

The contact normal force f^n is the summation of the normal spring force f_{hys}^n , and the normal damping force, f_d^n , $f^n = f_{hys}^n + f_d^n$. The relation between the hysteretic spring normal force f_{hys}^n and the normal overlap δ is expressed mathematically by Equations in (2-32).

$$f_{hys}^n = \begin{cases} f_o + k_1\delta & \text{if } k_2(\delta - \delta_o) \geq k_1\delta \\ f_o + k_2(\delta - \delta_o) & \text{if } k_1\delta > k_2(\delta - \delta_o) > -k_{adh}\delta \\ f_o - k_{adh}\delta & \text{if } -k_{adh}\delta \geq k_2(\delta - \delta_o) \end{cases} \quad (2-32)$$

The force displacement relationship can be described by the loading branch stiffness k_1 , the unloading branch stiffness k_2 , the plastic deformation δ_o , the constant pull-off force f_o and the adhesive branch stiffness k_{adh} . The Equations in (2-32) sequentially represent the force-overlap relations when the particle is in the initial loading, unloading phase and on the adhesive branch respectively. The force-deformation schematic of the Luding model in the normal direction is shown in Figure 2-7.

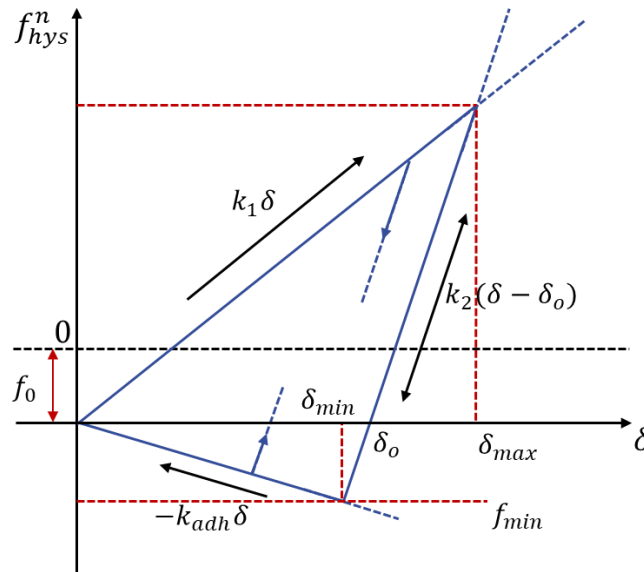


Figure 2-7: Force-deformation schematic of Luding model in the normal direction.

The total contact normal force f_n is the sum of the hysteretic spring force f_{hys}^n and the normal damping force f_d^n and is given by Equation (2-33).

$$\mathbf{f}_n = f^n \mathbf{n} = (f_{hys}^n + f_d^n) \mathbf{n} \quad (2-33)$$

where $\mathbf{n}$ is the unit normal vector pointing from the contact point to the centre of the particle. The normal damping force f_d^n is calculated as $f_d^n = \gamma_o v_{rel}^n$, where γ_o is the viscous damping coefficient and v_{rel}^n is the relative velocity in the normal direction.

The plastic contact deformation δ_o is the deformation at which the force on the unloading branch k_2 becomes equal to the pull-off force f_o and is determined from Equation (2-34).

$$\delta_o = \left(1 - \frac{k_1}{k_2}\right) \delta_{max} \quad (2-34)$$

where δ_{max} is the maximum particle overlap and is stored as the history dependent variable in the model. Further unloading below δ_o leads to the development of attractive adhesive forces until the maximum adhesive force or the minimum force of the particle interaction f_{min} (Equation (2-35)) is reached at the overlap δ_{min} (Equation (2-36)). Further unloading makes the force calculation switch to the adhesive branch till finally the separation of the contact occurs.

$$f_{min} = f_o - k_{adh} \delta_{min} \quad (2-35)$$

$$\delta_{min} = \left(\frac{k_2 - k_1}{k_2 + k_{adh}}\right) \delta_{max} \quad (2-36)$$

(Luding, 2008b) acknowledged that a non-linear force-overlap relation would be more realistic, and to account for the non-inclusion of this behaviour in the model, it was suggested to increase the unloading stiffness with deformation, with k_2 linked to δ_{max} as per Equation (2-37).

$$k_2 = \begin{cases} k_2^{max} & \text{if } \delta_{max} \geq \delta_{max}^* \\ k_1 + (k_2^{max} - k_1) \delta_{max} / \delta_{max}^* & \text{if } \delta_{max} < \delta_{max}^* \end{cases} \quad (2-37)$$

where k_2 interpolates or varies between k_1 and k_2^{max} , dependent also on the plastic flow limit overlap δ_{max}^* determined from Equation (2-38).

$$\delta_{max}^* = \frac{k_2^{max}}{k_2^{max} - k_1} \varphi_f \frac{2R_i R_j}{R_i + R_j} \quad (2-38)$$

where φ_f is the dimensionless plasticity depth, and R_i and R_j are the radii of particles i and j in contact.

The tangential spring force f_t is calculated as $f_t = -k_t \xi - \gamma_t v_{rel}^t$, where k_t is the tangential spring stiffness, ξ is the tangential spring length or deformation, γ_t is the tangential dissipation coefficient and v_{rel}^t is the relative velocity in the tangential direction. The tangential spring deformation is calculated incrementally similar to the method described earlier for other models.

V. EEPA model

The Edinburgh Elasto-Plastic Adhesive contact model (Morrissey, 2013; Thakur et al., 2014b) tries to account for the physical phenomena seen in adhesive contact between micron sized particles. A pair of particles or

small agglomerates when pressed together undergo elastic and plastic deformations. The adhesive contact between the particles leads to a development of an adhesive strength or pull-off force, which sees an increase when the plastic contact area between the particles increases. The EEPA model accounts for both the elasto-plastic deformation at the contact and the adhesion dependent on the contact area. The EEPA model is conceptually similar to the Luding model, but it provides the added versatility of using the non-linear force-deformation relationships for simulating the particle behaviour.

The EEPA contact model comprises of three distinct branches which can be used to model the particle interactions. The loading branch described by the virgin loading stiffness k_1 is used to calculate the contact force when the particles first come into contact. The contact force increases as the loading continues and overlap between the particles increases, the maximum particle deformation is termed as δ_{max} . The unloading branch described by the particle unloading/reloading stiffness k_2 is brought into action once the unloading of the particles starts. If reloading takes place, the contact force is calculated through the unloading branch until the force reaches the maximum force calculated by the loading branch k_1 , at this point the model switches back to the virgin loading stiffness branch. In case the unloading continues below the plastic overlap δ_p , an attractive adhesive force is developed until it reaches f_{min} , with the deformation at this point being δ_{min} . Further unloading beyond this point leads to a reduction in the adhesive force, the contact force is calculated by the adhesive branch described by the adhesive branch stiffness k_{adh} , the unloading continues until the particles are separated. If reloading takes place whilst on the adhesive branch, the reloading takes place along the unloading path until it reaches the virgin loading path. The constant pull-off force f_0 can be used to represent the ever-present cohesive forces such as the van der Waals forces acting between the particles.

The EEPA model can be operated both in linear mode and the non-linear mode by setting the power value i of the force-deformation ($f - \delta$) relation for the different branches, $f = k\delta^i$. A single value serves for both the unloading and loading branches, the value for the adhesive branch can be set independently from that of the loading/unloading branches. A power value of unity for the loading/unloading and adhesive branches helps operate the EEPA model in the linear ($f - \delta$) mode. If the loading and unloading branch stiffness are equal $k_1 = k_2$, and $i = 1$, the EEPA model serves as the linear spring model. The EEPA model can be operated as the Hertzian spring model if $k_1 = k_2$, and $i = 1.5$. For this doctoral study, the linear version of the EEPA model was utilized and the force-overlap schematic for the same is shown in Figure 2-8. The linear version of the EEPA model differs from the Luding model in some ways, the unloading/reloading stiffness k_2 is constant and does not vary, EEPA tracks δ_p as the history parameter instead of δ_{max} in the Luding model.

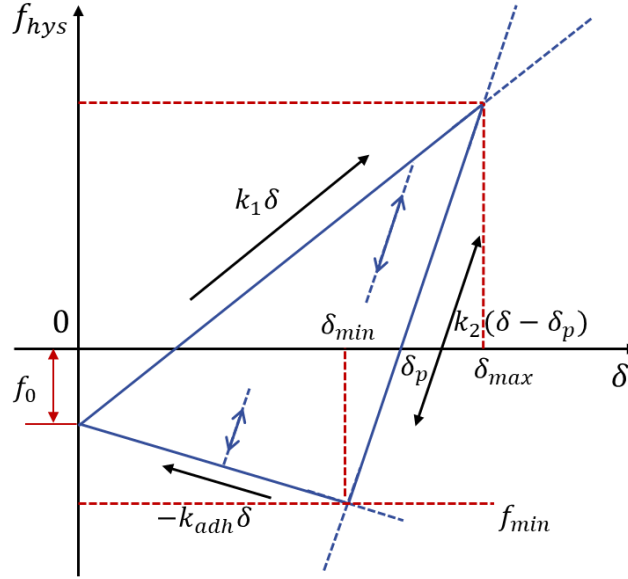


Figure 2-8: Linear mode of Edinburgh Elasto-Plastic Adhesive (EEPA) contact model.

The total contact normal force $\mathbf{f}_n$ is the sum of the hysteretic spring force f_{hys} and the normal damping force f_d^n and is given by Equation (2-39).

$$\mathbf{f}_n = f^n \mathbf{n} = (f_{hys} + f_d^n) \mathbf{n} \quad (2-39)$$

where $\mathbf{n}$ is the unit normal vector pointing from the contact point to the centre of the particle.

The relation between the hysteretic spring normal force f_{hys} and the normal overlap is expressed mathematically by Equations in (2-40).

$$f_{hys} = \begin{cases} f_0 + k_1 \delta & \text{if } k_2(\delta - \delta_p) \geq k_1 \delta \\ f_0 + k_2(\delta - \delta_p) & \text{if } k_1 \delta > k_2(\delta - \delta_p) > -k_{adh} \delta \\ f_0 - k_{adh} \delta & \text{if } -k_{adh} \delta \geq k_2(\delta - \delta_p) \end{cases} \quad (2-40)$$

The normal damping force f_d^n is determined by the expression $f_d^n = \beta_n v_{rel}^n$, where β_n is the normal dashpot coefficient and v_{rel}^n is the relative velocity in the normal direction, β_n can be determined from Equation (2-41)

$$\beta_n = \sqrt{\frac{4m^* k_1}{1 + \left(\frac{\pi}{\ln e}\right)^2}} \quad (2-41)$$

where m^* is the equivalent mass of the particles i and j in contact as described earlier, e is the coefficient of restitution.

The total contact tangential force f_t is the summation of the tangential spring force f_s^t , and the tangential damping force, f_d^t , $f_t = f_s^t + f_d^t$. The force overlap relationship for the tangential contact force f_s^t is expressed in incremental terms to calculate the tangential contact force at the current time-step $f_{s,t}^t$ from Equation (2-42).

$$f_{s,t}^t = f_{s,t-1}^t + \Delta f^t \quad (2-42)$$

where $f_{s,t-1}^t$ is the tangential force at the previous time-step, i.e., the sum of all force increments at the previous time-steps, Δf^t is the increment in the tangential force calculated as $\Delta f^t = k_t \Delta \delta_t$, k_t is the tangential spring stiffness, $\Delta \delta_t$ is the tangential displacement increment. The tangential damping force f_d^t is determined by the expression $f_d^t = \beta_t v_{rel}^t$, where β_t is the tangential dashpot coefficient and v_{rel}^t is the relative velocity in the tangential direction, β_t can be determined from Equation (2-43) below.

$$\beta_t = \sqrt{\frac{4m^*k_t}{1 + \left(\frac{\pi}{\ln e}\right)^2}} \quad (2-43)$$

The contact tangential force f_t is limited by the Coulombic-type friction law, the limiting tangential friction force f_c^t is determined as $f_c^t \leq \mu |f_{hys} + k_{adh} \delta - f_0|$, where μ represents the coefficient of static friction.

2.3.3 DEM coarse-graining

Although DEM enables modelling the particle interactions at the real particle scale, it is still prohibitively expensive to model industrial scale problems or granular systems having average size of particles below 200 μm , because larger number of particles in the system leads to a higher number of detected contacts, leading to a greater number of calculations for computing the forces. The computational resources available make modelling the particles at this real scale to be time consuming and expensive. The real particles often do not have a consistent repeatable shape which can be modelled in DEM, moreover the surface roughness and asperities are hard to model. Therefore, to model granular systems having particles in the micron range, the mesoscopic scale approach is used where each DEM particle is a representation of an intermediate length scale between the actual particle size and the bulk scale. A coarse-graining or meso-scale approach scales up the particle size and simplifies the particle shape to represent the collection of smaller particles. This helps reduce the number of particles in the system, resulting in lower computation costs, the larger particle is called the coarse-grain or meso-scale grain. For the larger particles to be able to represent the smaller particles accurately, scaling of the forces or the parameters involved in the simulation should be done appropriately. The coarse-graining approach has been used to model diverse particle handling processes such as pneumatic conveying, shear flow, mixing and compaction. Appendix A, Section I presents and discusses some of the scaling studies undertaken in literature, which are categorized into two primary approaches: parameter scaling and force scaling.

The coarse-graining studies often provide parameter scaling laws (Bierwisch et al., 2009; Lommen et al., 2019; Nakamura et al., 2020) or force scaling laws (Washino et al., 2023; Widartiningsih et al., 2020) to maintain the energies between the upscaled and original systems. The upscaling in these systems often requires determination of properties at the particle level such as contact stiffness, coefficient of friction, coefficient of

restitution, pull-off force etc., which are fed into the equations for modelling the particles. These parameters can be determined for original particles having d_{50} of mm range with relative ease and accuracy and thus can be used in the scaling laws for the coarse-grained systems, the results of the upscaled and original systems are compared to evaluate the efficacy of the scaling. These particle level properties are often difficult to obtain for particles which have d_{50} of a few microns, making it difficult to scale the parameters or forces required for scaling the system. For such systems, the coarse-grain particle properties are not directly scaled, but the properties are calibrated in order to match the bulk experimental response. These parameters are often further validated through validation experiments. There are various studies in literature (Ajmal et al., 2020; Orefice and Khinast, 2020; Pachón-Morales et al., 2019; Zeng et al., 2020) which calibrate the input parameters of the DEM model against the bulk experiments, this approach serves as a useful tool for modelling systems whose particle properties are not known.

Appendix A, Section II puts forward some studies conducted in literature where the coarse graining or upscaling approach has been used, and the DEM model is calibrated and validated. The efficacy of the coarse graining model is often evaluated by comparing the simulation results of the coarse grained model with the simulation results of the model which uses the original or unscaled particles. Generally, experiments conducted at the bulk level are used to calibrate the DEM model parameters. The DEM model parameters are calibrated in order for the experimental and the simulation results to have minimal discrepancy, suitable output variables are selected for comparing the experimental and modelled response. The calibrated model can then be used to study the particle level phenomena responsible for bulk behaviour. Further, the calibrated parameters can be validated by simulating a different experiment or characterization method and by comparing the experimental and simulation results.

The studies in Appendix A, Section II show that the coarse-grained model can be calibrated by using flow characterization experiments across static methods, dynamic methods and the defined consolidation methods. The coarse-graining technique is a useful modelling tool which conserves or reproduces the key characteristics of the original system whilst decreasing computational costs. It is important to choose the appropriate experimentation method for calibration based on the bulk and the particle level phenomena which is of interest, and which has to be studied further through DEM. For example, for studying the particle level phenomena or bulk flow in a shear cell, it is appropriate to calibrate the DEM model through experiments involving quasi-static shearing.

I. Shear testing DEM simulations

The shear cell test conducted based on the shear testing methodology introduced by Jenike is a widely used and a fundamental test to assess the powder's flowability. The Jenike shear tester (Barletta et al., 2019), Schulze RST (Schulze and Wittmaier, 2003), FT4 powder rheometer (Stavrou et al., 2020) and Anton Paar

rheometer (Zinatlou Ajabshir et al., 2024) are based on this methodology. The following paragraphs will discuss some studies pertaining to the simulation of the various shear testing devices.

Baran et al. (2009) studied the shearing behaviour of lunar soil by simulating the Schulze Ring Shear Tester with the JKR model, the lunar soil was represented by multi-sphere DEM particles in the simulations. The effect of shear cell size, particle shape, shearing velocity, static friction, shear modulus and surface energy on the shear stress was studied and compared to the experimental value. It was found that barring the shearing velocity and cell size, the other factors influenced the shear stress. A sensitivity analysis study on the effect of various DEM parameters on the steady state shear stress was conducted by Simons et al. (2015). The Hertz-Mindlin contact model was used to model the cohesionless coarse-grained particles in Schulze RST. The study showed that the Young's modulus, particle-particle static and rolling friction parameters impacted the shearing behaviour and thus the steady state shear stress, whilst the effect of Poisson's ratio and coefficient of restitution was minimal on the shear stress. The study recommended determining the Young's modulus of the experimental particles to be used in the DEM model or to set it to a pre-defined value before the start of the calibration process.

Karkala et al. (2019) studied the flow behaviour of cohesive granules using dynamic yield strength (DYS) and shear cell experiments, and used the JKR contact model to simulate these experiments in DEM. The parametric sensitivity conducted on the DYS simulations showed that the yield strength was dependent on the surface energy along with particle-particle sliding and rolling friction coefficients, with these parameters also impacting the steady state shear stress in the shear cell simulations. The study recommended calibrating first the surface energy through the DYS simulations followed by the calibration of the friction coefficients through the shear cell DEM simulations. This recommendation was based on the inability of the shear cell model to show adequate sensitivity to the surface energy on the ensuing shear stress. The surface energy could have been calibrated by the shear cell simulations if the shear/failure strength post consolidation of the sample was considered and used in the sensitivity study.

Jajcevic et al. (2024) discussed a calibration method for cohesive powders based on multiple characterization experiments at varying stress states. Coarse grained particles were used, and the DEM model was developed using the MEPA (Macro Elasto-Plastic Adhesion model) contact model. The different model parameters were calibrated using the varying characterization experiments. Figure 2-9 presents the DEM calibration algorithm used in the study. The workflow begins by selecting an appropriate DEM particle size distribution, an initial parameter set is then estimated from prior experience, optimization methods, or machine-learning-assisted approaches. Calibration proceeds sequentially through experimental tests that target specific DEM parameters; packing density, compressibility, and elastic recovery measurements are used to calibrate particle density, loading stiffness, and unloading stiffness. The experimental pre-shear shear stress, cohesion and internal friction angle values were used for calibrating the shear cell model, with the tangential to normal

stiffness ratio, adhesive stiffness and sliding friction coefficient calibrated using the FT4 shear cell. The rotating-drum experiments yielded the dynamic angle of repose and cohesive index, which are used to calibrate the restitution coefficient and pull-off force. The objective was to obtain a single parameter set capable of reproducing all characterization tests, with a target simulation error below 5% relative to experimental measurements.

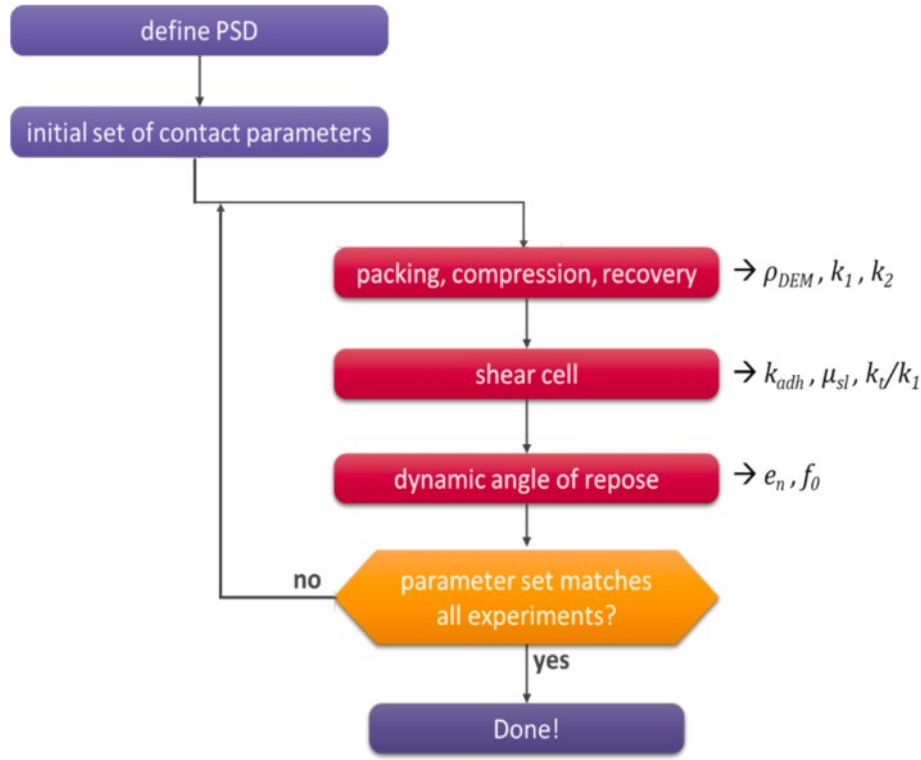


Figure 2-9: DEM model calibration algorithm (Jajcevic et al., 2024).

The calibrated shear cell model was able to reproduce the yield locus at one pre-shear normal loading (4 kPa), with good agreement between the experimental and simulated pre-shear shear stress and internal friction values. However, validation of the calibrated model was limited to a single pre-shear stress level, and its ability to reproduce yield loci across a wider range of pre-shear normal loads was not demonstrated. Thus, the robustness of the calibrated parameter set over the full stress range relevant to powder flow behaviour remains to be established.

Ketterhagen and Wassgren (2022) suggested using shear cell experiments to determine the particle-particle and particle-wall sliding friction coefficients to be used in the coarse-grained DEM model, the calibration approach was said to be applicable to powders with low to moderate cohesion and operating within the quasi-static regime. Chen et al. (2022) conducted a full factorial parametric sensitivity study to study the influence of the DEM model parameters on the cohesive powder flow in a ring shear tester. The study used the JKR contact model and the pre-shear shear stress and the shear stress at incipient flow for the calibration and validation studies. It was found that the particle friction significantly impacts the shear stress at pre-shear,

with an interaction effect of the Young's modulus and surface energy seen on the incipient flow shear stress. The calibrated DEM model was able to match the experimental shear stress values at incipient failure, and the steady state shear stress across varying pre-shear normal loads. The study could have studied the effectiveness of the calibrated model to match the experimental yield locus constructed using the pre-shear point and multiple shear points. The effectiveness of the calibrated model to reproduce a continuous, multi-point yield locus was not demonstrated. Thus, the ability of the calibration framework to reliably reproduce and predict standard bulk scale powder flow properties, such as cohesion and angle of internal friction remains to be established.

Louati et al. (2019) studied the effect of the sliding friction coefficient and surface energy on the internal friction angle and cohesion values obtained from the FT4 shear cell DEM model. The JKR contact model was used to model the cohesive monodisperse spherical particles, the simulations were carried out at varying pre-shear normal loads, full yield loci at each of these normal loads was used to obtain the internal friction and cohesion values for the cohesive and non-cohesive DEM powders. The results were used to find the correlations between the internal friction and sliding friction coefficient, and between the cohesion and surface energy. The study did not calibrate the DEM model or replicate the experimental results for a real powder but was meant for understanding the influence of the sliding friction and surface energy onto internal friction and cohesion of the DEM powder.

Lischka & Nirschl (2023) studied the flow behaviour of a cohesive powder used in battery cathodes through experiments and coarse-grained DEM modelling on Schulze RST. A coarse-graining factor of 200 was used to upscale the micron sized particles to be represented by mm scale particles in the simulation. The simplified JKR model was used to model the powder, the particle-wall static & dynamic friction coefficients & cohesion energy density were varied to minimize the difference between the experimental & DEM shear stresses. The shear stress time series curves of the DEM model showed that the shear stress before the shearing step was not reduced to zero (Figure 2-10), making the comparison with the experiments erroneous. Moreover, some of the shearing points did not show the post peak reduction in shear stress observed experimentally. Since this stress decrease is associated with dilation during incipient shearing representing real particle behaviour, the DEM model appears to inadequately capture the deformation and rearrangement mechanisms governing the powder's shear response.

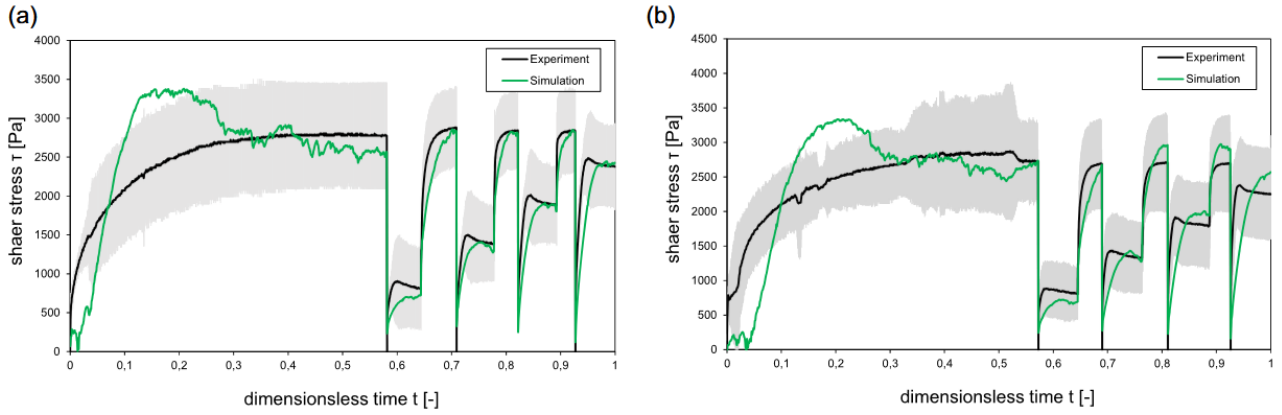


Figure 2-10: Comparison of experimental and simulated shear stress time series curves (Lischka and Nirschl, 2023).

Commercial DEM software suites such as EDEM provide the shear test simulation as a “calibration kit” or tool for calibrating the model with the experimental powder response, such that the calibrated parameter set can be determined, and used further in other simulations. Despite the shear cell test being fundamental for powder characterization, limited studies can be found in literature which try to simulate the Jenike shear testing procedure to determine the flow function of a cohesive powder. There have been studies where coarse graining has been used for simulating the shear test (Jajcevic et al., 2024; Ketterhagen and Wassgren, 2022; Pachón-Morales et al., 2019; Simons et al., 2015), these studies have not been able to reproduce the experimental flow function which is obtained by conducting the shear test at varying levels of pre-shear normal load, requiring stress history dependent/sensitive behaviour of the modelled particle system.

The existing DEM frameworks for shear cell modelling exhibit significant technical limitations. For instance, workflows like that of Jajcevic et al. (2024) which relied on a single pre-shear normal loading during calibration, the calibrated parameter sets remain unvalidated across broader pre-shear and consolidation stress ranges. Studies like Chen et al. (2022) focus exclusively on matching isolated steady state pre-shear and incipient flow stress points, rather than demonstrating the capacity to reconstruct a continuous, multi point experimental yield locus. Where full yield loci have been reconstructed, such as in the work of Louati et al. (2019), the model is used for understanding the influence of input parameters on the output responses of the DEM powder composed of monodisperse spheres, rather than reproducing experimental results for real, polydisperse powders. The DEM model by Lischka and Nirschl (2023) failed to adequately capture the sample dilation expected upon incipient shearing, while additionally failing to correctly reduce the shear stress during counter rotation of the sample to zero, prior to initiating incipient failure in the sample.

This study aims to address these gaps by developing and calibrating a coarse-grained cohesive DEM model capable of reproducing experimental flow behaviour, and to use this model to further study the microscopic phenomena influencing the bulk behaviour of the system.

2.4 Summary

This chapter reviewed the relevant literature necessary to understand the background of the work carried out in this thesis. The literature review presented in this chapter aimed to identify knowledge gaps in the current literature and established the need to address them. The chapter was divided into two parts; the first part addressed research pertaining to the experimental flow characterization of powders, specifically discussing and reviewing the Jenike shear testing methodology. The possibility of the powders to be partially consolidated was discussed together with the need to characterize the flowability of such powders, considering that the Jenike methodology determines flowability of powders under critical consolidation. The second part of this chapter discussed the research carried out to model powder systems using coarse-graining DEM methodology. The review identified a need for DEM modelling framework capable of reproducing stress history dependent flow behaviour across the full stress range.

condition. Therefore, the material may sustain stress states associated with further consolidation beyond those represented by the partially consolidated Mohr circle. Therefore, the point of tangency between the yield locus and the partially consolidated Mohr circle cannot be interpreted as the termination point of the yield locus. The coaxiality hypothesis, which assumes that the principal directions of stress and strain coincide, can be adopted (Hilden et al., 2008; Schulze, 2008) to determine the stress state and the major principal stresses for the powders under partial consolidation. Hilden et al. (2008) applied the coaxiality hypothesis to determine the flow function for critically consolidated powders using shear testing data.

Considering an element W of the bulk solid in the shear cell as shown in Figure 3-2, the x direction is horizontal and the y direction is vertical, the shear cell is rotated clockwise with a constant angular velocity ω for shearing the sample whilst subjected to a normal load N .

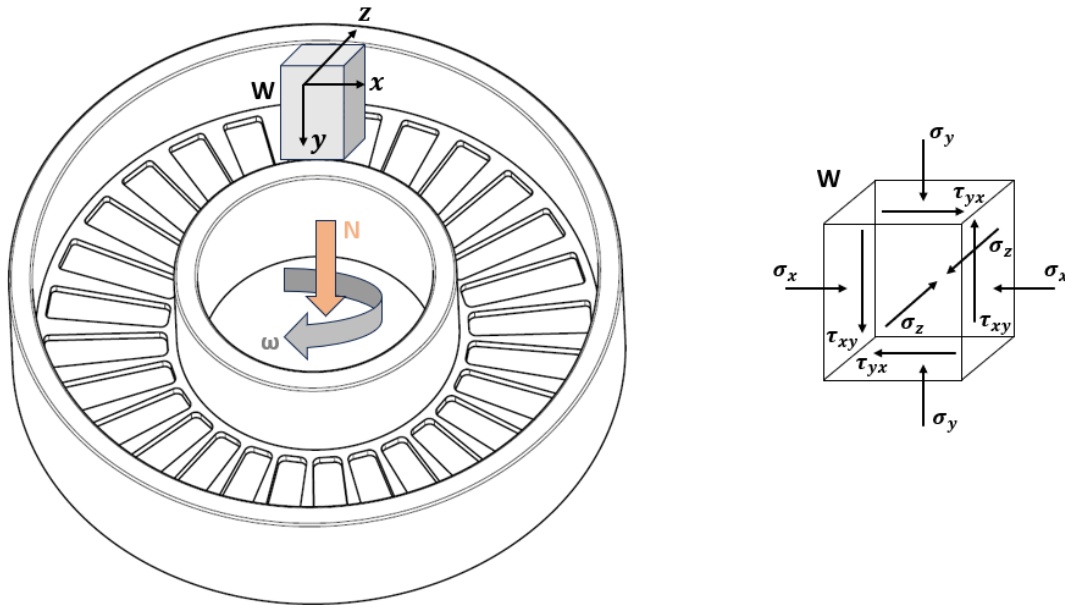
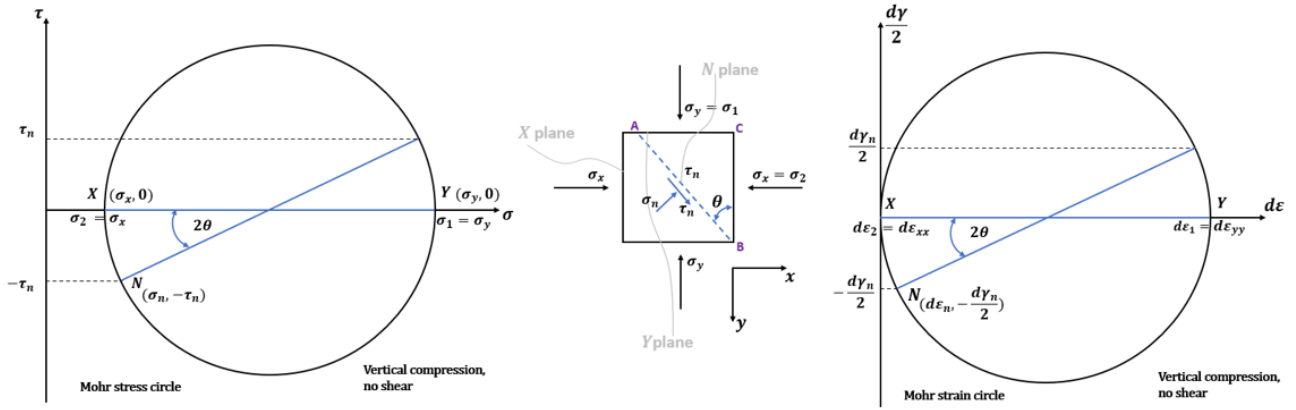
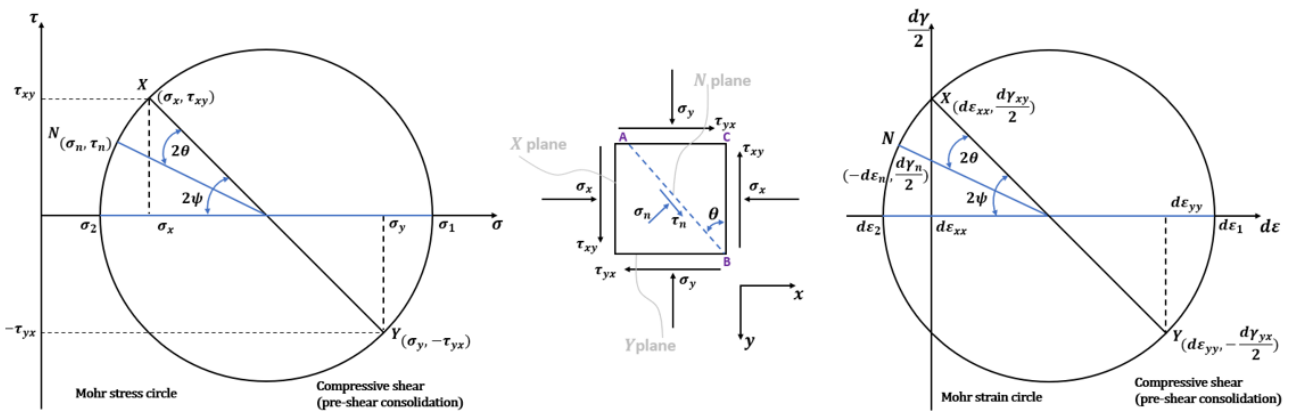


Figure 3-2: Bulk solid element in the shear cell.

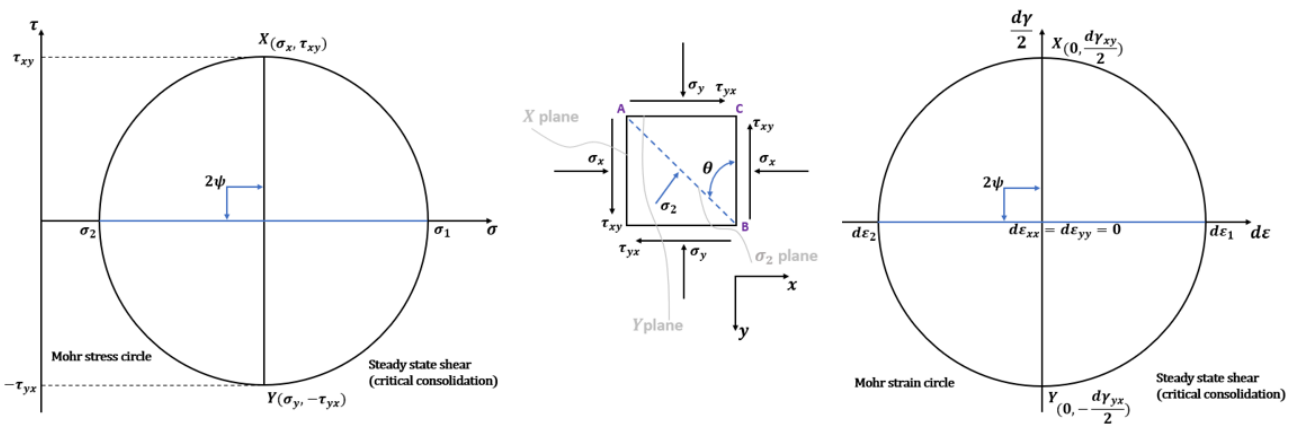
The Mohr stress and strain circles representing the stress and strain state in the bulk solid element in the $x - y$ plane are shown in Figure 3-3. The Mohr circles are constructed by considering planes of principal stress and principal strain to be coaxial. Figure 3-3a refers to vertical compression, that is a normal load applied to the lid, but no shearing action applied through trough rotation. Figure 3-3b, c, d, show the subsequent phases of shear testing in which the sample is sheared through trough rotation whilst under a normal load applied through the lid. Figure 3-3b refers to pre-shear consolidation, Figure 3-3c refers to critical consolidation, and Figure 3-3d refers to incipient failure of an over-consolidated sample. The compressive normal stresses and strains and the counterclockwise shear stresses and strains are taken as positive for constructing the Mohr stress and strain circles. The compressive strain ε_{yy} during the pre-shearing of the sample is caused by the densification of the bed with shearing.



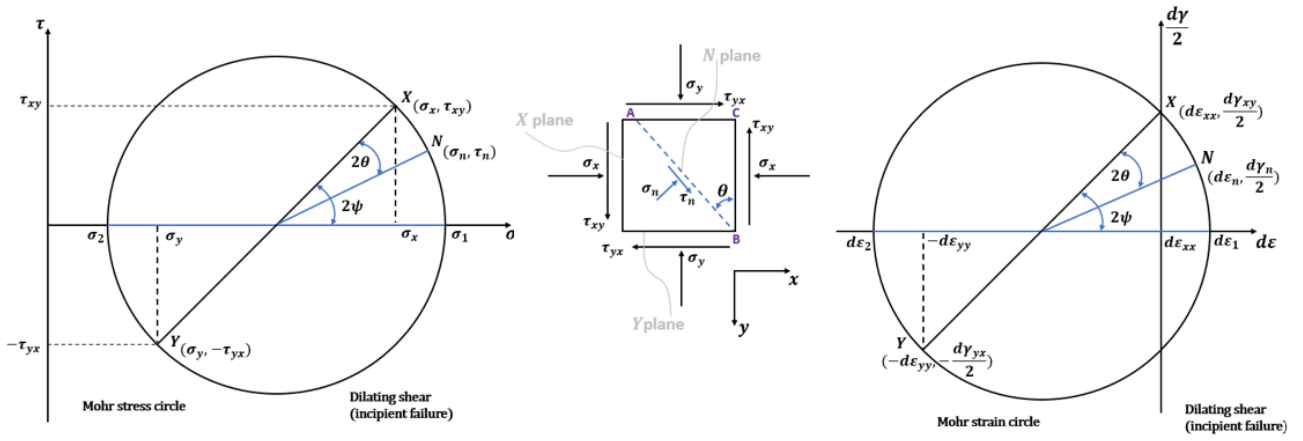
a)



b)



c)



d)

The principal plane is inclined at an angle ψ with the vertical plane. Stress and strain components for a generic plane at an angle θ to the vertical plane are reported with the footer n . The different conditions reported are, a) vertical compression, b) compressive shear, c) steady state shear ($\theta = \psi$), and d) dilating shear.

Figure 3-3: Mohr stress and strain circles.

In this reference system, the Mohr circles enable the calculation of stress and strains applied at any plane inclined at an angle θ to the vertical x plane, using the Mohr circle stress equations derived from the equilibrium of forces on the triangular bulk solid volume ABC. In the normal (Equation (3-1)) and tangential directions (Equation (3-2)), the stresses are:

$$\sigma_n = \frac{1}{2}(\sigma_x + \sigma_y) + \frac{1}{2}(\sigma_x - \sigma_y)\cos 2\theta - \tau_{xy}\sin 2\theta \quad (3-1)$$

$$\tau_n = \frac{1}{2}(\sigma_x - \sigma_y)\sin 2\theta + \tau_{xy}\cos 2\theta \quad (3-2)$$

Similarly, the normal strain ε_n and shear strain γ_n can be determined from Equations (3-3) and (3-4) respectively, describing the strain Mohr circle on the $\varepsilon - \gamma/2$ plane.

$$\varepsilon_n = \frac{1}{2}(\varepsilon_{xx} + \varepsilon_{yy}) + \frac{1}{2}(\varepsilon_{xx} - \varepsilon_{yy})\cos 2\theta - \frac{\gamma_{xy}}{2}\sin 2\theta \quad (3-3)$$

$$\frac{\gamma_n}{2} = \frac{1}{2}(\varepsilon_{xx} - \varepsilon_{yy})\sin 2\theta + \frac{\gamma_{xy}}{2}\cos 2\theta \quad (3-4)$$

Assuming coaxiality implies that the principal directions for stress and strain coincide. To determine the orientation or inclination of the principal strain plane from the x plane in a rotational shear tester, that is also the inclination by an angle ψ of the principal stress plane with respect to the x plane, incremental values of shear and normal strain can be considered at any shear condition. The sample is subjected to a constant normal load at a constant shear strain rate but changing shearing loads and normal strain occur during the test.

The shear strain increment $d\gamma_{xy}$ applied to the sample in the ring shear tester can be determined from Equation (3-5).

$$d\gamma_{xy} = \frac{r_{rad}d\theta_{ang}}{L} = \frac{r_{rad}\omega_{tr}}{L}dt \quad (3-5)$$

where $d\theta_{ang}$ is the incremental angular displacement, r_{rad} is the radial distance of the powder element under consideration from the centre of rotation, L is the instantaneous powder bed thickness, ω_{tr} is the trough angular velocity, and dt is the time increment. The normal strain increment, $d\varepsilon_{yy}$, can be determined from Equation (3-6).

$$d\varepsilon_{yy} = \frac{dV_{bed}}{V_{bed}} = \frac{1}{V_{bed}} \frac{dV_{bed}}{dt} dt = \frac{1}{V_{bed}} \frac{d(m_{bed}/\rho_b)}{dt} dt = \frac{m_{bed}}{V_{bed}} \frac{d(1/\rho_b)}{dt} dt = \rho_b \left(-\frac{1}{\rho_b^2}\right) \frac{d\rho_b}{dt} dt = -\frac{1}{\rho_b} \frac{d\rho_b}{dt} dt \quad (3-6)$$

where m_{bed} and V_{bed} are the mass and volume of the powder bed, respectively, ρ_b and dV_{bed} are the density and change in volume of the powder bed, respectively, $d\rho/dt$ is the rate of change of density with time, and dt is the time increment.

By definition, the shear strain γ_n is zero at the plane of principal strain. In the horizontal direction, $d\varepsilon_{xx}$ is assumed to be zero as the powder sample is confined by the shear cell's vertical walls. With the above constraints, the angle of inclination of the principal strain and stress plane from the x plane, ψ can be determined from Equation (3-8) by simplifying Equation (3-4).

$$0 = \frac{1}{2}(0 - d\varepsilon_{yy})\sin 2\psi + \frac{d\gamma_{xy}}{2}\cos 2\psi \quad (3-7)$$

$$\psi = \frac{1}{2}\tan^{-1}\left(\frac{d\gamma_{xy}}{d\varepsilon_{yy}}\right) \quad (3-8)$$

Shear stress τ_n at the principal plane is zero, and from Equation (3-2),

$$0 = \frac{1}{2}(\sigma_x - \sigma_y)\sin 2\psi + \tau_{xy}\cos 2\psi \quad (3-9)$$

Consequently, Equation (3-10) can be used to determine the normal stress in the horizontal direction σ_x from the applied pre-shear normal stress σ_y and the shear stress τ_{xy} measured by the shear tester during shearing

$$\sigma_x = \sigma_y - 2\tau_{xy}\cot 2\psi \quad (3-10)$$

The major principal stress σ_1 can be determined from the following Equation (3-11),

$$\sigma_1 = \frac{\sigma_y + \sigma_x}{2} + \sqrt{\left(\frac{\sigma_y - \sigma_x}{2}\right)^2 + \tau_{xy}^2} \quad (3-11)$$

When the powder is subjected to a steady state condition (also referred to as “critical state”, Figure 3-3c), the normal strain increment $d\varepsilon_{yy}$ is zero as the bulk density is considered constant at steady state, and the lid should not move in the vertical direction. For the case of steady-state shear, $\psi = 45^\circ$ calculated from Equation (3-8) is used to determine σ_1 and plot the flow function curves as per the coaxiality hypothesis. For the partially consolidated states, ψ is calculated also from Equation (3-8) and used to determine σ_1 . The

unconfined yield strengths for the steady and partially consolidated states are derived by plotting a Mohr circle tangent to the corresponding yield locus and passing through the origin, following the Jenike methodology. The second intersection point of this Mohr circle on the σ -axis gives the value of σ_c as seen in Figure 3-1.

The yield locus as per the Jenike procedure is constructed as the locus of the shear points, with it generally passing through the incipient shear points, as seen in Figure 3-1. The coaxiality hypothesis can also be applied to the incipient shear points to determine the plane of principal strain from the dilation angle and then to determine the principal stresses to construct the Mohr circle to represent the state of stress for the shear point. The yield locus can then be constructed as the envelope or common tangent line to the Mohr stress circles for incipient failure points (Hilden et al., 2008; Schulze, 2008).

Although, the displacement sensor recorded the overall variation in lid position over longer durations (typically of the order of tens of seconds) satisfactorily, helping for example in determining the density during pre-shearing, it exhibited significant fluctuations in instantaneous lid position during incipient shearing. The sensor data could not be used reliably to determine the instantaneous change in bulk density or bed dilation. The displacement sensor recorded measurements at intervals of 0.2 s; however, accurate determination of dilation requires precise capture of the small volumetric changes that occur during incipient shearing. Due to the inability of the Schulze RST displacement sensor to measure instantaneous powder bed height changes accurately, the dilation of the powder sample during incipient shearing was assumed to be negligible (i.e., zero). Considering dilation to be zero leads the principal strain and stress planes to be inclined at $\psi = 45^\circ$ to the horizontal plane. In such a condition, the horizontal and vertical normal stresses are equal, $\sigma_y = \sigma_x$. The major principal stresses can thus be determined from the normal and shear stress data at points of incipient failures from Equation (3-12).

$$\sigma_1, \sigma_2 = \sigma_y \pm \tau_{xy} \quad (3-12)$$

The yield locus can be considered to be the envelope of the incipient shear failure Mohr circles, as seen in Figure 3-4. In this study, the major principal stress during the sample consolidation (steady and partially consolidated states) was determined by using the coaxiality hypothesis. The yield locus was determined using the Jenike method and the coaxiality hypothesis. The yield locus for both cases was assumed to be linear, and the material was considered to be an ideal Coulomb material (Nedderman, 1992), where the yield criterion of the powder sample can be described by the following Equation (3-13).

$$\tau = \mu\sigma + c \quad (3-13)$$

Where τ and σ are the shear and normal stress acting on a failure plane, respectively, μ and c are the coefficient of friction and the cohesion of the material, respectively.

In the hypothetical case where dilation could be determined accurately, $\psi \neq 45^\circ$ and $\sigma_x > \sigma_y$ (Figure 3-3d), the major principal stresses would be greater in comparison to the case where $\psi = 45^\circ$. This would lead to the incipient shear failure Mohr circles in Figure 3-4 to shift towards right. Since the yield locus is constructed as the envelope of these circles, the resulting yield locus could shift downward in the low-normal-stress region, leading potentially to lower cohesion, and potentially lower unconfined yield strength. The effect of non-zero dilation on the calculated principal stresses, the resulting Mohr circles, and the corresponding yield locus was not considered in the present study and may be investigated in future work.

The stress state at failure can be represented by Mohr circles, which are tangent to the yield locus. Figure 3-1 shows the case of the yield locus according to the Jenike procedure, and Figure 3-4 shows the case of yield locus according to the coaxiality hypothesis. Yield loci can be used to determine the yield strength of the unconfined sample, σ_c . The flow functions in this study are also generated by determining the major principal stress and the unconfined yield strength from the coaxiality hypothesis. Such curves are labelled by adding coaxiality to the curve label, for example “ESKAL_500_coaxiality”. The flow functions generated by using the coaxiality hypothesis to estimate the major principal stresses and the Jenike method to determine the unconfined failure strength are labelled simply with the name of the powder, such as “ESKAL_500”.

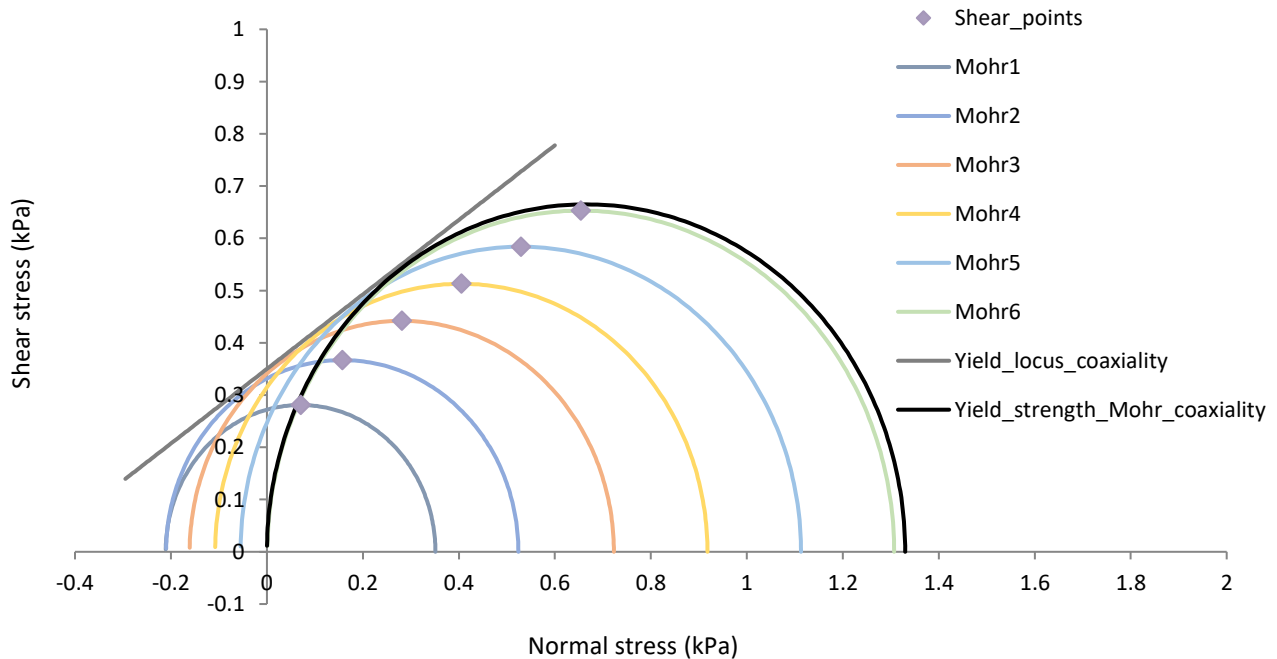


Figure 3-4: Yield locus construction using the coaxiality assumption.

The tensile normal stress needed to fracture a compacted powder sample under no shear force application is defined as the tensile strength of the powder (García-Triñanes et al., 2019). At very low normal stresses, the yield loci shift away from being a straight line and become more curved. Ashton et al. (1964) calculated the tensile strength of cohesive powders and used it to find the cohesion of the powders by interpolating between the yield locus and the tensile strength value. Ashton et al. (1965) found that the data could be well

represented by Equation (3-14), which is known as the Warren Spring equation. It can be used to fit the yield loci for cohesive powders, it is especially suited to describe the curved nature of the yield loci at lower normal stresses and to match the experimentally calculated tensile strength.

$$\left(\frac{\tau}{c}\right)^s = \frac{\sigma}{T} + 1 \quad (3-14)$$

where τ and σ are the shear and normal stress acting on a failure plane, respectively, s is the shear index whose value varies between 0 and 1, T and c are the tensile strength and cohesion of the material, respectively.

The yield locus approximated by the coaxiality hypothesis, as seen in Figure 3-4, could be well fitted by the Warren Spring equation if there is a need to calculate the tensile strength of the powder. The calculation of the yield strength of the powder by the coaxiality hypothesis would not be significantly affected by linearly approximating the yield locus (3-13), as the yield locus approximated by the Warren Spring equation is curved only for the very low normal stresses for the Mohr circles falling in the tensile stress region and is linear otherwise when the Mohr circles are in the compressive stress region.

3.3 Materials

Magnesium Carbonate (MgCO_3) and Calcium Carbonate (CaCO_3 , ESKAL 500) were used for experimentation, with their material parameters listed in Table 3-1. The particle densities of MgCO_3 (Barletta et al., 2007) and ESKAL 500 (Cabiscol et al., 2020) were 2600 kg/m^3 and 2868 kg/m^3 , respectively.

Table 3-1: Particle size distribution parameters of the tested materials at varying dispersion pressures.

Powder	Dispersion pressure (bar)	Specific Surface Area (m^2/kg)	d_{10} (μm)	d_{50} (μm)	d_{90} (μm)	Span
MgCO_3	0.5	2480	1.076	3.725	12.454	3.054
	2	2769	0.981	3.050	10.544	3.135
	3.5	3130	0.866	2.650	11.398	3.974
ESKAL 500	0.5	1810	2.529	5.316	9.392	1.291
	2	2027	2.228	4.893	8.696	1.322
	3.5	2068	2.151	4.693	8.729	1.402

The Scanning electron microscopy (SEM) images of MgCO_3 and ESKAL 500 at varying magnifications are seen in Figure 3-5 and Figure 3-6 respectively. The SEM images for both materials reveal irregular shapes, not exhibiting a repeatable particle shape. MgCO_3 is characterized by large fractions of fines. ESKAL 500 particles show a tendency to agglomerate.

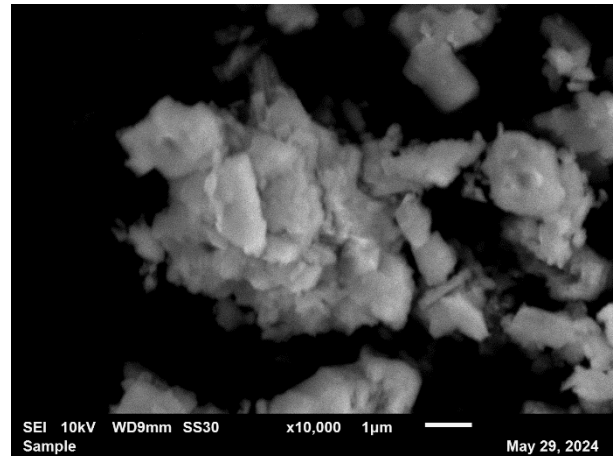
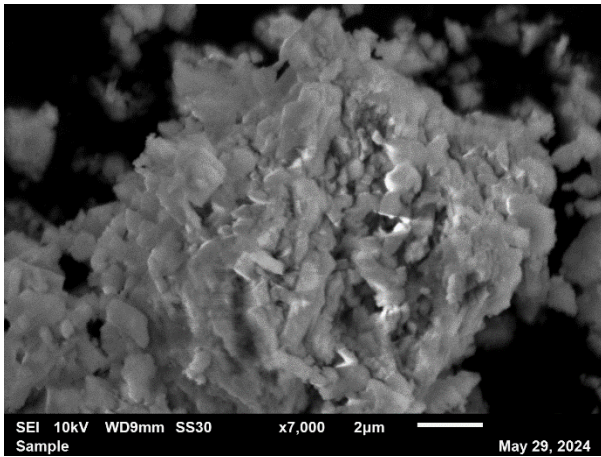
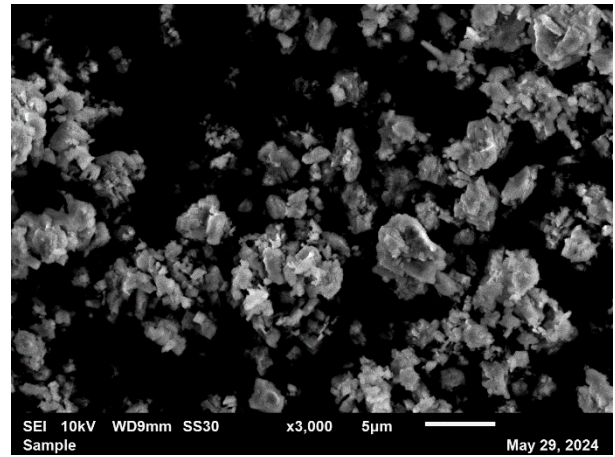
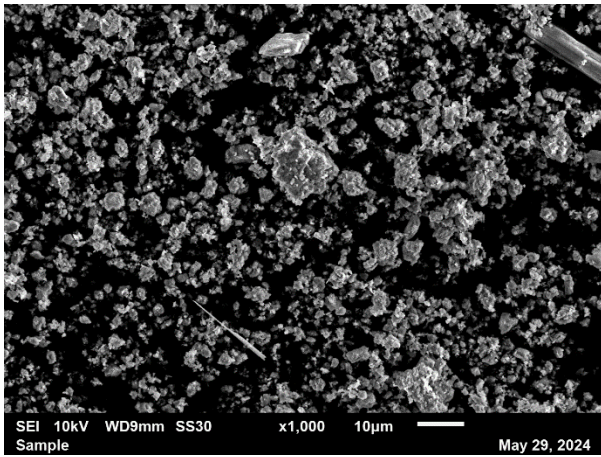
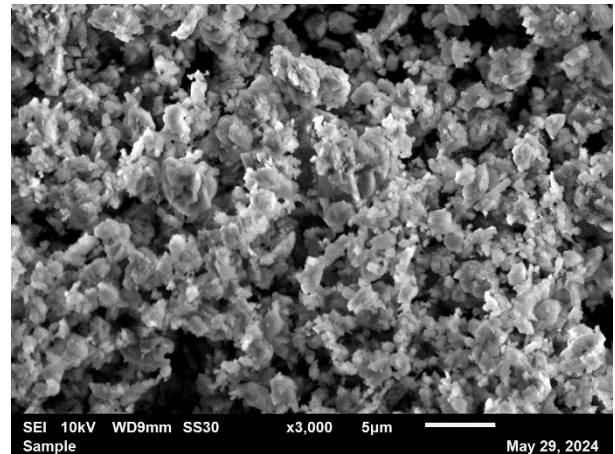
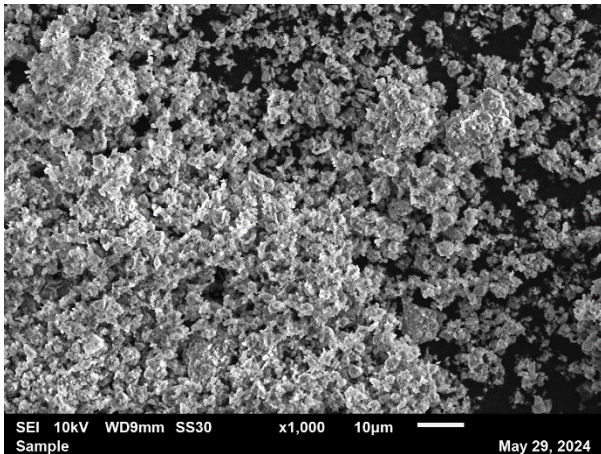


Figure 3-5: SEM images of MgCO_3 at four different magnifications: 1000x (top left), 3000x (top right), 7000x (bottom left), and 10000x (bottom right).



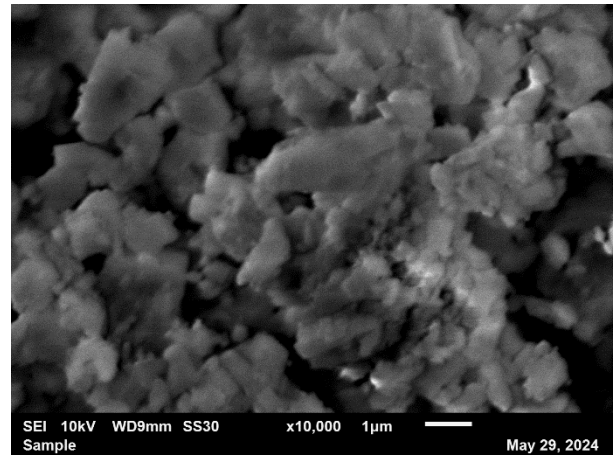
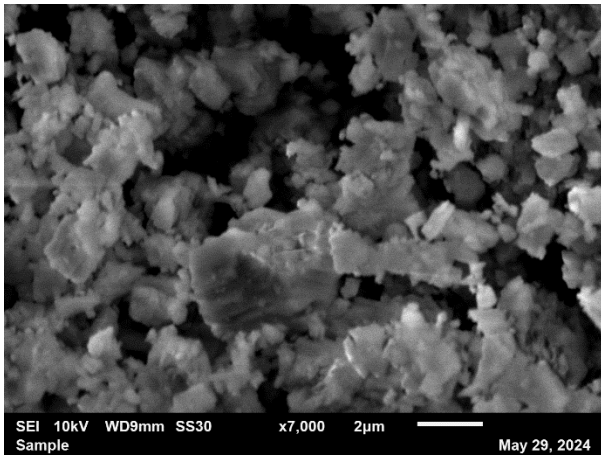


Figure 3-6: SEM images of ESKAL500 at four different magnifications: 1000x (top left), 3000x (top right), 7000x (bottom left), and 10000x (bottom right).

Figure 3-7 and Figure 3-8 show the particle size distribution (PSD) curves of MgCO_3 and ESKAL 500, respectively, at varying air pressures adopted in the dispersion device to reduce particle aggregates. PSD characterization tests were conducted on Malvern Mastersizer 3000.

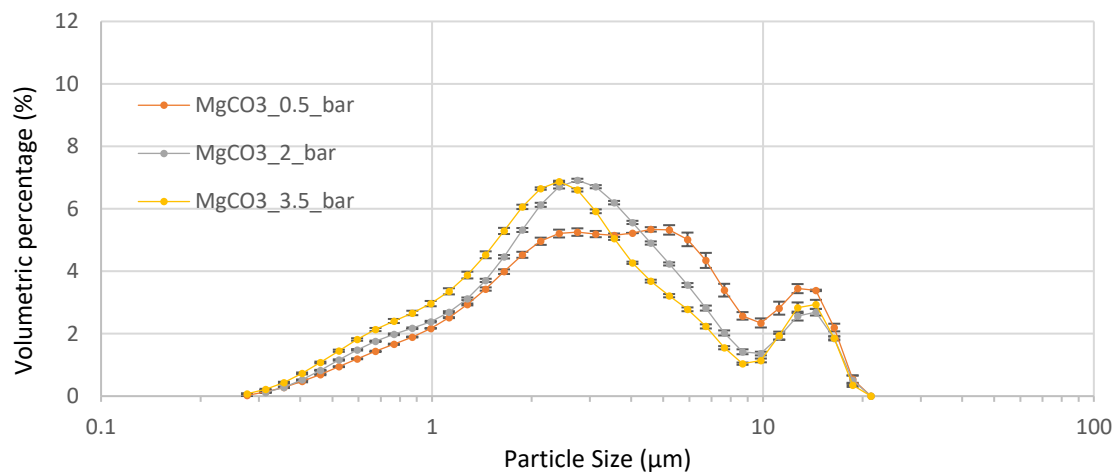


Figure 3-7: Particle size distribution curves of MgCO_3 at varying dispersion pressures.

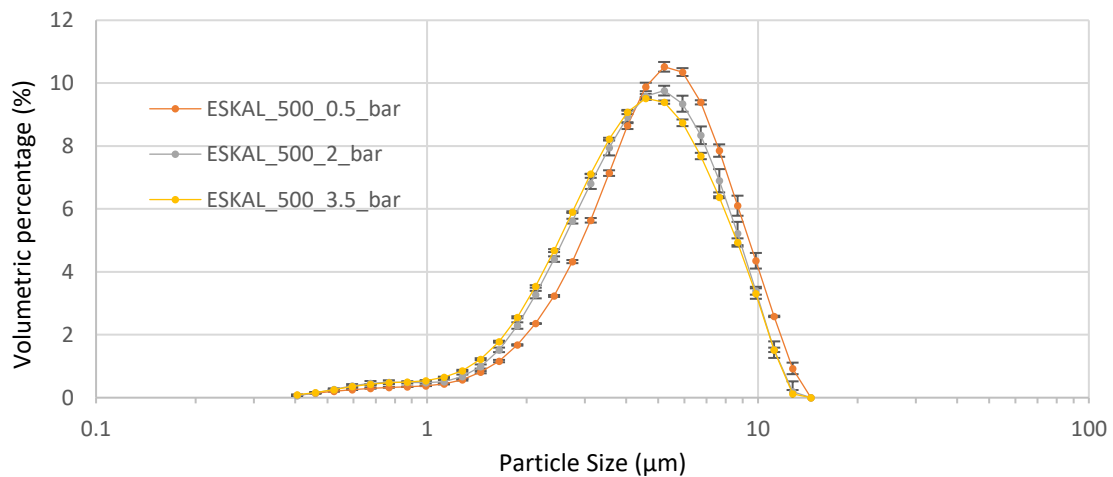


Figure 3-8: Particle size distribution curves of ESKAL 500 at varying dispersion pressures.

The PSD curves for ESKAL 500 at varying dispersion pressures exhibited a unimodal distribution; those for MgCO_3 displayed a bimodal distribution. Coherently with what was previously observed in SEM images, the particle size distributions in Figure 3-7 and Figure 3-8, and Table 3-1 indicate more fines in the MgCO_3 powder than in the ESKAL 500 powder. It also appears that the dispersion pressure of 0.5 bar was insufficient to fully disperse the MgCO_3 powder.

3.4 Flow behaviour characterization through shear testing under varying consolidation states

3.4.1 Schulze Ring Shear Tester (RST) apparatus

The Schulze RST is a direct shear tester used to measure the flowability of the powder sample. The manual Schulze RST, “RST-01”, was utilised in this work to measure the flowability of the two powders. The powder sample is loaded into the shear cell, which consists of an annular-shaped bottom ring, a trough, and an annular lid, as shown in Figure 3-9. Grooves on the trough bottom and vanes on the lid prevent wall shear of the sample during the test. The trough can be rotated by an electric motor connected to its base. The lid is joined to a cross beam to apply a normal load to the powder sample. Weights can be mounted on a hanger rod hooked onto the cross beam to impose a normal load on the powder specimen through the lid. The cross beam is also connected to two tie rods, each of which is connected to its respective load cell fixed on the testing bench. The weight of the lid connected to the tie rods, screw fasteners, the hanger rod, and the cross beam is exactly balanced by an adjustable counterweight exerting a lifting force F_a on the cross beam through a hook connection. Thus, the normal stress applied to the powder depends only on the load applied by the weight on the hanger, and the minimum load is independent of the lid weight. Once the powder sample is filled into the bottom ring and placed on the rotating base of the tester bench, the lid is connected to the cross beam, which is hooked to the counterbalance weight. Next, the weight hanger rod is hooked onto the cross beam, and the tie rods are placed to connect the cross beam to the load cells. The lid is then gently lowered into the powder sample and loaded with the normal load required for pre-shearing the sample, allowing the lid vanes to penetrate the sample. The motor is started to rotate the trough whilst keeping the lid rotationally fixed in place. The relative motion between the bottom and the lid applies a shear deformation to the powder sample. The couple necessary to keep the lid in place is measured by the load cells connected to the crossbeam via the two tie rods. The measured couple allows for determining the average shear stress experienced by the material in the volume between the tip of the vane and through the bottom. The vertical displacement of the lid is tracked by a displacement sensor which helps to determine the sample volume and the resulting bulk density ρ_b . Further details about the setup and the shear testing methodology can be found in (Barletta et al., 2019) and (Schulze, 2008).

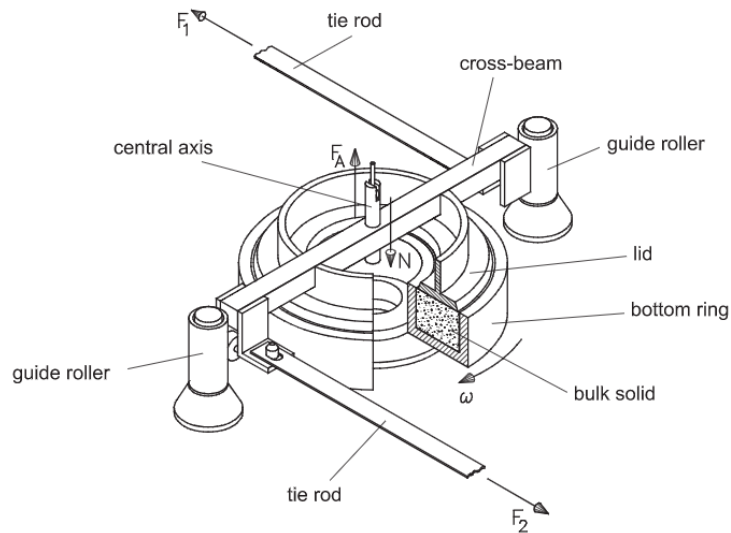


Figure 3-9: Schulze Ring Shear Tester (Schulze and Wittmaier, 2003).

3.4.2 Testing methodology on Schulze RST

The manually operated Schulze Ring Shear Tester (RST) was utilised to characterise the flowability of the powders at steady and partially consolidated states. The small (S) and medium (M) shear cells of the Schulze RST were used. Before each experiment, to ensure the powders to be moisture-free, they were dehydrated overnight in a furnace at 110°C. To break any large agglomerates and relieve the powder stress history, for the sample preparation, the shear cell was filled by first sieving the powder through a 300 μm sieve tray.

The manual version of Schulze RST enables control over the normal stress and monitoring of the shear stress subjected to the powder during the pre-shear step. Also, the rotational movement of the shear cell is manually controlled. These tester features enable the resulting shear stress to be read at any moment during the pre-shear stage and stop the pre-shear when the shear stress has reached an intended fraction of the steady state shear stress value. Applying this procedure at a predefined fraction κ of the steady state shear stress allows for shearing a partially consolidated powder and evaluating its flow behaviour with the shear step.

For testing a powder at the partially consolidated state, the powder is subjected to the same pre-shear normal stress as in the case of the steady state, as seen in Figure 3-10, but the shearing of the sample is stopped before the sample reaches the steady state such that the shear stress in the partially consolidated state is a fraction of the shear stress obtained in the steady state. This procedure allows bringing the sample to a reproducible, partially consolidated state. Afterwards, as in the standard procedure, the sample is sheared after applying a normal stress lower than the pre-shear normal stress. The peak shear stress is used to obtain a point of the yield locus. The whole yield locus is obtained by repeating the procedure with different normal stresses applied during the shear phase.

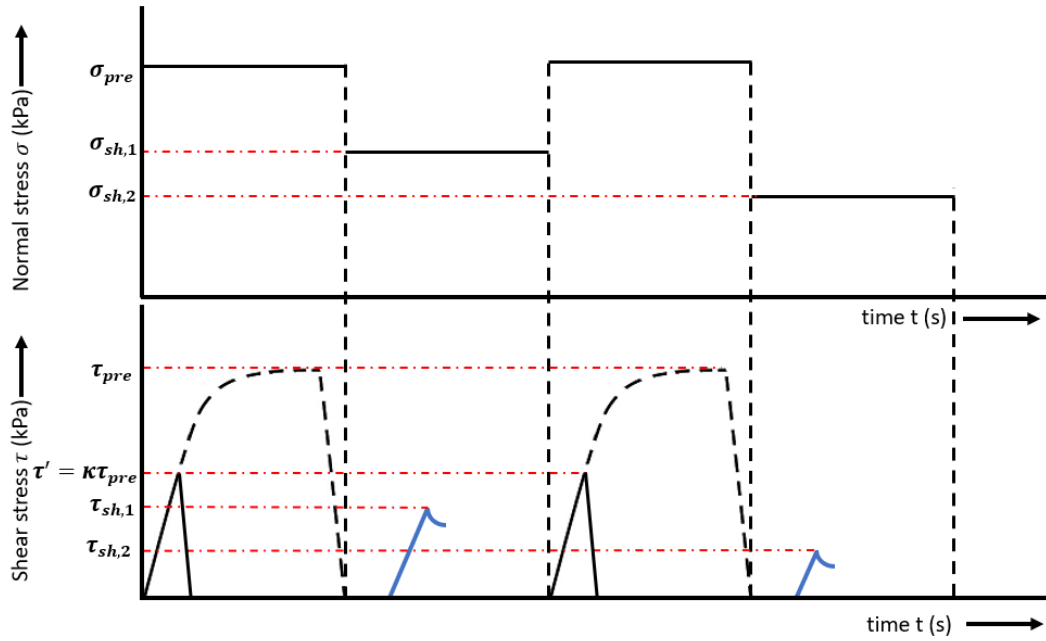


Figure 3-10: Shear test to obtain yield loci at under-consolidated or partially consolidated states.

MgCO₃ and ESKAL 500 were tested according to the Jenike methodology to obtain the flow function curves. The pre-shear normal loads of 1.5 kg (1.858 kPa), 1.2 kg (1.486 kPa), 1 kg (1.239 kPa) and 0.7 kg (0.867 kPa) were applied to the powder samples in the Schulze RST small (S) shear cell, and 1.5 kg (0.651 kPa), 1.2 kg (0.521 kPa), 1 kg (0.433 kPa) and 0.7 kg (0.303 kPa) in the Schulze RST medium (M) shear cell. The resulting pre-shear normal stress acting on the sample in the shear zone is higher than the applied pre-shear normal load because of the additional stress applied by the material inside the shear lid vanes. Each test conducted at the pre-shear normal load produced a yield locus, and these eight tests across the two shear cells (S & M) were used to determine the flow function curves for the two materials. Tests at the partially consolidated state were also conducted. After obtaining the flow function at the critical consolidation or the steady state, tests with the same pre-shear normal loads were repeated by stopping the pre-shear when registering a shear torque equal to 0.75 or 0.5 times the steady-state values. The shear stress subjected to the sample is to be 75% and 50% of the pre-shear shear stress τ_{pre} . This condition is reported in the sketch of Figure 3-10 as $\tau' = \kappa\tau_{pre}$. All the test conditions were repeated thrice for both the steady and partially consolidated states. As an example, Figure 3-11 reports shear testing raw data for ESKAL 500 at 1.5 kg pre-shear normal load (1.858 kPa) tested on the S cell for the steady state (labelled SS) and 50% partially consolidated state (labelled 0.50). Differences in the shear stresses at the end of the pre-shear step and shear steps are clearly visible. The bulk density ρ_b of the powder tested at the steady state is higher than that of the partially consolidated powder at the end of the pre-shear step.

The complete shear test experiments involved multiple pre-shear and shear-to-failure steps, with the first shear cycle (pre-shear + shear to failure) completed approximately around data point 3000 and subsequent cycles continuing until approximately data point 6500. Consequently, some degree of particle attrition or

morphological evolution may have occurred during repeated shearing. However, the applied pre-shear normal load (1.858 kPa) was relatively low, and therefore significant particle breakage is not expected under the present testing conditions, although minor surface abrasion, asperity smoothing, or deagglomeration effects cannot be ruled out. Such effects, if present, could contribute to gradual changes in packing behaviour and bulk density during the later stages of testing. A detailed investigation of particle morphology evolution and breakage during repeated shear cycling was beyond the scope of the present work and could form part of a future study.

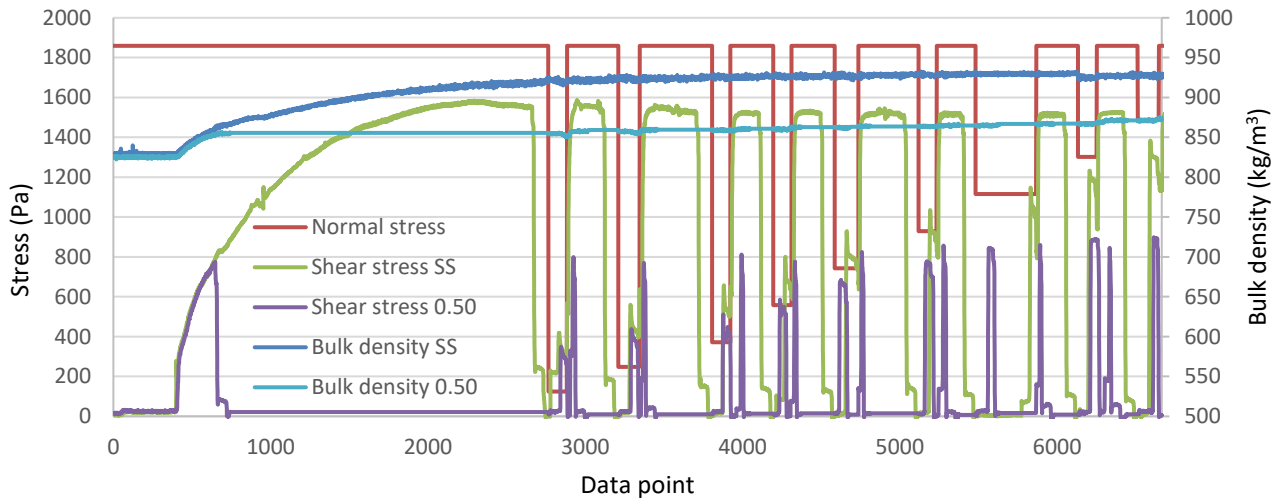


Figure 3-11: Shear testing raw data of ESKAL 500 at steady and partially consolidated states.

Table 3-2 reports the results from the shear tests conducted on MgCO_3 and ESKAL 500 at the steady state with the Jenike shear testing methodology and using it to determine σ_1 and σ_c . ϕ_e and ϕ_{lin} represent the slopes of the effective and the linearised yield locus as shown in Figure 3-1.

Table 3-2: Results obtained from standard shear testing (Jenike methodology).

MgCO ₃ (steady state)						
σ_{pre} (kPa)	σ_1 (kPa)	σ_c (kPa)	Std. dev. σ_c (kPa)	ρ_b (kg/m ³)	ϕ_e (°)	ϕ_{lin} (°)
1.896	4.191	2.346	0.064	938	51.3	34.2
1.523	3.46	2.08	0.132	906	53.5	34.9
1.274	2.857	1.848	0.081	883	54.3	33.1
0.901	1.995	1.422	0.051	842	58.1	33.8
0.683	1.431	0.934	0.092	827	53.3	31.3
0.552	1.13	0.742	0.006	803	53.5	31.2
0.465	0.965	0.705	0.004	780	57.6	31.5
0.333	0.694	0.509	0.029	747	57.8	31.6

ESKAL 500 (steady state)						
σ_{pre} (kPa)	σ_1 (kPa)	σ_c (kPa)	Std. dev. σ_c (kPa)	ρ_b (kg/m ³)	ϕ_e (°)	ϕ_{lin} (°)

1.896	3.834	1.368	0.033	920	44.3	34.5
1.523	3.042	1.227	0.036	899	44.6	33.1
1.274	2.591	1.09	0.023	881	44.9	32.8
0.901	1.829	0.907	0.009	845	48.1	33.3
0.683	1.373	0.807	0.006	822	48.7	29.2
0.552	1.142	0.716	0.018	805	50	28.4
0.465	0.966	0.643	0.011	788	51.5	27.7
0.334	0.696	0.506	0.005	760	54.9	27.6

3.4.3 Effect of consolidation state on yield loci and powder flowability

Figure 3-12 presents the yield loci and the Mohr circles constructed to determine the unconfined yield strength determined using the approach coherent with the coaxiality hypothesis and the Jenike method. As can be seen, the cohesion and unconfined failure strength determined by applying the coaxiality hypothesis is higher than the values calculated by the Jenike method. Figure 3-13 shows additionally the construction of the Mohr circles to determine the major principal stress acting in the critically consolidated sample. As can be seen, the Mohr circle for the coaxiality approach has its apex as the pre-shear point. The Mohr circle drawn according to the Jenike method is tangent to the yield locus and passes through the pre-shear point, with these conditions putting the pre-shear point lying left to the apex of the Mohr circle. As a result, the major principal stress calculated by the Jenike method is higher than the one determined from the coaxiality approach. Schulze (2008) noted that the coaxiality approach can lead to an overestimation of the unconfined yield strength, the yield strength determined from the Jenike method has proven to provide safe silo design results leading to outlet dimensions which avoid arching. Therefore, the Jenike method can be preferred over the coaxiality approach to estimate the material's failure strength for both critically and partially consolidated powders. However, for reasons discussed earlier the use of coaxiality approach is preferred over the Jenike approach in determining the major principal stress for powders under partial consolidation.

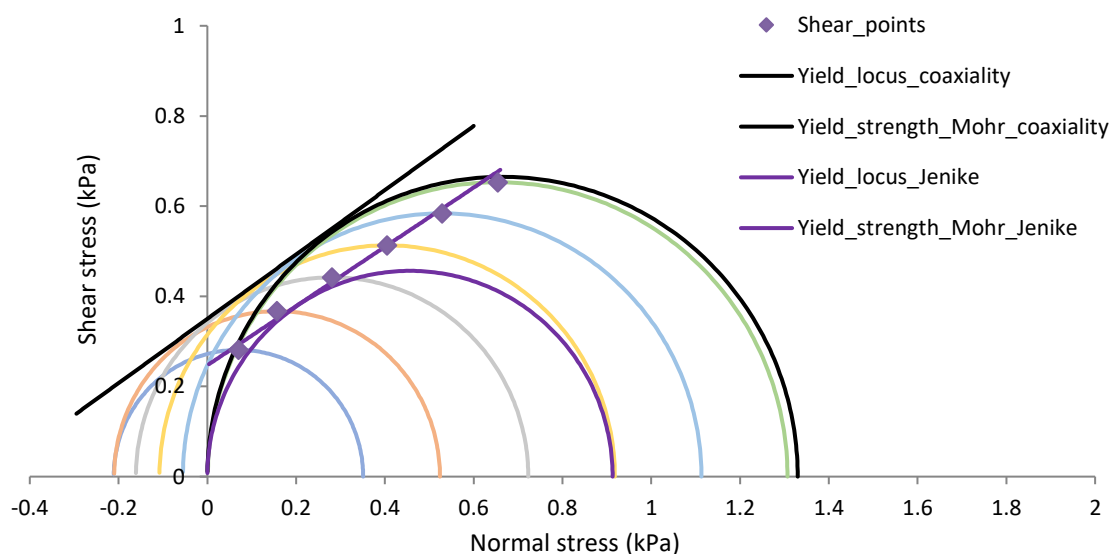


Figure 3-12: Yield locus construction by coaxiality hypothesis and Jenike method (ESKAL 500).

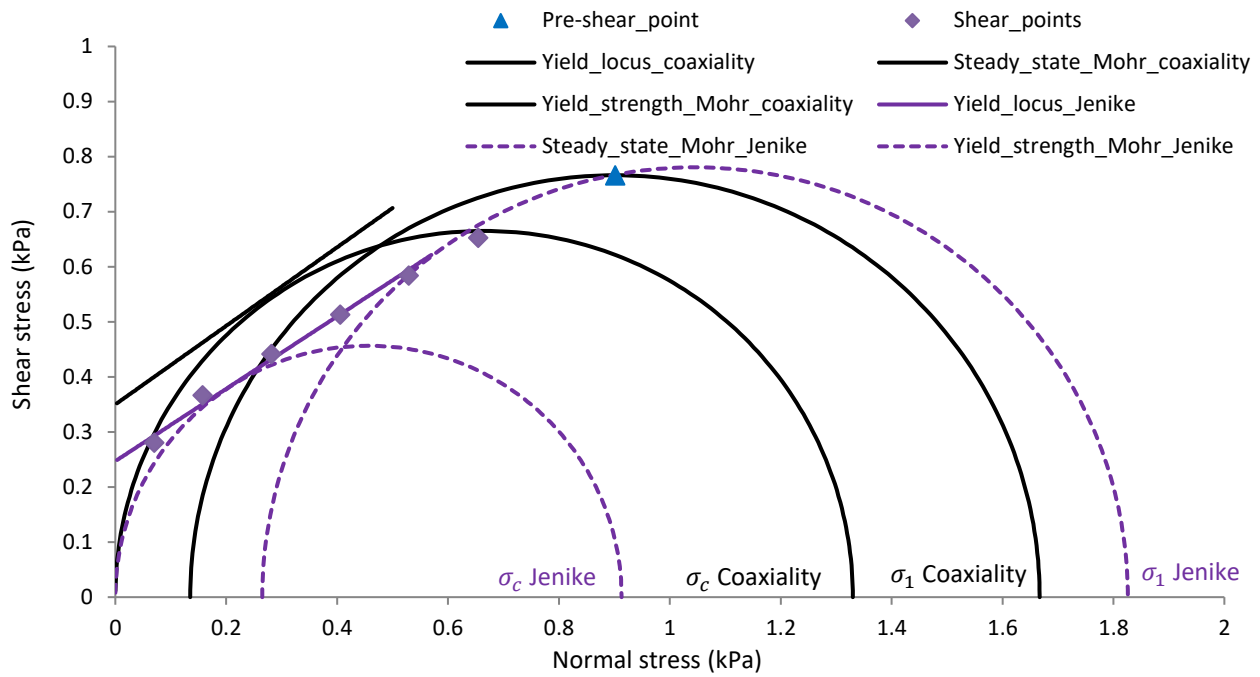
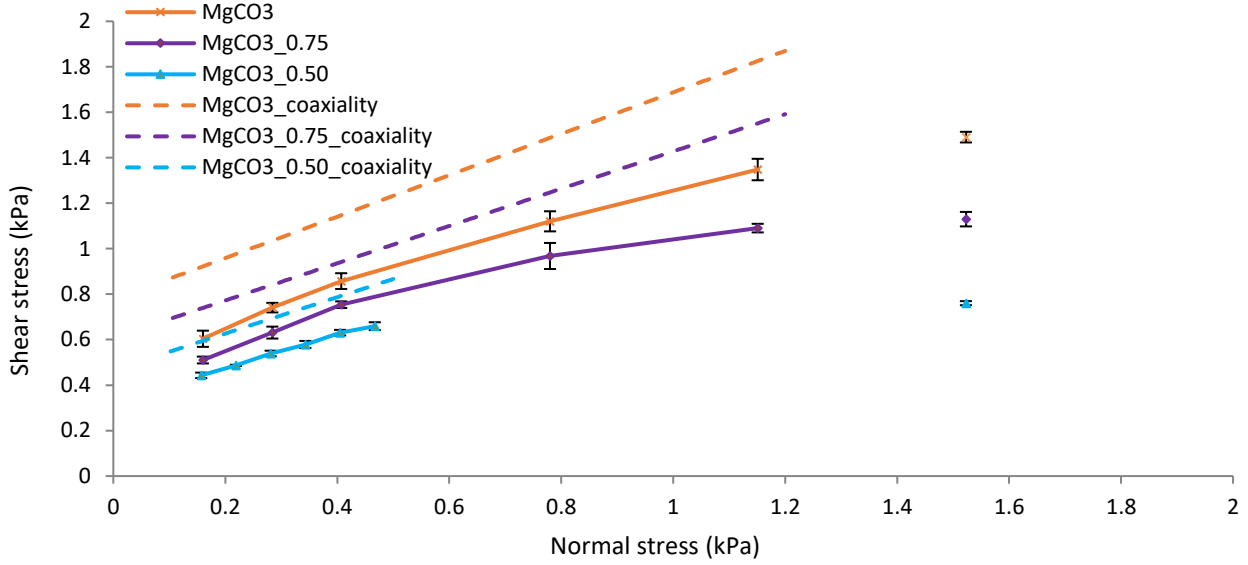


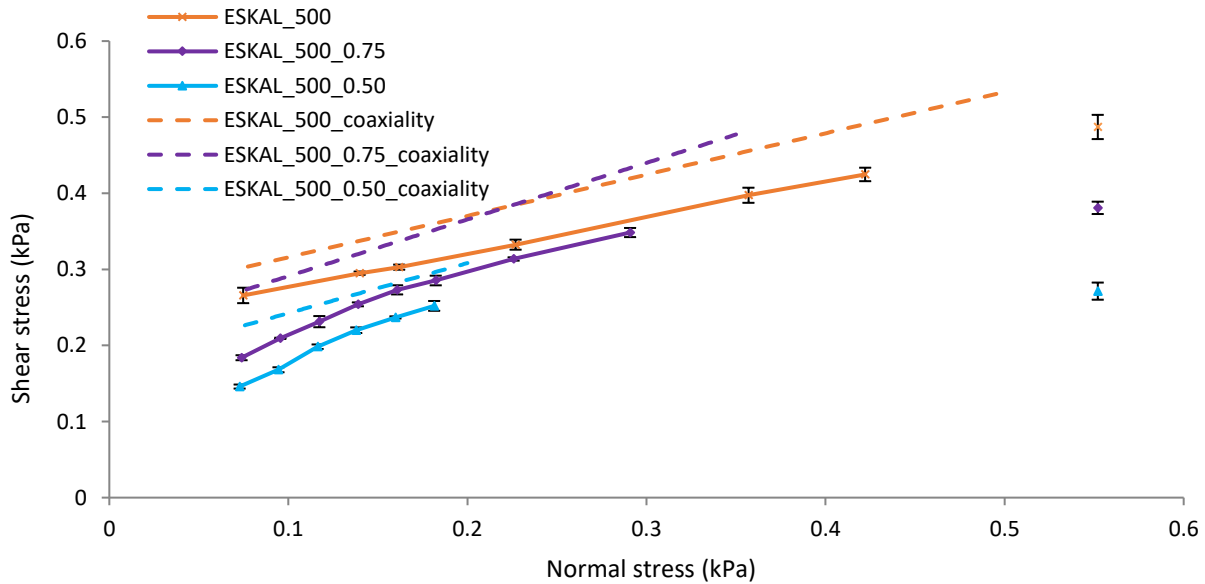
Figure 3-13: Determination of major principal stress during consolidation and unconfined yield strength of the over-consolidated powder by coaxiality hypothesis and Jenike method (ESKAL 500).

Figure 3-14 and Figure 3-15 report the yield loci at varying consolidation states for MgCO_3 and the ESKAL powders, respectively. The error bars in Figure 3-14 (and all subsequent Figures where error bars are used in this thesis) represent $\pm$ one standard deviation of the measured values. For each data point, the standard deviation was determined using the corresponding set of repeated measurements, and the positive and negative error bars indicate the spread of the measured values about the mean. The yield loci are drawn according to the Jenike method, and by applying the coaxiality hypothesis. Inspection of these Figures shows that yield loci are dependent on the state of consolidation. The shear stress necessary to cause incipient failure in the powder at a particular normal stress value reduces as we move away from the critical consolidation towards the partially consolidated states. The yield locus is a critical output of the shear test, and parameters such as the cohesion c , slope of the linearized yield locus ϕ_{lin} and unconfined yield strength σ_c can be obtained from the yield locus and the Mohr Circle corresponding to unconfined yield, as seen in Figure 3-1, which helps compare the cohesive nature of the powder. Coherently with the example in Figure 3-12, yield loci calculated according to the coaxiality hypothesis provide significantly higher yield values at comparable normal stress than yield loci obtained according to the Jenike method. Differences in yield between these two methods appear to be more related to a different estimation of the yield locus intercept, the cohesion, rather than to its slope, the internal friction angle.



✕, MgCO₃ (steady state); ◆, MgCO₃_0.75 (75% partially consolidated state); ▲, MgCO₃_0.50 (50% partially consolidated state); —, MgCO₃_coaxiality (steady state); —, MgCO₃_0.75_coaxiality (75% partially consolidated state); —, MgCO₃_0.50_coaxiality (50% partially consolidated state).

Figure 3-14: Yield loci curves of MgCO₃ tested on S cell at 1.52 kPa pre-shear normal stress, according to the Jenike method and the coaxiality hypothesis.

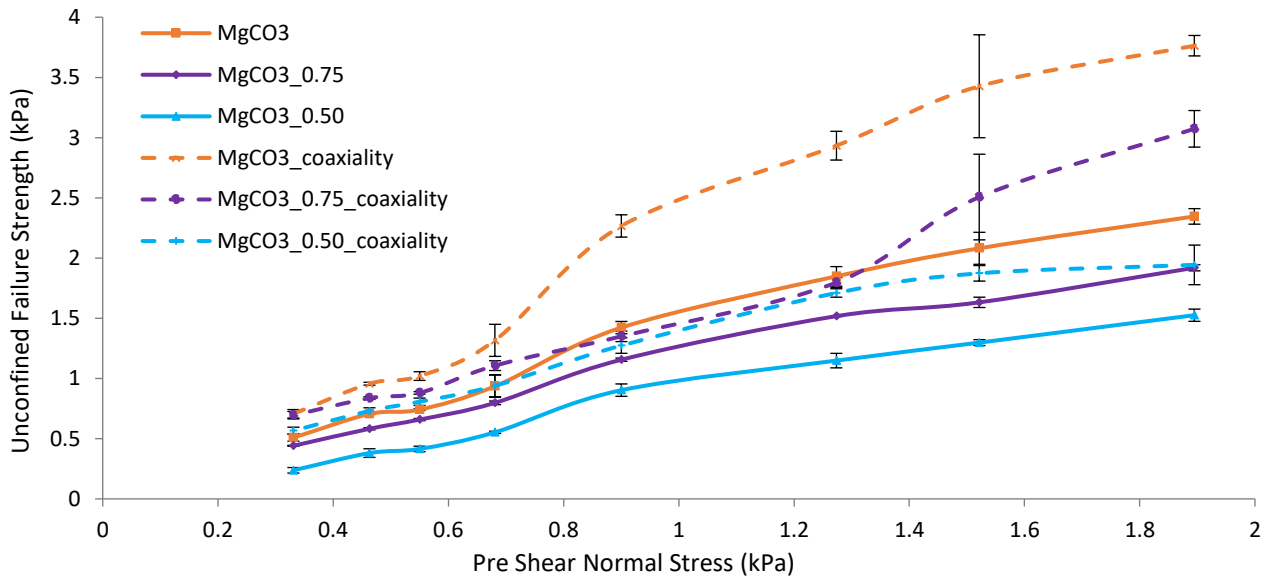


✕, ESKAL_500 (steady state); ◆, ESKAL_500_0.75 (75% partially consolidated state); ▲, ESKAL_500_0.50 (50% partially consolidated state); —, ESKAL_500_coaxiality (steady state); —, ESKAL_500_0.75_coaxiality (75% partially consolidated state); —, ESKAL_500_0.50_coaxiality (50% partially consolidated state).

Figure 3-15: Yield loci curves of ESKAL 500 tested on M cell at 0.55 kPa pre-shear normal stress, according to the Jenike method and the coaxiality hypothesis.

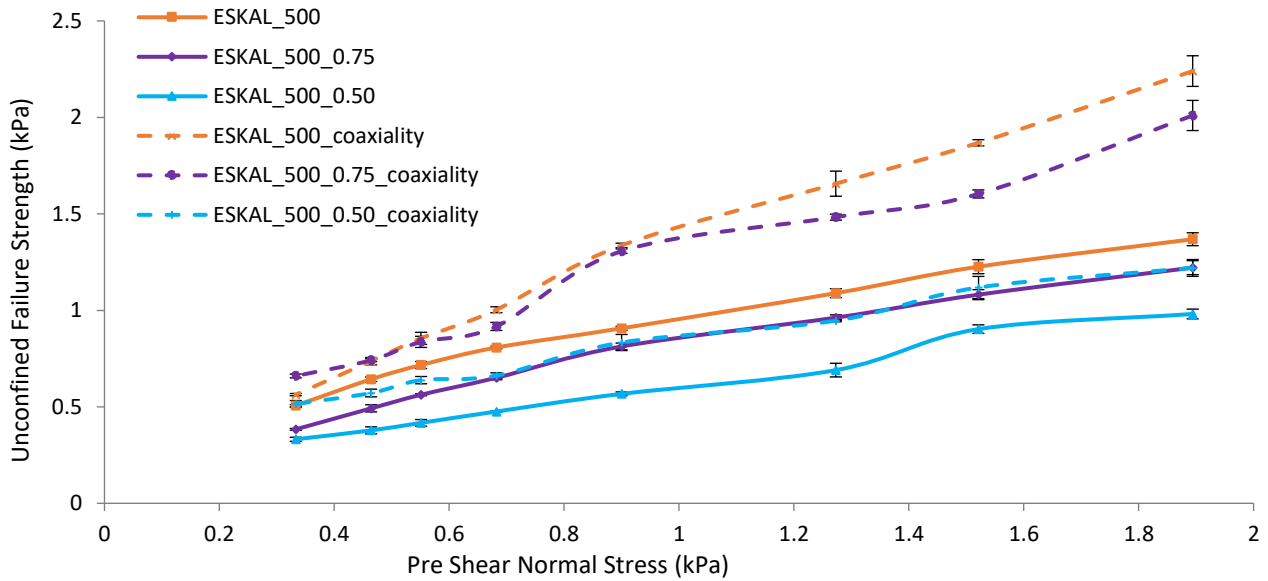
The unconfined yield strength for both powders is presented in Figure 3-16 and Figure 3-17 as a function of the pre-shear normal stress for the different states of consolidation. The Figures show a significant

dependence of yield strength on the state of consolidation. For both materials, the critically consolidated powder had higher failure strength values than the 50% partially consolidated state and the 75% partially consolidated state is intermediate between the two. The unconfined yield strength values for both powders, estimated by the coaxiality hypothesis at varying states of consolidation, were higher than the corresponding values calculated using the yield loci constructed by the Jenike method. Figure 3-12 and Figure 3-13 report a typical case from which it appears that the Mohr circle for the unconfined yield, which needs to pass through the origin and be tangent to the yield locus, is much smaller if drawn according to the Jenike method than according to the coaxiality hypothesis.



■, MgCO₃ (steady state); ◆, MgCO₃_0.75 (75% partially consolidated state); ▲, MgCO₃_0.50 (50% partially consolidated state); ×, MgCO₃_coaxiality (steady state); ●, MgCO₃_0.75_coaxiality (75% partially consolidated state); +, MgCO₃_0.50_coaxiality (50% partially consolidated state).

Figure 3-16: Unconfined failure strength curves of MgCO₃, according to the Jenike method and the coaxiality hypothesis.



■, ESKAL_500 (steady state); ◆, ESKAL_500_0.75 (75% partially consolidated state); ▲, ESKAL_500_0.50 (50% partially consolidated state); ×, ESKAL_500_coaxiality (steady state); ●, ESKAL_500_0.75_coaxiality (75% partially consolidated state); +, ESKAL_500_0.50_coaxiality (50% partially consolidated state).

Figure 3-17: Unconfined failure strength curves of ESKAL 500, according to the Jenike method and the coaxiality hypothesis.

Independent of the method to estimate yield loci and unconfined yield strength, a suitable explanation of the changing properties during the consolidation process can be provided by hypothesising that, when a powder sample is partially consolidated or in the process of achieving critical consolidation, the number of contacts between the particles is limited, as the particles are still in the process of rearrangement. As a result, they face lower resistance to flow. Hence, this leads to lower cohesion and lower unconfined yield strength. In the process of activation and increase of the contacts between the particles, the friction and the cohesive resistance to flow increase, resulting in increasing cohesion and unconfined yield strength. The shear stress of the powder progressively increases when the powder achieves the 50% partially consolidated state, the 75% partially consolidated state, and the steady state. This increase in the number of interparticle contacts can be expressed in terms of the coordination number, which increases with denser packing, reaching a maximum at steady state, and can be associated with increased particle cohesion, higher shear stress, and higher failure strength. DEM simulations of the powder at steady and partially consolidated states can help shed light on this hypothesis.

Table 3-3 presents the data for powder bed compression dL during the shearing of the sample and the final height of the powder bed column for ESKAL 500 tested on the S cell. The powder bed compression dL for steady state across the varying pre-shear normal loads is greater than the compression seen for the 75% partially consolidated state, with this value being the least for the 50% partially consolidated state. The powder bed column height, which is the final height of the specimen after shearing, is the least for the steady state and the greatest for the 50% state, with the 75% state lying between the two, thus indicating a denser

packing for the steady state than the partially consolidated states and potentially a higher coordination number.

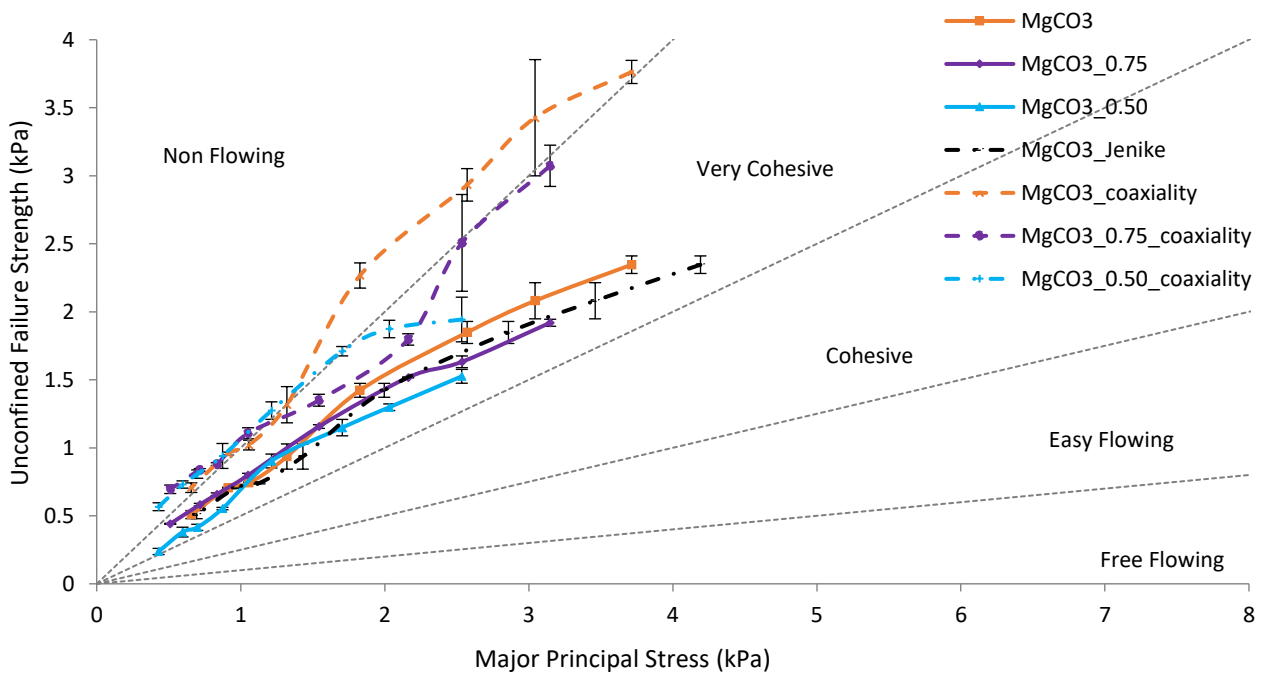
Table 3-3: Powder bed compression during shearing of ESKAL 500.

	Steady state			75% partially consolidated state			50% partially consolidated state		
Pre-shear normal load (kPa)	Mass of sample (g)	dL (mm)	Powder bed column height (mm)	Mass of sample (g)	dL (mm)	Powder bed column height (mm)	Mass of sample (g)	dL (mm)	Powder bed column height (mm)
1.858	133	1.41	19.3	134	1.13	19.6	136	0.634	20.4
1.486	131	1.45	19.6	137	1.07	20.6	138	0.549	21.0
1.239	134	1.32	20.3	138	1.06	21.1	134	0.611	21.1
0.867	133	1.42	20.9	136	1.12	21.5	136	0.589	22.0

The flow functions depicting the relationship between the unconfined yield strength and the major principal stress for MgCO_3 and ESKAL 500 are shown in Figure 3-18 and Figure 3-19 respectively. The major principal stresses of the flow functions of MgCO_3 and ESKAL 500, were evaluated both following the Jenike method and the coaxiality hypothesis. The dashed curves labelled Jenike in Figure 3-18 and Figure 3-19 represent the steady-state flow function with the major principal stresses and unconfined yield strength obtained according to the Jenike methodology. The data points of the curves labelled simply by the powder name (for example, ESKAL_500_0.50 or MgCO_3 _0.75) are obtained using the coaxiality hypothesis applied to determine the stress state at consolidation in order to determine the major principal stresses, and the Jenike approach to determine the yield locus and the unconfined failure strength. The flow functions derived following Jenike's methodology indicate higher σ_1 values at corresponding σ_c values when compared to their coaxiality counterparts. The higher σ_1 values for the Jenike flow functions appears clearly in the example provided in Figure 3-13 which is generally representative of the differences found between the Jenike method and the application of the coaxiality approach. In the application of the Jenike method, the apex of the steady state Mohr circle is generally to the right of the pre-shear point, whereas, assuming coaxiality, the apex of the Mohr circle is forced at the pre-shear point ($\psi = 45^\circ$ calculated from Equation (3-8), leading to lower σ_1 than found with the Jenike method. For the partially consolidated states, ψ is calculated also from Equation (3-8) and used to determine σ_1 . The angle ψ , averaged to be approximately 40° for the partially consolidated state with 75% of the pre-shear shear stress, and about 33° for the partially consolidated state with 50% of the pre-shear shear stress. Figure 3-18 and Figure 3-19 also present the flow functions obtained using the coaxiality hypothesis to determine both the major principal stresses and failure strength; such curves are labelled "coaxiality". As noted by Hilden et al. (2008), the flow functions obtained according to the coaxiality hypothesis provide a more conservative description of powder flow than those determined according to the Jenike method. In fact, as exemplified in Figure 3-13, the use of the procedure coherent with the coaxiality

hypothesis leads to estimating higher σ_c values for lower σ_1 values for the same set of experiments made by a single pre-shear normal stress and a complete set of shear normal stress, than the σ_c and σ_1 values obtained with the Jenike method. Comparing the flow functions in Figure 3-18 and Figure 3-19 at the relevant consolidation state shows that the flow function determined by the coaxiality approach (for both σ_1 and σ_c) is more conservative or has worse flowability than the flow function determined by the coaxiality approach for calculating σ_1 and Jenike method for σ_c .

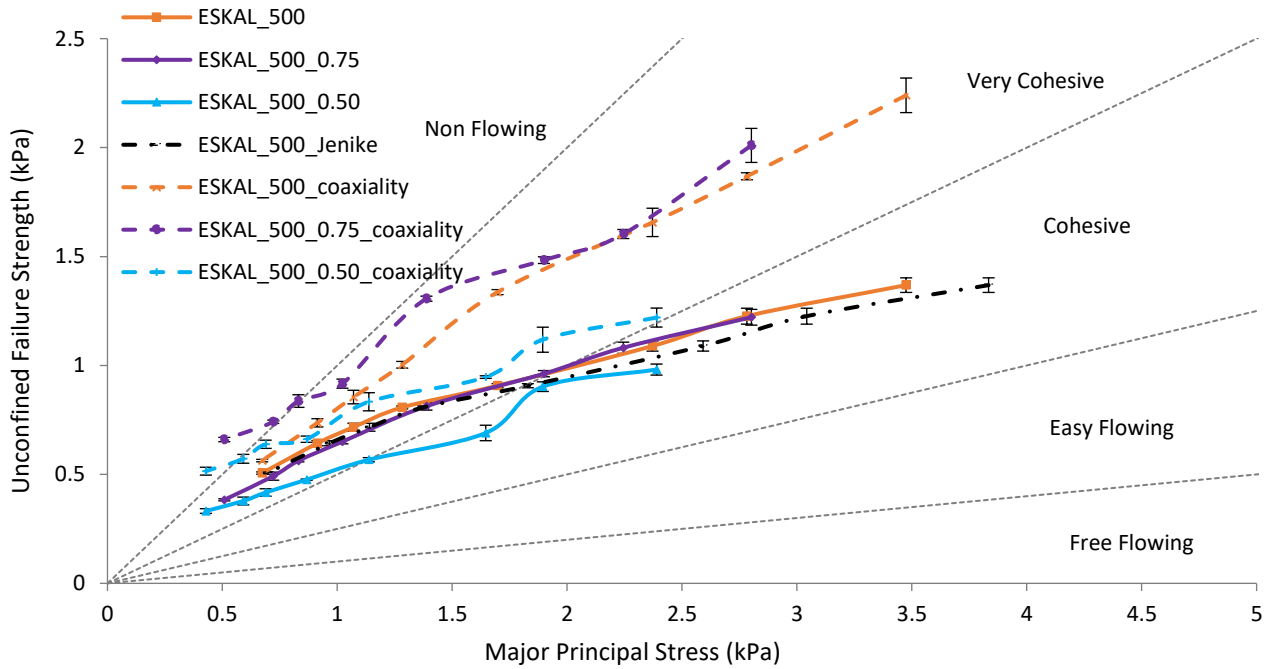
The flow function curves for MgCO_3 (solid curves: coaxiality approach for calculating σ_1 and Jenike method for σ_c), as shown in Figure 3-18, indicate that the flowability of the powders at lower major principal stresses, when deformed to a partially consolidated state (or under-consolidated state), showed no significant difference in flowability. At the higher major principal stresses, the flow function with a pre-shear shear stress equal to 75% of the steady state shear stress, referred to as “75% partially consolidated state”, lies between the curves of the steady state and that obtained with a pre shear stress equal to 50% of the steady state shear stress, referred to as “50% partially consolidated state”. The flow function curves for ESKAL 500 (solid curves: coaxiality approach for calculating σ_1 and Jenike method for σ_c), as shown in Figure 3-19, indicate no significant change in flowability for the critically consolidated and 75% partially consolidated powder across the range of the major principal stresses. The powder at the 50% partially consolidated state had a slightly better flowability than the other two states. The 75% partially consolidated state exhibits slightly worse flowability than the critically consolidated powder for the coaxiality case (coaxiality approach for both σ_1 and σ_c).



■, MgCO_3 (steady state); ◆, $\text{MgCO}_3_{0.75}$ (75% partially consolidated state); ▲, $\text{MgCO}_3_{0.50}$ (50% partially consolidated state); –, $\text{MgCO}_3_{\text{Jenike}}$ (steady state, Jenike methodology); ×, $\text{MgCO}_3_{\text{coaxiality}}$ (steady

state); ●, MgCO₃_0.75_coaxiality (75% partially consolidated state); +, MgCO₃_0.50_coaxiality (50% partially consolidated state).

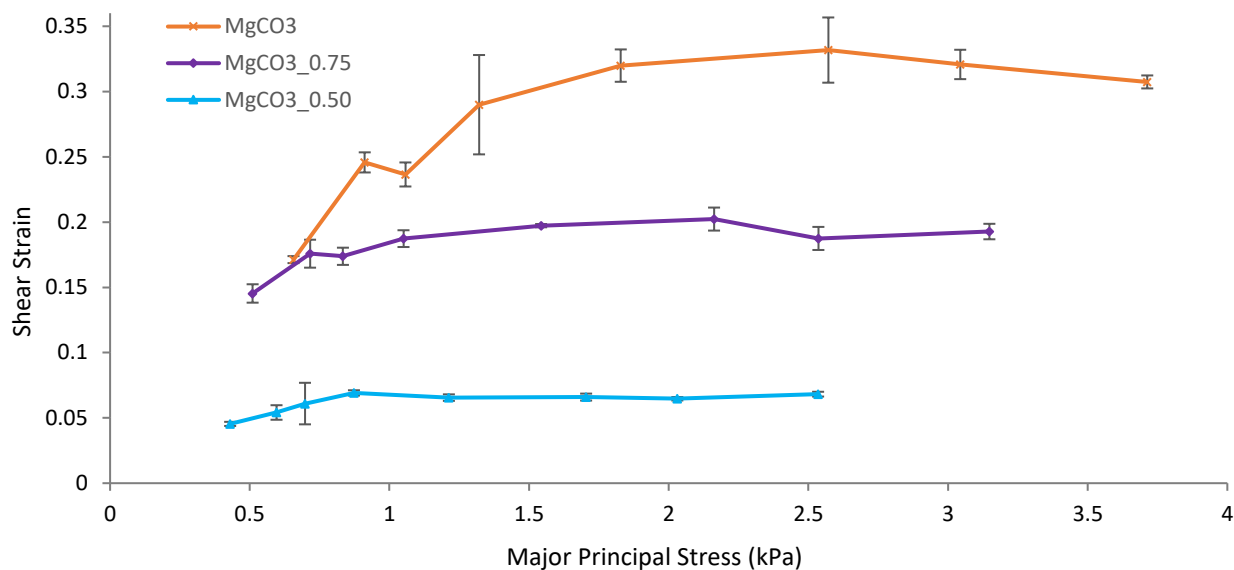
Figure 3-18: Flow function curves of MgCO₃ (Schulze RST).



■, ESKAL_500 (steady state); ◆, ESKAL_500_0.75 (75% partially consolidated state); ▲, ESKAL_500_0.50 (50% partially consolidated state); —, ESKAL_500_Jenike (steady state, Jenike methodology); ×, ESKAL_500_coaxiality (steady state); ●, ESKAL_500_0.75_coaxiality (75% partially consolidated state); +, ESKAL_500_0.50_coaxiality (50% partially consolidated state).

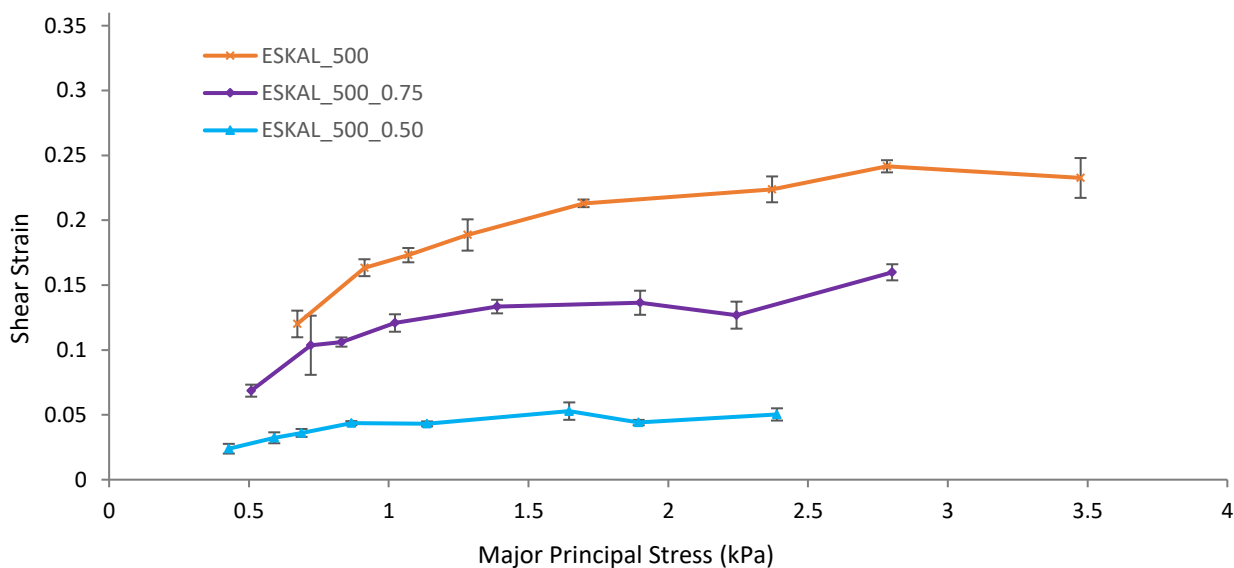
Figure 3-19: Flow function curves of ESKAL 500 (Schulze RST).

The shear strain to reach steady state (critical state) and partially consolidated states (under-consolidated states) in the shear experiments was measured for MgCO₃ (Figure 3-20) and ESKAL 500 (Figure 3-21). As the steady state is reached asymptotically, the shear strain necessary for the steady state is assumed to be the one at which the shear stress reaches 90% of the steady state shear stress value. The deformation required varies quite significantly with the consolidation state to be reached. The deformation needed to achieve the steady state was nearly five times larger than that required to achieve a shear stress value equal to 50% of that at steady state.



x, MgCO₃ (steady state); ♦, MgCO₃_0.75 (75% partially consolidated state); ▲, MgCO₃_0.50 (50% partially consolidated state).

Figure 3-20: Shear strain – major principal stress curves of MgCO₃.



x, ESKAL_500 (steady state); ♦, ESKAL_500_0.75 (75% partially consolidated state); ▲, ESKAL_500_0.50 (50% partially consolidated state).

Figure 3-21: Shear strain – major principal stress curves of ESKAL 500.

An inspection of the flow function and the strain curves for both the powders (Figure 3-18 - Figure 3-21) indicates that the flowability of the powder depends weakly on the extent of deformation and the achieved consolidation state (partial or steady). If the flowability of the powder, evaluated at the stress state relevant to the handling and process conditions, varied with the stress state, it could directly affect the design of the handling/storage equipment to ensure powder flow. According to the reported results, instead, the

deformation or consolidation state of the powder in this study marginally affected the flowability. This finding indicates that the flowability of the powder evaluated at the critical consolidation is a good approximation also for the flowability at the partially consolidated states. Therefore, designing the equipment while considering steady-state flow would produce a design solution that is safe for partially consolidated states and would not lead to an overly conservative design. From the results, we can see that the state of consolidation affects the major principal stress and the yield loci, as well as the resulting yield strength values, at similar values of pre-shear normal stress. However, the change in flowability or the flow function is not significant. The coaxiality approach to estimate the yield locus shifts the flow function to poorer flowability. For some cases, for example, MgCO_3 , the flow function shifts to the hardened or non-flowing region. This result can also be observed for some of the points at lower stresses of the ESKAL flow function. The method utilized (σ_c determined by Jenike yield locus and σ_1 determined by coaxiality hypothesis) predicts higher flowability than the coaxiality approach (σ_1 and σ_c determined by coaxiality approach) with the latter producing higher failure strengths.

The shear tests conducted on the shear cell at varying states of consolidation for the two cohesive powders show that the flowability of these powders is marginally affected by their state of consolidation, or in other words, the state of deformation of the powders slightly impacts their flowability. To verify this finding, experimentation was conducted in a model silo apparatus that is likely to have the conditions necessary for the powders to flow under partial consolidation. The details of this apparatus, along with the testing methodology and corresponding results, are discussed in the next section.

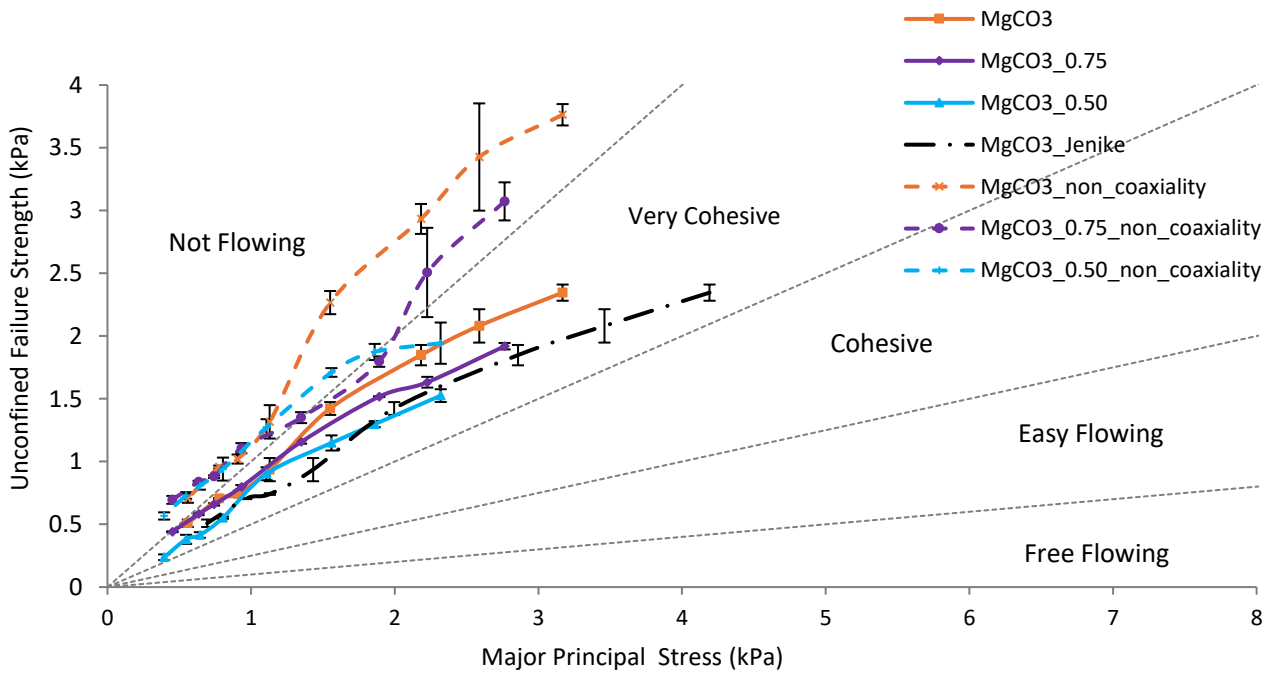
3.4.4 Coaxiality hypothesis & consideration of non-coaxiality

There are some experimental studies on the hollow cylinder apparatus which determine the principal strain and stress directions experimentally. In the research work of Cai (2010), the author explains the working of the hollow cylinder apparatus in which the soil sample is subjected to normal axial load together with a torque and internal and external pressures, the axial and radial strains are calculated experimentally. The stress & pressure values are used to compute the stress tensor; the experimental strain values are used to compute the strain tensor. From stress and strain tensors, the principal stress and strain values are calculated which are then used to compute non-coaxiality. The study found that the principal stress and strain directions are non-coaxial and the level of non-coaxiality depends on the specimen density, stress/loading path, stress level and particle properties. In another experimental study (Yang et al., 2015) using the hollow cylinder apparatus, the authors found that principal stress and strain rate directions are non-coaxial and that in addition of other factors it also depends on the angularity of the particles tested.

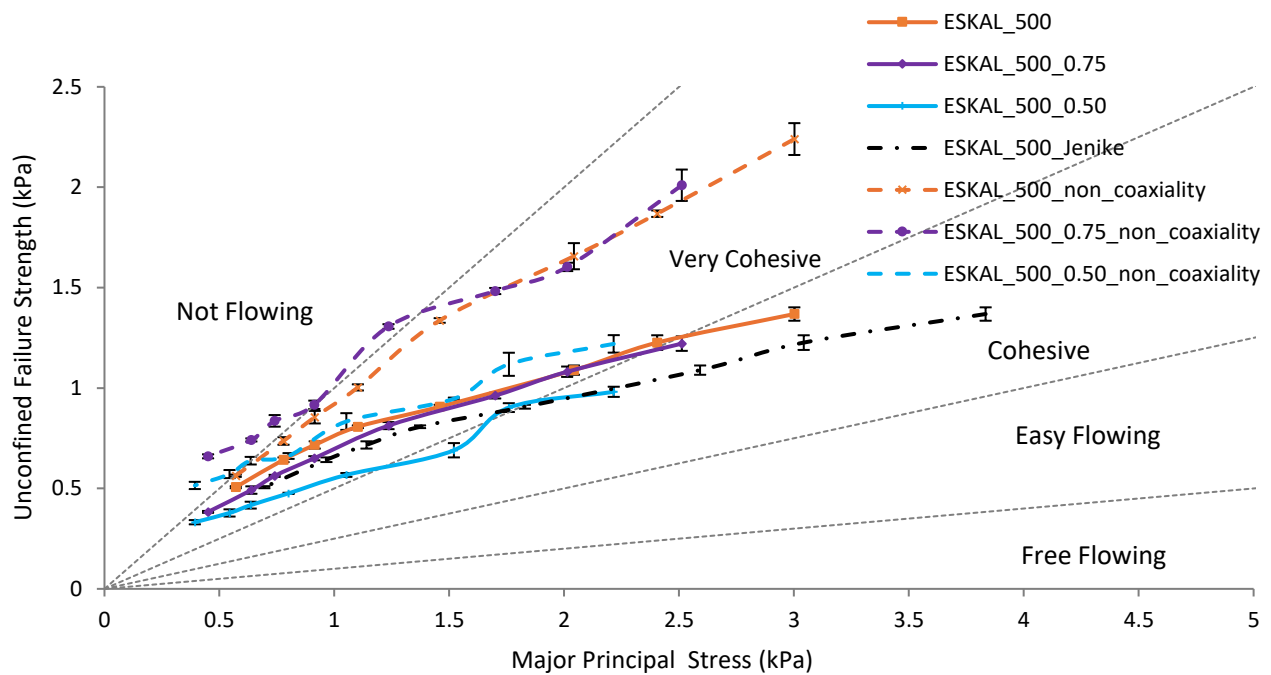
Thornton & Zhang (2006) carried out “simple shear” or translational shear simulations with spherical particles. The paper studies the evolution of the angle of non-coaxiality with increasing shear strain. The paper shows that the level of non-coaxiality depends on rate of dilation, initial stress state, loading path and

the shearing resistance angle. The study concludes that when the flow is fully developed the stress and strain rate directions become coaxial.

This thesis determines the stress state of the powders under varying consolidation states by assuming coaxiality between the principal stress and strain planes. Literature such as Cai (2010) and Yang et al. (2015) negate this assumption through experiments, whereas the work by Thornton & Zhang (2006) supports this assumption when the flow is fully developed. To study the possible effect of non-coaxiality on flowability, the major principal stresses in Figure 3-22 and Figure 3-23 are determined by considering non-coaxiality of -10° and $+10^\circ$ respectively between the principal strain and stress planes for both the powders.

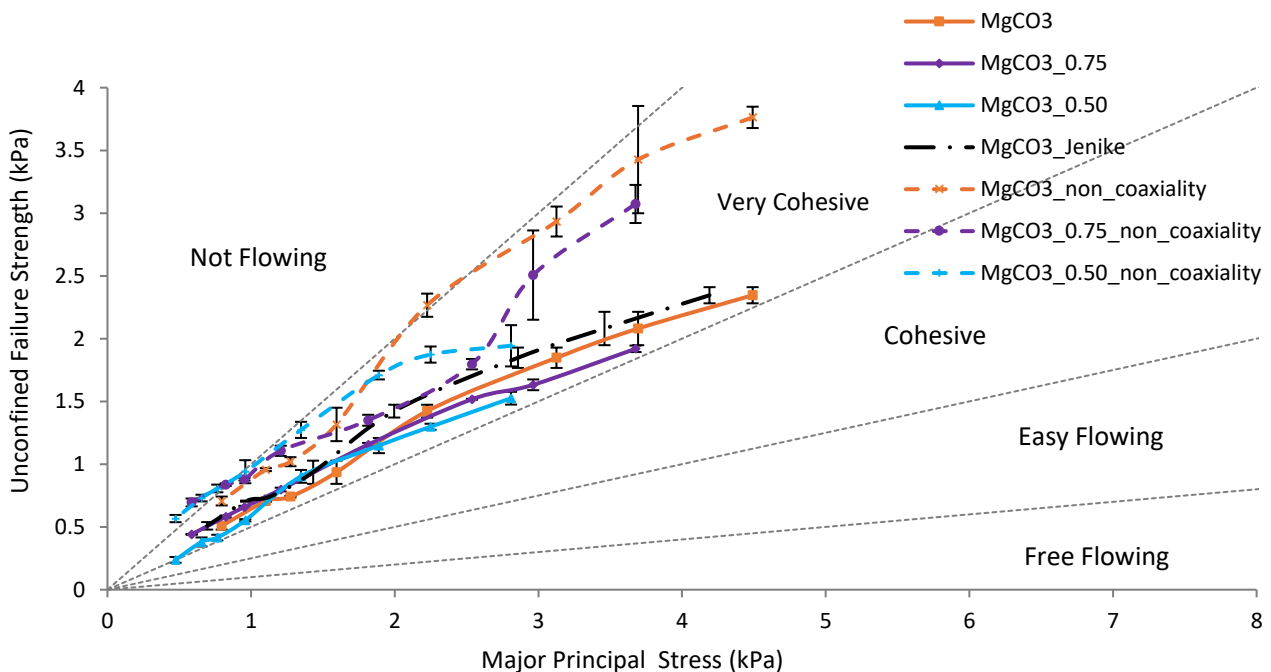


a) Flow function curves of MgCO_3 , ■, MgCO_3 (steady state); ◆, $\text{MgCO}_3_{0.75}$ (75% partially consolidated state); ▲, $\text{MgCO}_3_{0.50}$ (50% partially consolidated state); —, $\text{MgCO}_3_{\text{Jenike}}$ (steady state, Jenike methodology); ×, $\text{MgCO}_3_{\text{non_coaxiality}}$ (steady state); ●, $\text{MgCO}_3_{0.75_{\text{non_coaxiality}}}$ (75% partially consolidated state); +, $\text{MgCO}_3_{0.50_{\text{non_coaxiality}}}$ (50% partially consolidated state).

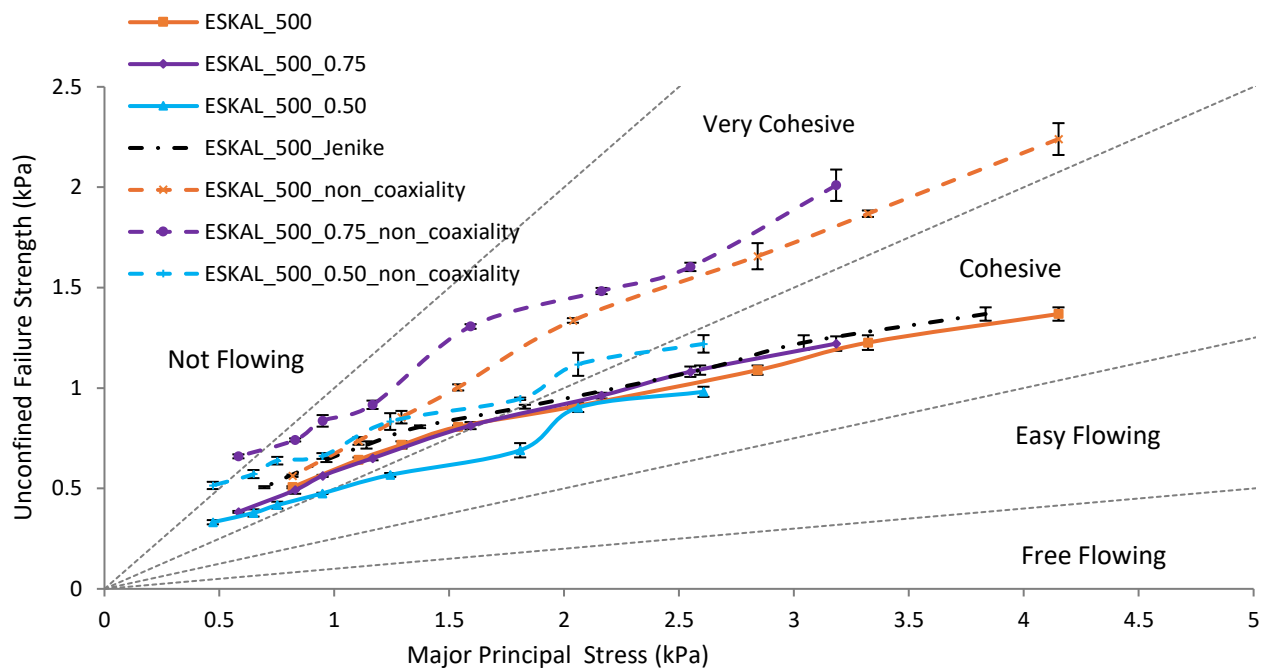


b) Flow function curves of ESKAL 500, ■, ESKAL_500 (steady state); ♦, ESKAL_500_0.75 (75% partially consolidated state); ▲, ESKAL_500_0.50 (50% partially consolidated state); —, ESKAL_500_Jenike (steady state, Jenike methodology); ×, ESKAL_500_non_coaxiality (steady state); ●, ESKAL_500_0.75_non_coaxiality (75% partially consolidated state); +, ESKAL_500_0.50_non_coaxiality (50% partially consolidated state).

Figure 3-22: Flow function curves of a) MgCO_3 and b) ESKAL 500 (Schulze RST) considering non-coaxiality of -10° between the principal strain and stress planes.



a) Flow function curves of MgCO_3 , ■, MgCO_3 (steady state); ♦, MgCO_3 _0.75 (75% partially consolidated state); ▲, MgCO_3 _0.50 (50% partially consolidated state); —, MgCO_3 _Jenike (steady state, Jenike methodology); ×, MgCO_3 _non_coaxiality (steady state); ●, MgCO_3 _0.75_non_coaxiality (75% partially consolidated state); +, MgCO_3 _0.50_non_coaxiality (50% partially consolidated state).



b) Flow function curves of ESKAL 500, ■, ESKAL_500 (steady state); ◆, ESKAL_500_0.75 (75% partially consolidated state); ▲, ESKAL_500_0.50 (50% partially consolidated state); —, ESKAL_500_Jenike (steady state, Jenike methodology); ×, ESKAL_500_non_coaxiality (steady state); ●, ESKAL_500_0.75_non_coaxiality (75% partially consolidated state); +, ESKAL_500_0.50_non_coaxiality (50% partially consolidated state).

Figure 3-23: Flow function curves of a) MgCO_3 and b) ESKAL 500 (Schulze RST) considering non-coaxiality of $+10^\circ$ between the principal strain and stress planes.

The flow functions labelled simply with the name of the powder, such as “ESKAL_500” estimate the unconfined failure strength from the Jenike method and the major principal stresses are determined by assuming a non-coaxiality between the major principal strain and stress planes. The flow functions labelled by adding non_coaxiality to the curve label, for example “ESKAL_500_non_coaxiality” determine the unconfined yield strength by assuming the principal strain and stress planes to be coaxial at incipient shearing and assume a non-coaxiality between the major principal strain and stress planes for determining the major principal stresses.

Non-coaxiality of -10° would mean if plane of principal strain is inclined at 40° , plane of principal stress would be inclined at 30° . The results show that the angle between the principal strain and stress planes shifts the major principal stress to higher values as non-coaxiality changes from -10° to $+10^\circ$. Importantly, it does not change the finding that was observed assuming coaxiality, i.e., state of consolidation marginally impacts the flowability of the powders.

3.5 Silo flow experimentation for verification of flowability hypothesis

3.5.1 Silo apparatus

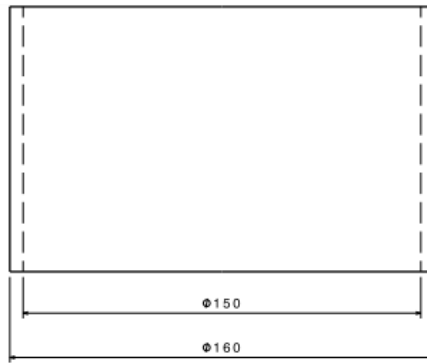
An experimental silo apparatus similar to Miccio et al. (2013) was designed and fabricated to verify if the powder deformation state affects its flowability. The silo apparatus enabled testing of the powders at various

states of consolidation. Front views of the silo, base disc, annulus, and the plug can be seen in Figure 3-24. The flat-bottom silo apparatus has a cylindrical Perspex wall placed on a base disc, held by supporting rails, as seen in Figure 3-25a. The base disc can accommodate several combinations of annular discs and plugs to vary the outlet flow size of the silo apparatus from 30 mm to 100 mm in increments of 10 mm. The plug is held in position by rotary flaps, as seen in Figure 3-25b, which are connected to the base disc. These are used to hold the plug in position during consolidation and are then rotated to remove the plug.

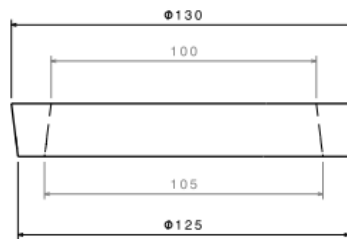
The base disc is shaped to fit the cylindrical silo walls precisely. The annulus features an external downward taper to fit snugly into the base and prevent movement when the silo is loaded. The plug has an upward taper so that it only experiences the weight of the powder and minimal friction on its sides when in position. The shape eases the plug removal without disturbing other components of the silo apparatus.

The plug is held in position by rotating the rotary flaps during consolidation. The flaps are rotated to open the plug and observe the powder flow, particularly if piping (Figure 3-26b) or arching (Figure 3-26c) occurs. In a different experiment, the plug is held in position by a strut connected to a load cell to register the vertical load exerted by the powder bed onto it. To facilitate operation, the bottom of the plug features an extruded base that accommodates a cylindrical cavity for the strut tip, as illustrated in Figure 3-25a. The base disc, annular discs and plugs are fabricated by additive manufacturing using PLA material.

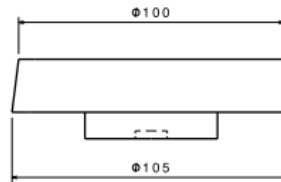
Silo
Front view
Scale: 1:1



Annulus
Front view
Scale: 1:1



Plug
Front view
Scale: 1:1



Base disc
Front view
Scale: 1:1

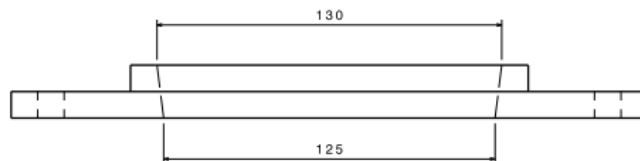


Figure 3-24: CAD drawing of the silo, base disc, annulus, and plug.

3.5.2 Testing methodology on silo apparatus

The experiments on the silo apparatus were conducted with two different procedures. One used a purely vertical compression of the powder in the silo. The vertical compression tests were conducted using a flat compression lid (Figure 3-25c). The other method used a combination of vertical compression and horizontal shear to consolidate the powder in the silo by means of a shearing lid equipped with vanes (Figure 3-25d). The sequence of steps for the two procedures was similar:

1. An annular disc and its fitting plug are selected to operate the silo with a particular outlet diameter.
2. The apparatus is set up by affixing the cylindrical silo section, the annular disc and the plug onto the base disc.
3. The silo is filled with a powder sample (Figure 3-26a), and the powder surface is levelled.
4. The powder bed is consolidated. For this purpose:
 - a. In the case of purely vertical compression, the flat compression lid is used and loaded with the desired weights

- b. In the case of vertical load and shear for compression, the shearing lid is used and loaded with the weights. A fixed number of twists is applied to the lid.
5. The lid and the weight are removed.
6. The outlet plug is removed to observe any piping or arching (Figure 3-26b or c).

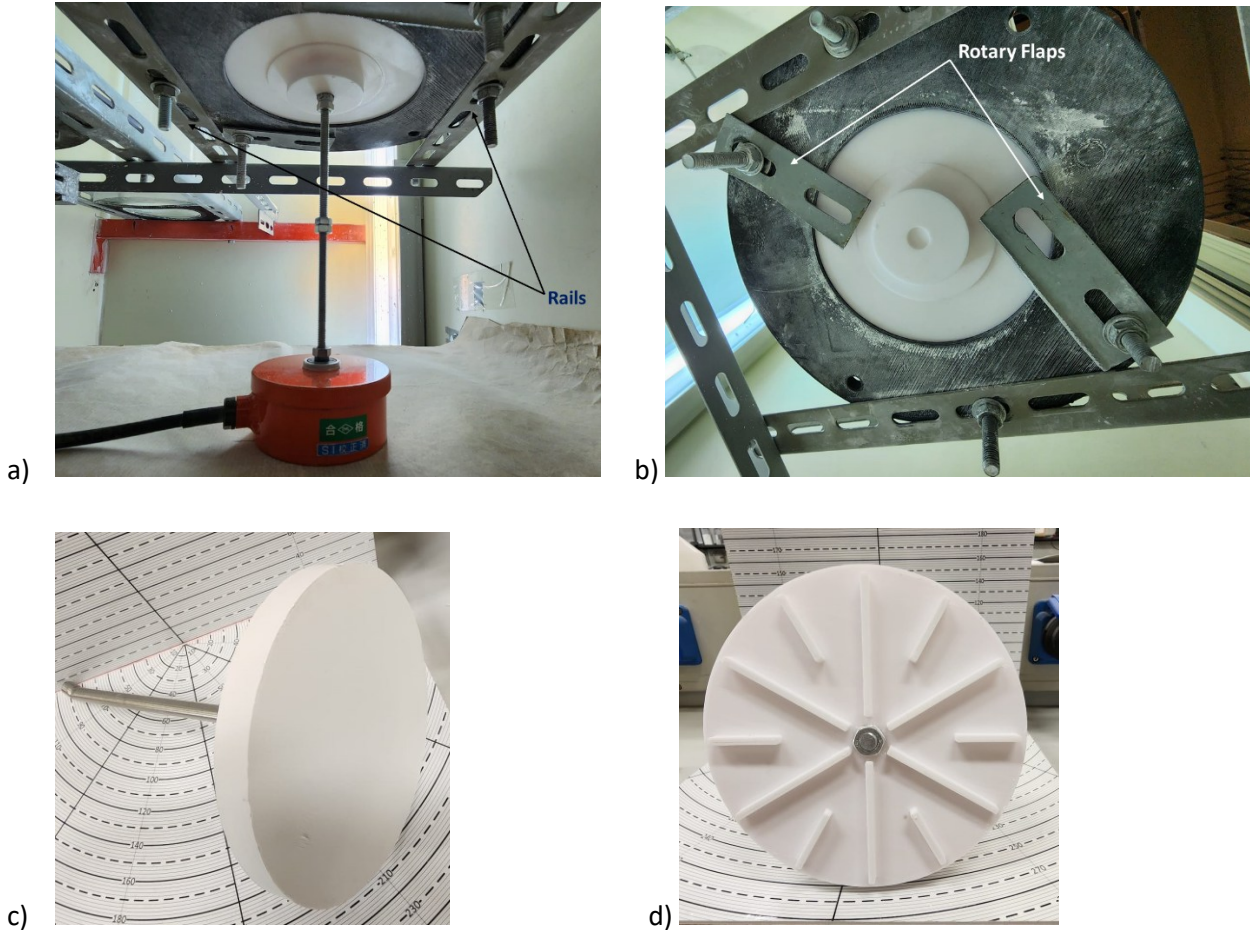


Figure 3-25: Silo apparatus; a) set up to measure the load acting on the outlet plug by a load cell with a strut, b) rotary flaps holding the outlet plug, c) flat compression lid for the uniaxial compression, and d) shearing lid provided with vanes to combine vertical load and shear during consolidation.

Experiments are repeated for each outlet diameter, and various consolidation loadings are tested to find the loading at which the transition between arching and piping flow occurs. This load is identified as the transition load, N_{tran} , unique for the powder quantity being tested and the silo and the outlet diameter. The resulting stress is called the transition stress σ_{tran} which is calculated as $(N_{tran}g/A)$, where A is the area of the compression or shearing lid. The Jenike methodology for silo design assumes that an arch collapses when the stress at the arch abutment exceeds the material strength.

The consolidation stage in the experiments with simultaneous compression and shearing on the apparatus is shown in Figure 3-26a. A series of preliminary experiments was conducted to determine the twisting angle required to apply to the material to reach a critical consolidation. In these set of experiments, a large twisting angle was applied to reasonably correspond to the more than necessary shear deformation to achieve the

critical consolidation state in the powder (450° for MgCO_3 and 1800° for ESKAL 500). Although the twisting motion is applied at the lid surface, experimental observations indicated that the induced shear deformation propagated through a substantial portion of the powder bed depth. To qualitatively assess shear transmission, a lightweight thin wooden needle was inserted into the MgCO_3 powder bed at different depths, and the angular rotation required to observe noticeable needle deflection was recorded. The total powder depth was 80 mm and the wooden needle was inserted from the bottom through a hole in the transparent Perspex sheet (Figure 3-26d) at the designated testing depth, enabling observations of deflection upon applying shear from top. Deflection was observed at all investigated depths, including near the bottom of the powder bed, although progressively larger lid rotations were required with increasing powder depth calculated from the top surface. For example, noticeable deflection occurred after approximately 90° of rotation at a depth of 40 mm from top, compared to approximately 450° at a depth of 70 mm from top. These observations suggest that shear transmission through the powder bed is not confined solely to the upper layers, although the intensity and rate of shear propagation decrease with depth.

The transition load for arching/no arching with this twisting angle (450° for MgCO_3 and 1800° for ESKAL 500) was identified by changing the weight loaded on the lid in the experiments. In the next set of experiments, the twisting angle of the shearing lid in the consolidation stage was gradually reduced by keeping the load just above the transition load. The twisting angle, providing a transition between arching/piping, was assumed to be the minimum required to achieve critical consolidation in the silo. If experiments are repeated under the same load but with a lower twisting angle of the shearing lid, the sample is under-consolidated, and the arch is not formed. It was assumed that the arching/piping transition load for partially consolidated states can be determined by changing the load in experiments at which consolidation is carried out using twisting angles that are a fixed fraction of the angle providing a critical consolidation. The experiments were carried out with varying outlet flow diameters for both the powders tested, first to identify the deformation required for the steady state and the corresponding transition load and then conducted to obtain the corresponding transition loads at the partially consolidated states.

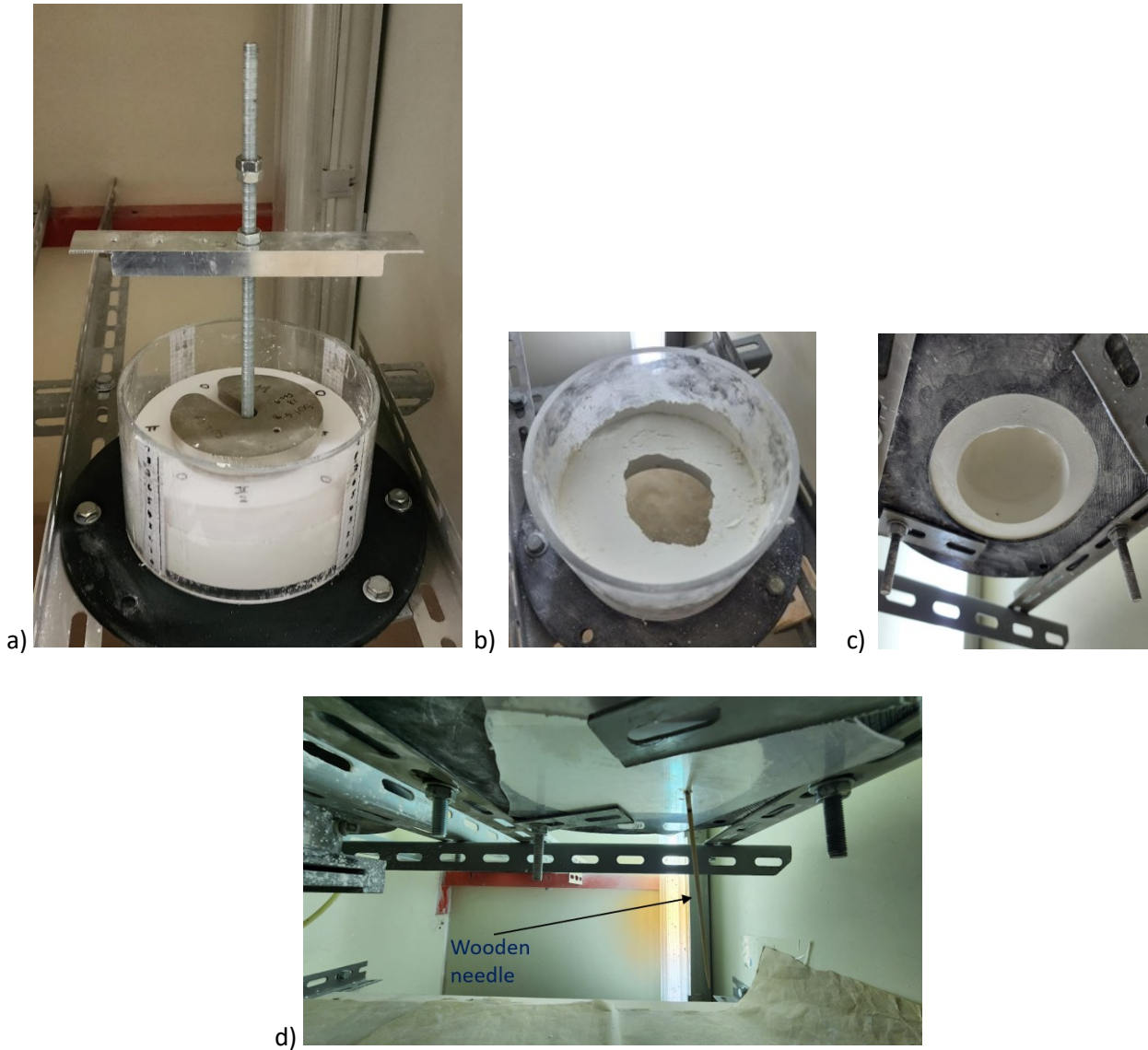


Figure 3-26: a) Shearing experimentation in silo apparatus, type of flow observed in silo; b) pipe flow and c) arching, d) shear transmission experimentation.

To explain the process followed, let us consider a case in which 0.5 kg ESKAL 500 is tested in the silo apparatus. The annulus and the plug are selected to have the outlet flow diameter of 60 mm. The experiments were started with a rotation of 1800° of the shearing lid, the transition load was determined at this rotation. Next, experiments are carried out by reducing the rotation of the shearing lid whilst imposing a load slightly above the transition load to identify the shear deformation required to achieve the steady state. The steady state in the silo is defined as the state of critical consolidation, a minimum value of rotation is required for the sample to be critically consolidated or be under the steady state, increasing the rotation or shearing further than this minimum value still keeps the sample critically consolidated but if a lesser shearing than this value is provided, the sample would be under consolidated or be at partially consolidated state. This minimum value for 0.5 kg ESKAL 500 tested with 60 mm flow diameter is identified as 900° . If experiments are repeated under the same transition load but lower shearing lid rotation the sample is partially consolidated, and the arch is not formed,

or the sample is not critically consolidated. After this the samples are sheared to achieve $1/5^{\text{th}}$ shear deformation to achieve the 50% partial consolidation state, as per the shear strain results from Schulze RST. For the case under consideration, powder was sheared by rotating the shearing lid by 180° to attain the 50% partial consolidation state and the experiments conducted at this rotation to identify the transition load.

After the transition load is determined, the consolidation procedure is repeated using the plug connected to the load cell to measure the normal load effectively transferred to the bottom, N_{eff} . In the case of purely vertical compression applied to the powder bed, the normal stress experienced at the bottom of the powder bed is considered to be close to the major principal stress σ_1 . For cases where consolidation combines vertical stress and shear, since direct measurement of the shear stress at the silo bottom is not possible, the major principal stress at steady-state consolidation, and 50% partial consolidation were calculated, assuming the same ratio k_σ between the major principal stress and the applied normal stress in the corresponding shear test experiments. σ_{1sil} is the consolidation stress at the hopper outlet in the silo experiment and is determined from Equation (3-15).

$$\sigma_{1sil} = \frac{N_{eff} g}{A_1} k_\sigma \quad (3-15)$$

$$k_\sigma = 1 \quad \text{for purely vertical consolidation experiments}$$

$$k_\sigma = \left(\frac{\sigma_1}{\sigma_{pre}} \right)_{critical\ state\ shear\ test} \quad \text{for critical consolidation experiments}$$

$$k_\sigma = \left(\frac{\sigma_1}{\sigma_{pre}} \right)_{50\% \ consolidation\ shear\ test} \quad \text{for 50% partial consolidation experiments}$$

where A_1 is the area of the plug. The Jenike expression to calculate the abutment stress, $\sigma'_1 = D_0 \rho_b g/2$ in a conical hopper is adopted (Schulze, 2008) to determine the unconfined yield strength. Since at transition, it is assumed that $\sigma'_1 = \sigma_{cJ}$, then

$$\sigma_{cJ} = \frac{D_0 \rho_b g}{2} \quad (3-16)$$

where D_0 is the plug diameter and ρ_b is the critical bulk density. A different procedure to determine σ_c is suggested by Miccio et al. (2013) to account for the non-ideality of arch breakage in silo testing:

$$\sigma_{cM} = \frac{D_0 \rho_b g}{2} \left(1 + \frac{1.2(1 - \sin \varphi_e) D_0}{4 \tan \varphi_e h_a} \right) \quad (3-17)$$

where it was suggested that, for the arch, effective thickness is $h_a = 2 \times 10^{-3}$ m.

The consolidation stress σ_{1sil} and unconfined yield strength σ_c (either σ_{cJ} or σ_{cM}) can be plotted as a point of the flow function curve, and the test results obtained with the different outlet flow diameters are the “silo” flow function curve.

3.5.3 Flow evaluation at varying consolidation states

Table 3-4 and Table 3-5 present the main experimental data obtained from silo testing for MgCO_3 and ESKAL 500, respectively. The Schulze RST flow characterization experiments show that the shear strain required for obtaining the steady state is approximately five times that of the strain required to obtain the 50% partially consolidated state. In the silo testing, the twisting angles, θ_t , necessary to critically consolidate the powder with a 60 mm flow diameter and for 0.375 kg of MgCO_3 and 0.75 kg of ESKAL 500 were identified as 450° and 900° after testing, respectively. The corresponding horizontal strain values in the vertical direction were calculated from the bed height H_{bed} as Equation (3-18).

$$\gamma_{xy} = \frac{D_0 \theta_t}{2H_{bed}} \quad (3-18)$$

Table 3-4: MgCO_3 silo experimentation at varying consolidation states, a) test results, and b) standard deviations for unconfined yield strength.

a)

MgCO_3 : Steady state

D_0 (m)	N_{tran} (kg)	σ_{tran} (kPa)	θ_t ($^\circ$)	γ_{xy}	ρ_b (kg/m^3)	σ_{eff} (kPa)	σ_{1silo} (kPa)	σ_{cJ} (kPa)	σ_{cM} (kPa)
0.05	0.25	0.149	270	6.42	607	0.302	0.596	0.149	0.291
0.06	0.55	0.327	450	7.58	705	0.594	1.171	0.207	0.446
0.07	0.7	0.416	270	3.56	743	0.695	1.372	0.255	0.597
0.08	0.9	0.535	450	5.00	765	0.837	1.651	0.300	0.759

MgCO_3 : 50% partial consolidation state

D_0 (m)	N_{tran} (kg)	σ_{tran} (kPa)	θ_t ($^\circ$)	γ_{xy}	ρ_b (kg/m^3)	σ_{eff} (kPa)	σ_{1silo} (kPa)	σ_{cJ} (kPa)	σ_{cM} (kPa)
0.05	0.5	0.297	54	1.31	624	0.454	0.596	0.153	0.300
0.06	0.7	0.416	90	1.57	684	0.684	0.898	0.201	0.432
0.07	0.85	0.505	54	0.70	699	0.847	1.112	0.240	0.561
0.08	1.15	0.683	90	1.01	740	0.984	1.291	0.291	0.735

MgCO_3 : Vertical compression

D_0 (m)	N_{tran} (kg)	σ_{tran} (kPa)	θ_t ($^\circ$)	γ_{xy}	ρ_b (kg/m^3)	σ_{eff} (kPa)	σ_{1silo} (kPa)	σ_{cJ} (kPa)	σ_{cM} (kPa)
0.05	0.8	0.475	-	-	667	0.655	0.655	0.164	0.320
0.06	1.05	0.624	-	-	704	0.914	0.914	0.207	0.445
0.07	1.2	0.713	-	-	706	1.013	1.013	0.242	0.567
0.08	1.85	1.099	-	-	762	1.452	1.452	0.299	0.757

b)

MgCO_3 : Steady state (standard deviations for unconfined yield strength)

D_0 (m)	σ_{tran} (kPa)	σ_{eff} (kPa)	σ_{1silo} (kPa)	σ_{cJ} (kPa)	Std. dev. σ_{cJ} (kPa)	σ_{cM} (kPa)	Std. dev. σ_{cM} (kPa)	ρ_b (kg/m^3)
0.05	0.149	0.302	0.596	0.149	0.0046	0.291	0.0091	607

0.06	0.327	0.594	1.171	0.207	0.0040	0.446	0.0086	705
0.07	0.416	0.695	1.372	0.255	0.0082	0.597	0.0192	743
0.08	0.535	0.837	1.651	0.300	0.0062	0.759	0.0158	765

MgCO₃: 50% partial consolidation state (standard deviations for unconfined yield strength)

D_0 (m)	σ_{tran} (kPa)	σ_{eff} (kPa)	σ_{1silo} (kPa)	σ_{cJ} (kPa)	Std. dev. σ_{cJ} (kPa)	σ_{cM} (kPa)	Std. dev. σ_{cM} (kPa)	ρ_b (kg/m ³)
0.05	0.297	0.454	0.596	0.153	0.0061	0.300	0.0120	624
0.06	0.416	0.684	0.898	0.201	0.0020	0.432	0.0043	684
0.07	0.505	0.847	1.112	0.240	0.0062	0.561	0.0147	699
0.08	0.683	0.984	1.291	0.291	0.0040	0.735	0.0102	740

MgCO₃: Vertical compression (standard deviations for unconfined yield strength)

D_0 (m)	σ_{tran} (kPa)	σ_{eff} (kPa)	σ_{1silo} (kPa)	σ_{cJ} (kPa)	Std. dev. σ_{cJ} (kPa)	σ_{cM} (kPa)	Std. dev. σ_{cM} (kPa)	ρ_b (kg/m ³)
0.05	0.475	0.655	0.655	0.164	0.0077	0.320	0.0151	667
0.06	0.624	0.914	0.914	0.207	0.0075	0.445	0.0161	704
0.07	0.713	1.013	1.013	0.242	0.0050	0.567	0.0118	706
0.08	1.099	1.452	1.452	0.299	0.0078	0.757	0.0199	762

Table 3-5: ESKAL 500 silo experimentation at varying consolidation states, a) test results, and b) standard deviations for unconfined yield strength.

a)

ESKAL 500: Steady state

D_0 (m)	N_{tran} (kg)	σ_{tran} (kPa)	θ_t (°)	γ_{xy}	ρ_b (kg/m ³)	σ_{eff} (kPa)	σ_{1silo} (kPa)	σ_{cJ} (kPa)	σ_{cM} (kPa)
0.04	0.25	0.149	720	12.69	692	0.396	0.752	0.136	0.318
0.05	0.35	0.208	720	7.47	783	0.535	1.016	0.192	0.515
0.06	0.65	0.386	900	9.61	835	0.781	1.483	0.246	0.742
0.07	1	0.594	1080	11.24	841	1.013	1.925	0.289	0.969

ESKAL 500: 50% partial consolidation state

D_0 (m)	N_{tran} (kg)	σ_{tran} (kPa)	θ_t (°)	γ_{xy}	ρ_b (kg/m ³)	σ_{eff} (kPa)	σ_{1silo} (kPa)	σ_{cJ} (kPa)	σ_{cM} (kPa)
0.04	0.4	0.238	144	2.46	697	0.452	0.574	0.137	0.321
0.05	0.5	0.297	144	1.39	750	0.647	0.821	0.184	0.494
0.06	0.85	0.505	180	1.88	818	0.997	1.266	0.241	0.727
0.07	1.3	0.772	216	2.31	838	1.096	1.392	0.288	0.966

ESKAL 500: Vertical compression

D_0 (m)	N_{tran} (kg)	σ_{tran} (kPa)	θ_t (°)	γ_{xy}	ρ_b (kg/m ³)	σ_{eff} (kPa)	σ_{1silo} (kPa)	σ_{cJ} (kPa)	σ_{cM} (kPa)
0.04	0.55	0.327	-	-	694	0.583	0.583	0.136	0.319
0.05	0.7	0.416	-	-	738	0.794	0.794	0.181	0.486
0.06	1.15	0.683	-	-	776	1.051	1.051	0.229	0.690
0.07	1.6	0.951	-	-	801	1.238	1.238	0.275	0.923

b)

ESKAL 500: Steady state (standard deviations for unconfined yield strength)

D_0 (m)	σ_{tran} (kPa)	σ_{eff} (kPa)	σ_{1sil} (kPa)	σ_{cJ} (kPa)	Std. dev. σ_{cJ} (kPa)	σ_{cM} (kPa)	Std. dev. σ_{cM} (kPa)	ρ_b (kg/m ³)
0.04	0.149	0.396	0.752	0.136	0.0055	0.318	0.0129	692
0.05	0.208	0.535	1.016	0.192	0.0013	0.515	0.0036	783
0.06	0.386	0.781	1.483	0.246	0.0073	0.742	0.0221	835
0.07	0.594	1.013	1.925	0.289	0.0038	0.969	0.0129	841

ESKAL 500: 50% partial consolidation state (standard deviations for unconfined yield strength)

D_0 (m)	σ_{tran} (kPa)	σ_{eff} (kPa)	σ_{1sil} (kPa)	σ_{cJ} (kPa)	Std. dev. σ_{cJ} (kPa)	σ_{cM} (kPa)	Std. dev. σ_{cM} (kPa)	ρ_b (kg/m ³)
0.04	0.238	0.452	0.574	0.137	0.0012	0.321	0.0030	697
0.05	0.297	0.647	0.821	0.184	0.0014	0.494	0.0038	750
0.06	0.505	0.997	1.266	0.241	0.0024	0.727	0.0075	818
0.07	0.772	1.096	1.392	0.288	0.0048	0.966	0.0161	838

ESKAL 500: Vertical compression (standard deviations for unconfined yield strength)

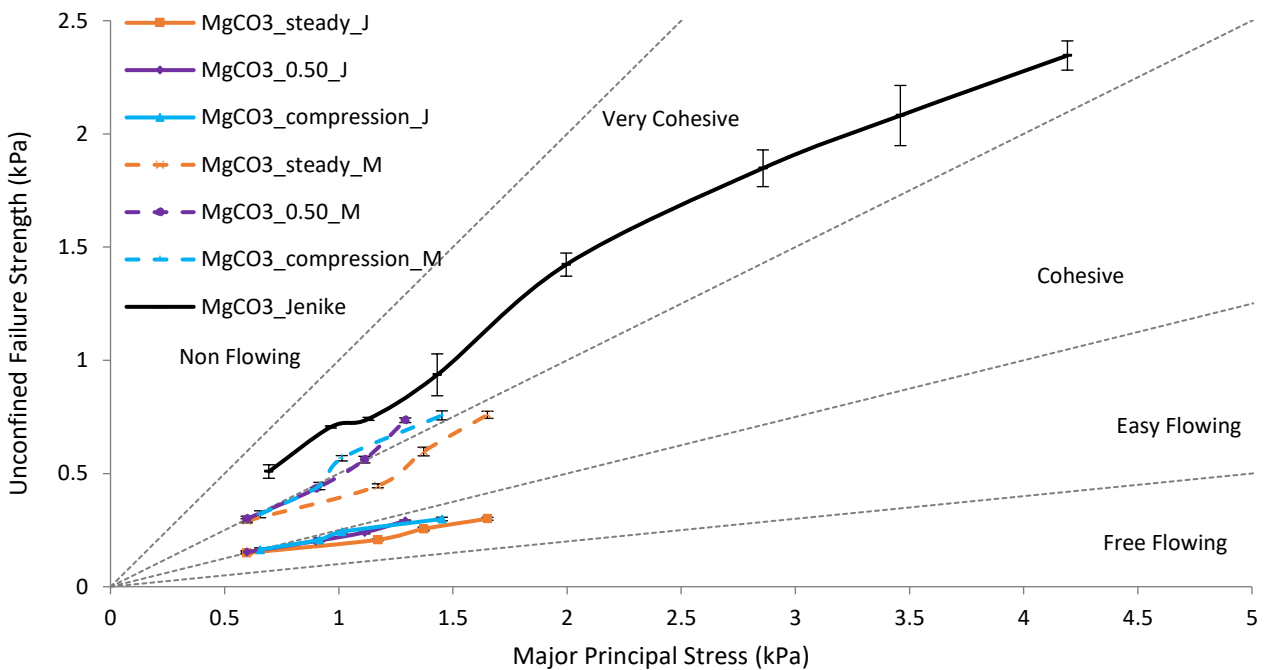
D_0 (m)	σ_{tran} (kPa)	σ_{eff} (kPa)	σ_{1sil} (kPa)	σ_{cJ} (kPa)	Std. dev. σ_{cJ} (kPa)	σ_{cM} (kPa)	Std. dev. σ_{cM} (kPa)	ρ_b (kg/m ³)
0.04	0.327	0.583	0.583	0.136	0.0003	0.319	0.0008	694
0.05	0.416	0.794	0.794	0.181	0.0016	0.486	0.0045	738
0.06	0.683	1.051	1.051	0.229	0.0034	0.690	0.0105	776
0.07	0.951	1.238	1.238	0.275	0.0017	0.923	0.0058	801

The larger shear strain required in the silo as compared to the shear cell can be attributed to a nonlinear dependence of the local powder strain on the bed depth in the silo experimentation, whose source is perhaps related to non-uniform powder consolidation or wall resistance effects that deserve further studies and can be the object of a future numerical modelling investigation. As the shear strain required for obtaining the steady state is approximately five times that of the strain required to obtain the 50% partially consolidated state, the powder in the silo was sheared by twisting the shearing lid by 90° for MgCO₃ and by 180° for ESKAL 500 to attain the 50% partially consolidated state to identify the transition loads between arching and piping. In the consolidation step, the shearing action was combined with the normal stress. In the absence of data, the major principal stress at steady-state consolidation, and 50% partial consolidation were calculated, assuming the same ratio between the major principal stress and the applied normal stress estimated in the corresponding shear test experiments.

The flow function curves of MgCO₃ (Figure 3-27) and ESKAL 500 (Figure 3-28) are obtained from the silo apparatus, independently from the method adopted to determine σ_c (either σ_{cJ} from (3-16); curves labelled “J”, or σ_{cM} from (3-17); curves labelled “M”). The curves labelled “J” confirm the finding from the shear cell experimentation, which was that the deformation state of the powder marginally impacts the flowability. The curves labelled “Jenike” in both Figure 3-27 and Figure 3-28 present the flow function derived from the shear cell by utilising the Jenike shear testing methodology to determine both σ_1 and σ_c . Evaluating the curves labelled “J”, the flowability of both the powders at 50% partial consolidation is worse than the flowability at critical consolidation but not significantly different. The flowability of the sample, which was not consolidated

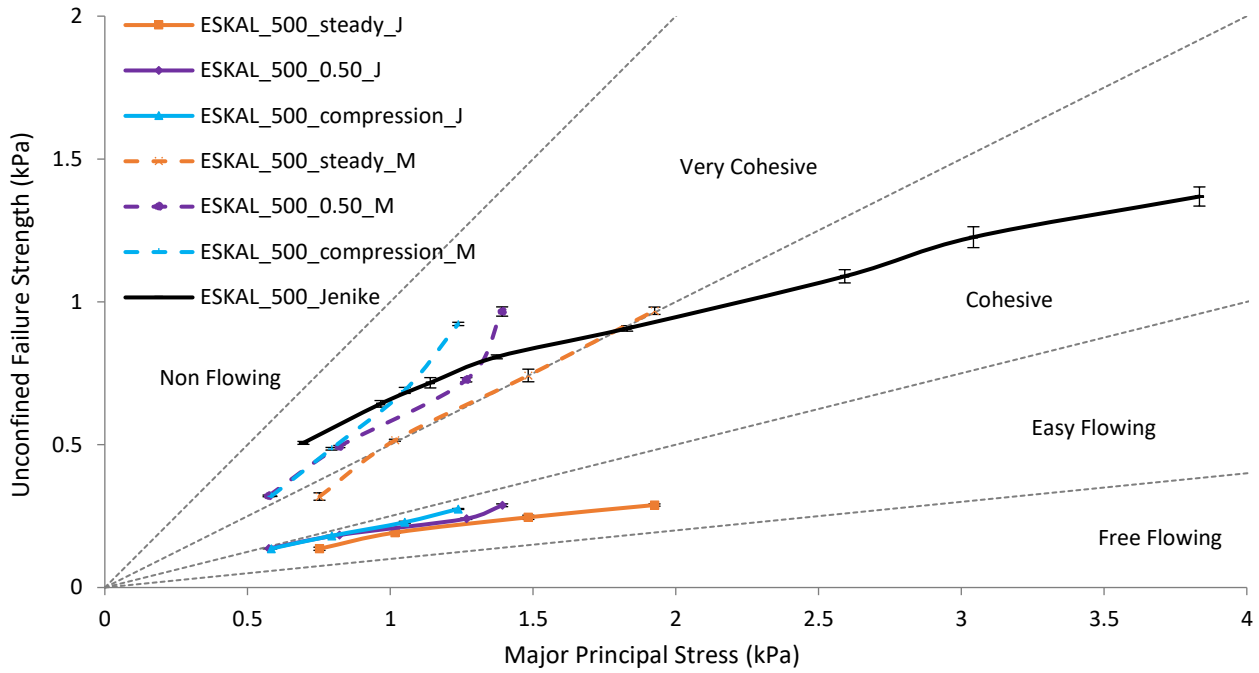
under shearing and was only consolidated under a constant vertical load, exhibited almost the same flowability as the 50% partial consolidation samples, with the flow function curves overlapping almost entirely.

Considering the different approaches used to calculate the unconfined yield strength, the “silo” flow function, evaluating the material strength σ_{cJ} according to Equation (3-16), underestimates the flow function obtained from shear cell experiments more than the “silo” flow function, evaluating the material strength σ_{cM} according to Equation (3-17). The agreement between the “silo” flow function, evaluating the material strength σ_{cM} according to Equation (3-17) and the Jenike flow function is slightly better for ESKAL 500 than for MgCO_3 .



Shear cell Jenike flow function (—) and “Silo” flow functions estimated from experiments, determining the major principal stress according to (3-15), for different consolidation methods and procedures to determine the unconfined yield strength. ■ (MgCO_3 _steady_J), steady state and σ_{cJ} according to (3-16); ◆ (MgCO_3 _0.50_J), 50% partially consolidated state and σ_{cJ} according to (3-16); ▲ (MgCO_3 _compression_J), uniaxial compression and σ_{cJ} according to (3-16); × MgCO_3 _steady_M), steady state and σ_{cM} according to (3-17); ● (MgCO_3 _0.50_M), 50% partially consolidated state and σ_{cM} according to (3-17); + (MgCO_3 _compression_M), uniaxial compression and σ_{cM} according to (3-17).

Figure 3-27: Flow function curves of MgCO_3 (silo experimentation).



Shear cell Jenike flow function (—) and “Silo” flow functions estimated from experiments determining the major principal stress according to (3-15), for different consolidation methods and procedures to determine the unconfined yield strength. ■ (ESKAL_500_steady_J), steady state and σ_{cJ} according to (3-16); ◆ (ESKAL_500_0.50_J), 50% partially consolidated state and σ_{cJ} according to (3-16); ▲ (ESKAL_500_compression_J), uniaxial compression and σ_{cJ} according to (3-16); × (ESKAL_500_steady_M), steady state and σ_{cM} according to (3-17); ● (ESKAL_500_0.50_M), (50% partially consolidated state and σ_{cM} according to (3-17); + (ESKAL_500_compression_M) uniaxial compression and σ_{cM} according to (3-17).

Figure 3-28: Flow function curves of ESKAL 500 (silo experimentation).

3.6 Summary

This Chapter first discussed the determination of the state of stress for the steady and partially consolidated states using the shear testing data. Further, the shear testing methodology to characterize the powder flowability at the partially consolidated states was discussed. Flowability results for two cohesive powders at varying consolidation states were presented and discussed, it was found that the powders’ flowability was marginally impacted by the state of consolidation. To verify if the consolidation state impacts the flow, flat-bottomed silo flow tests were carried out with the two powders at varying consolidation states.

The results indicated that the deformation or the extent of shearing did not significantly affect the cohesive blockage and the discharge of the powder from the silo, verifying the results obtained from the shear tester. The work contributed to the development of a modified consolidation methodology on a rotational shear tester (Schulze RST), in which consolidation was performed at defined fractions of the steady state shear stress to generate partially consolidated states. Further, this work presented a framework for determining the stress state associated with partial consolidation states.

Chapter 4 Meso-scale DEM modelling of cohesive powders

4.1 Introduction

This chapter presents the DEM model development for the cohesive powder (ESKAL 500) and the parametric sensitivity study of the model parameters conducted to ascertain the critical parameters, followed by a design of experiments (DOE) study of the identified critical parameters. A multi-objective genetic algorithm was then used to achieve calibration of the DEM model by minimizing selected objective functions. To gauge the accuracy of the modelled system, responses such as the internal friction angle, yield locus and the flow function were compared to the experimental results. In order to verify if the calibrated parameter set can predict the flow behaviour in a different experiment, shear tests simulations for the partially consolidated states were run.

4.2 Powder flowability characterisation and DEM powder model development

4.2.1 Experimental powder flowability characterization

The shear test experiments to characterize the powder flowability were carried out on the small (S) cell of the manually operated Schulze Ring Shear Tester (RST-01). The shear test experiments were carried out following the Jenike shear testing methodology (Jenike, 1964). The test starts with filling the powder sample into the shear cell trough, followed by loading it with the desired pre-shear normal load through the annular shearing lid. The shearing of the sample was then initiated by rotation of the shear cell trough whilst under a constant pre-shear normal load applied through the annular shearing lid, this step is known as the pre-shear step. The annular lid is prevented from rotating through the tie rods which are connected to the cross beam affixed to the shearing lid. The tie rods record the shearing force experienced by the lid through the load cells; this shear force or the torque represents the shear force or torque required to shear the powder sample. The shearing of the sample is stopped once the sample is critically consolidated, characterized by a constant shear stress and bulk density of the sample. The shear cell trough is then counter rotated to relieve the sample of the shear stress. Next, the normal load on the sample is lowered to a value lower than the pre-shear normal load and the sample is sheared until incipient flow/failure. The normal and shear stress at the point of incipient failure represents one of the points of the yield locus. The process of critical consolidation and shear to failure at normal loads lower than the pre-shear normal load is repeated to obtain other points of the yield locus. The yield locus and the pre-shear point (normal and shear stress at critical consolidation) are used to determine the major principal stress σ_1 and the unconfined yield strength σ_c (Figure 2-1) of the sample by following the Jenike methodology. The yield locus is unique to the pre-shear normal load; therefore, the shear test is conducted at varying pre-shear normal loads to obtain the other (σ_1, σ_c) values which are then plotted on the $\sigma_1 - \sigma_c$ plane to obtain the flow function curve. The shear tests were also conducted at the partially consolidated state where the pre-shearing of the sample was stopped after attaining a certain fraction of the

critical consolidation shear stress. After that, the same test procedure as the regular shear test was followed to determine the yield loci at several pre-shear normal loads.

4.2.2 Cohesive powder flow modelling in shear cell using DEM

The small (S) cell of the Schulze RST (Barletta et al., 2019; Schulze, 2008) (cell volume $\approx$ 200 mL) as seen in Figure 4-1 (dimensions in mm) was simulated using the real scale geometrical dimensions. The shear test on the RST was simulated using Altair EDEM 2023.0 software and the full shear cell (360°) was simulated. Similar to the experimental procedure of filling the shear cell where the powder was filtered through a sieve and made to fall into the shear cell, the particles in the DEM simulations were deposited into the shear cell through the “rainfall” method where particles are generated randomly in a virtual cylindrical geometry and are made to fall under gravity. The excess particles after filling were scraped off through a blade to obtain a smooth powder surface level with the surface of the shear cell trough. The particles were then allowed to achieve mechanical equilibrium, allowing the particles to fully settle for the system to have negligible kinetic energy. The powder in the shear cell trough was then loaded with the pre-shear normal load, with this load being applied through the shearing lid. The normal load was applied through the “force controller” in EDEM which allows precise application of the desired force with it being distributed over the elements of the shearing lid, thus achieving a constant average vertical stress over the powder bed. Next, the powder in the trough was pre-sheared by rotating the trough with the shearing lid applying normal force on the sample whilst free to move vertically. The rotational velocity of the trough was suitably chosen to ensure quasi-static shearing (Francia et al., 2021; GDR MiDi, 2004) of the sample with the Inertial number maintained as < 0.001 (Modenese et al., 2012). The shearing of the sample was stopped after attaining the desired consolidation state, the trough was then counter rotated to relieve the sample of the shear stress whilst reducing the normal load to the normal load of the proceeding shearing step. The sample was then sheared to failure under the reduced normal load. The failure point provides a single point on the yield locus along with the pre-shear point. This process of pre-shearing, counter rotation and subsequent shearing was repeated to obtain the other points of the yield locus. The full shear test conducted in this study has 4 shear points accompanied by the pre-shear point used to determine the major principal stress and the unconfined yield strength.

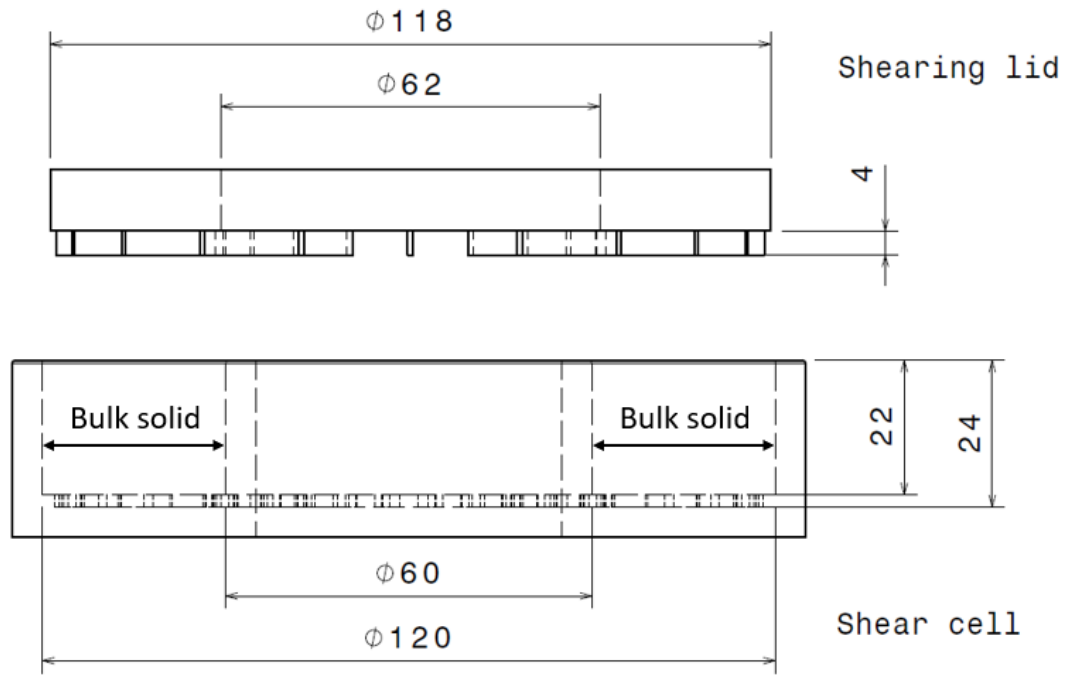


Figure 4-1: Schulze Ring Shear Tester S cell geometry

4.2.3 DEM contact model

Several particle contact models are available to model particle interactions such as Linear spring model (Cundall and Strack, 1979), Hertz-Mindlin model (Mindlin and Deresiewicz, 1953; Tsuji et al., 1992), JKR model (Johnson et al., 1971) and EEPA model (Thakur et al., 2014b). To model the stress history dependent flow behaviour of the fine cohesive powder ESKAL 500, a visco-elasto-plastic adhesive contact model known as the Edinburgh Elasto-Plastic Adhesive (EEPA) contact model was used. The EEPA contact model was selected as it has been previously successfully utilized to model the stress history dependent behaviour of cohesive bulk material in the uniaxial compression test (Thakur et al., 2016) and the Schulze ring shear tester (Mohajeri et al., 2020). For this study, the linear version of the EEPA model (Section 2.3.2) was used and the normal force-overlap schematic is shown in Figure 4-2. The meso-scale cohesive DEM model of shear testing was developed using the EEPA model to model the powder with powder-geometry interactions modelled by Hertz-Mindlin model. Type-C rolling friction model (Ai et al., 2011) implemented in EDEM was used to model the rolling friction between the particle-particle & particle-geometry contacts. This model includes both a non-viscous spring torque, which is proportional to the relative rotation angle, and a viscous damping torque, which is proportional to the relative angular velocity. The total rolling resistance torque is capped by a Coulomb-type limit based on the normal contact force.

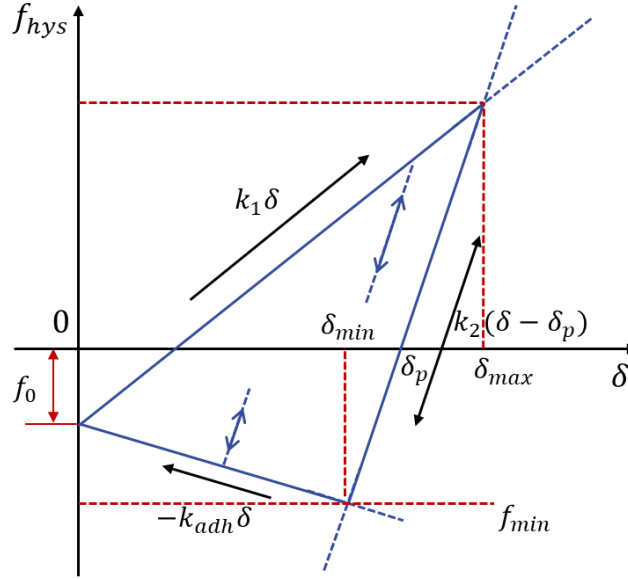


Figure 4-2: Linear mode of Edinburgh Elasto-Plastic Adhesive (EEPA) contact model

The relation between the hysteretic spring normal force f_{hys} and the normal overlap δ is expressed mathematically by the Equations in (4-1).

$$f_{hys} = \begin{cases} f_0 + k_1\delta & \text{if } k_2(\delta - \delta_p) \geq k_1\delta \\ f_0 + k_2(\delta - \delta_p) & \text{if } k_1\delta > k_2(\delta - \delta_p) > -k_{adh}\delta \\ f_0 - k_{adh}\delta & \text{if } -k_{adh}\delta \geq k_2(\delta - \delta_p) \end{cases} \quad (4-1)$$

The total contact normal force $\mathbf{f}_n$ is the sum of the hysteretic spring force f_{hys} and the normal damping force f_n^d and is given by Equation (4-2).

$$\mathbf{f}_n = (f_{hys} + f_n^d)\mathbf{n} \quad (4-2)$$

where $\mathbf{n}$ is the unit normal vector pointing from the contact point to the centre of the particle.

4.2.4 Upscaled particle size and shape to represent the cohesive powder

ESKAL 500 (Calcium Carbonate CaCO_3) was used for experimentation in the shear cell and for DEM modelling in this study. It was selected as it was used as the reference solid for the collaborative European project PARDEM, further the ESKAL series of powders produced by KSL Staubtechnik GmbH have been previously used as a reference testing powder for the collaborative European project T-MAPPP (Shi et al., 2016). ESKAL 500 is a fine cohesive powder with a $d_{50} \approx 4.9 \mu\text{m}$. The Scanning electron microscopy (SEM) images of the powder at various magnifications as seen in Figure 4-3 show that the particles have irregular shape and exhibit agglomeration, as a result the particles do not have a consistent repeatable shape which can be modelled in DEM. Modelling particles at the real particle scale of $5 \mu\text{m}$ is prohibitively expensive, therefore the particles are represented as a meso-scale particle with an increased particle size. The representation of particles with the meso-particle is reasonable, as it can be seen in the SEM images that the particles are agglomerated and seldom exist as individual particles due to the high cohesive forces. It should be pointed out that real

agglomerates may undergo breakage or fragmentation under applied loading or shearing, meaning the agglomerates can break into its constituent smaller particles. On the contrary, the meso-particles used in the present DEM study cannot break or fragment upon normal or shear load application. The meso-particle approach captures the overall bulk cohesive behaviour and agglomerated nature of the powder, but does not explicitly account for agglomerate fragmentation or morphology evolution during shearing. A dual-sphere particle with an aspect ratio of 1.2, comprised of two overlapping spheres of 1.5 mm diameter each is used to model the powder. The dual-sphere particle with some degree of non-sphericity has previously been found to be adequate for representing the real particle shape (Thakur et al., 2014b, 2014a). For the parameter sensitivity analysis and the one factor at a time (OFAT) studies, mono sized particle size distribution was used to model the particles to increase the speed of the simulations, the model parameters used for these studies are listed in Table 4-1.

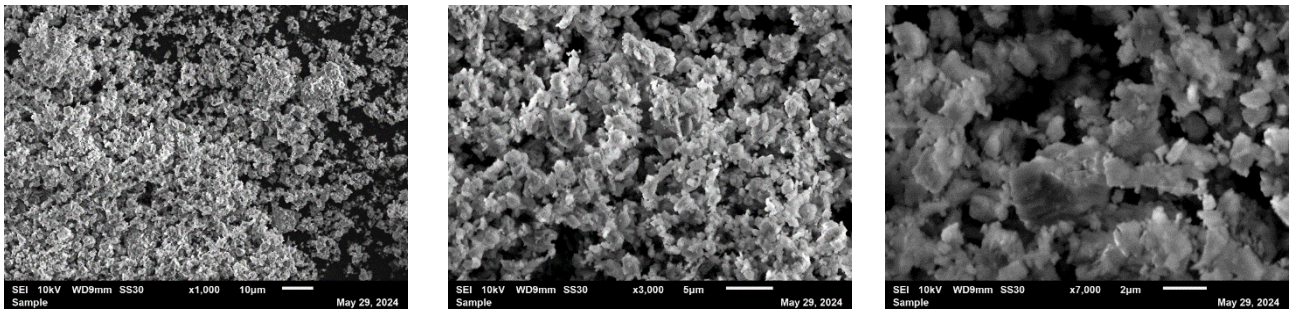


Figure 4-3: SEM images of ESKAL 500 at varying magnifications.

Table 4-1: DEM model parameters for modelling ESKAL 500.

Model (EEPA) parameters	Particle properties	System characteristics
Pull-off force f_0 (accounts for van der waals forces): -0.002 N, Adhesion energy $\Delta\gamma$: 0.1 J/m ²	Meso-particle size: 2 spheres of 1.5 mm diameter each (particle aspect ratio: 1.2), PSD: Monodisperse	Rotational velocity of trough: 0.035 rad/s (pre-shear)
Particle's modulus of elasticity E : 1×10^7 Pa Plasticity ratio $\lambda_p = 0.5$	Meso-particle shape: Dual sphere, Particle's solid density: 4000 kg/m ³ Particle's Poisson's ratio: 0.25	Time step: 7.5% of Rayleigh time step
Geometry's (steel) modulus of elasticity: 2.1×10^{11} Pa Geometry's (steel) Poisson's ratio: 0.25 Geometry's (steel) solid density: 7000 kg/m ³	Particle-particle coefficient of static friction $\mu_{s\ p-p} = 0.5$ Particle-particle coefficient of rolling friction $\mu_{r\ p-p} = 0.25$, Type C model Particle-particle coefficient of restitution $COR_{p-p} = 0.175$	 Particle shape
Tangential stiffness multiplier: 0.667	Particle-geometry coefficient of static friction $\mu_{s\ p-g} = 0.5$	Total particles $\approx 44k$ Powder mass ≈ 0.4 kg

	Particle-geometry coefficient of rolling friction $\mu_{r\ p-g} = 0$ Particle-geometry coefficient of restitution $COR_{p-g} = 0.175$	Bulk density $\approx 2150\text{ kg/m}^3$
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Deng et al., (2021a, 2021b) experimentally measured the granular Bond number of a family of fine ESKAL powders and reported values of ≈ 10 . The granular Bond number Bo_g (Capece et al., 2015) defined as the ratio of the cohesive forces to the weight of the particle can be used to broadly ascertain if the particle system is cohesive ($Bo_g > 1$) or non-cohesive ($Bo_g < 1$), it can be determined from Equation (4-3) below.

$$Bo_g = \frac{F_{cohesive}}{W_g} = \frac{\frac{2}{3} \sqrt{\frac{\beta r}{2}} \pi \Delta \gamma + |f_o|}{m_p * g} \quad (4-3)$$

Where, β is mean normal overlap, r is mean particle radius, $\Delta \gamma$ is the surface energy, f_o is constant pull off force and m_p is mass of the particle.

The constant pull off force f_o for the initial simulation runs was chosen as -0.002 N which resulted in $Bo_g \approx 22$ such that it stays close to the experimentally determined value, also it was one of the values used to represent ESKAL particle's f_o value (Thakur et al., 2014b). The plasticity ratio, λ_p represents the contact plasticity and can be used to determine the plastic deformation δ_p of the particles, an initial value of 0.5 for λ_p was selected to model the stress history dependent behaviour of the cohesive powder during the shear test, a value of $\lambda_p = 0.75$ was used by Mohajeri et al. (2020) to model cohesive coal. Cundall and Strack (1979) reported the analytical results obtained by (Mindlin, 1949), for the ratio of tangential and normal stiffness (tangential stiffness multiplier) k_t/k_n for elastic bodies in contact with elliptical contact areas to be between 2/3 and 1. In this study this ratio was chosen as 2/3, similar to the value adopted by Mohajeri et al. (2020).

For a real particle, small contact deformations are expected to remain purely elastic and should not exhibit yielding (irreversible plastic deformation). Plasticity ratio λ_p of 0.5 would suggest that for a single real particle, half of even very small deformations would be permanently plastic. It would not be physically realistic to assume this, since small deformations would generally remain predominantly elastic. However, in the present study the DEM particles are meso-particles that represent the collective mechanical response of many real particles, rather than a single particle. At this mesoscale, irreversible deformation may occur due to mechanisms such as internal rearrangement, asperity deformation, micro-slip within the represented particle assembly, even at small apparent overlaps between meso-particles. The constant plasticity ratio in this study is not intended to represent the true elastic-plastic response of a single physical particle contact, but rather an effective parameter used to capture the bulk behaviour of the coarse-grained material system at the mesoscopic scale.

The solid density of the dual sphere meso-particle was taken as 4000 kg/m^3 , which was higher than the actual ESKAL 500 solid density of 2868 kg/m^3 , the higher bulk density was chosen to increase the simulation time step in order to decrease the simulation time. The scaling of density can cause changes in the inertia effects. In a quasi-static system, the particle behaviour is dependent on the contact forces and not on the inertial effects. In the shear cell simulations, which follow quasi-static shearing, the inertial number is low as the shear rates are low which leads to lower inertial forces in the system. Contact or the frictional forces dominate the inertial forces; therefore, the density of the meso-particle can be scaled to increase the time-step of the simulations (Sheng et al., 2004).

The ASTM D6773 testing method (ASTM International, 2022) for bulk solids using Schulze RST provides a guidance on the maximum particle size of the bulk solid being tested relative to the shear cell size. For the small (S) shear cell, the recommended maximum particle size is 2.5 mm for monodisperse particle distribution. The dual sphere meso-particle's size (diameter) considering its volume to be enclosed in a perfect sphere is 1.64 mm which is around 34% less than the recommended maximum size of 2.5 mm. There would be orders of magnitude more particles between the trough boundary and centre of the trough annulus in the experiments when compared to the DEM simulations, leading to possible influence of the force chains from boundaries to the particles at the centre, possibly more than the experimental system. DEM particles aim to represent the collection of real particles, and the SEM images indicated agglomeration of primary particles, the effective particle size governing the bulk flow behaviour in the real system may be larger than the primary particle size alone. Still, it is acknowledged that the DEM simulations contain significantly fewer effective particles across the shear cell width than the real powder system, and therefore boundary-induced force-chain effects are likely to be more pronounced in the simulations. Also, unlike real agglomerates, the DEM meso-particles do not undergo fragmentation or restructuring during shearing. These limitations should therefore be recognised when interpreting the simulation results. The use of meso-particle adhering to the ASTM particle size guidelines and the calibration of the DEM model to reproduce the experimental bulk behaviour should suggest that the dominant bulk response was not highly sensitive to the chosen meso-particle size.

Prior to conducting the one-factor-at-a-time (OFAT) study for identifying critical parameters and Design of Experiments (DOE) study for model calibration, a model verification study was performed to assess the numerical stability of the DEM model. The particle Young's modulus was varied at 3 levels from 10^6 Pa to 10^8 Pa while monitoring the shear stress time series response. The model exhibited stable behaviour for 10^6 Pa and 10^7 Pa cases; however, at a Young's modulus of 10^8 Pa , oscillatory (sinusoidal) variations were observed in the shear stress response as seen from Figure 4-4, indicating numerical instability. To mitigate these oscillations and ensure numerical stability, lid damping was introduced during the normal load imposition and pre-shearing of the powder sample. The oscillations in the shear stress were sensitive to the lid damping

coefficient, and on the mass and stiffness of the lid. This dependence is expected; the damping characteristics depend on the damping coefficient and the natural frequency of oscillation (dependent on mass and stiffness of the lid). The damping ratio ζ can be determined as $\zeta = \frac{C}{C_c}$, $C_c = 2m\omega_n$, where C is the damping coefficient, C_c is the critical damping coefficient, ω_n is the natural frequency of shearing lid, $\omega_n = \sqrt{\frac{k_{lid}}{m_{lid}}}$ (k_{lid} = stiffness of lid, m_{lid} = mass of lid).

The damping coefficient and the mass & stiffness of lid interplay, as the damping ratio is dependent on the damping coefficient and the critical damping coefficient. The damping coefficient of 20 Hz together with the lid mass of 1 kg (Young's modulus = $2.1 \cdot 10^{11}$ Pa) were able to successfully suppress oscillation in the shear stress, as seen from the curve labelled " $\mu_{s p-p} 0.8 \mu_{r p-p} 0$ (lid damping)" in Figure 4-4. This enabled stable simulations at the higher stiffness values. The mass of the lid did not contribute to normal loading of the sample through the EDEM force controller, with only the actual experimental normal load being applied through it.

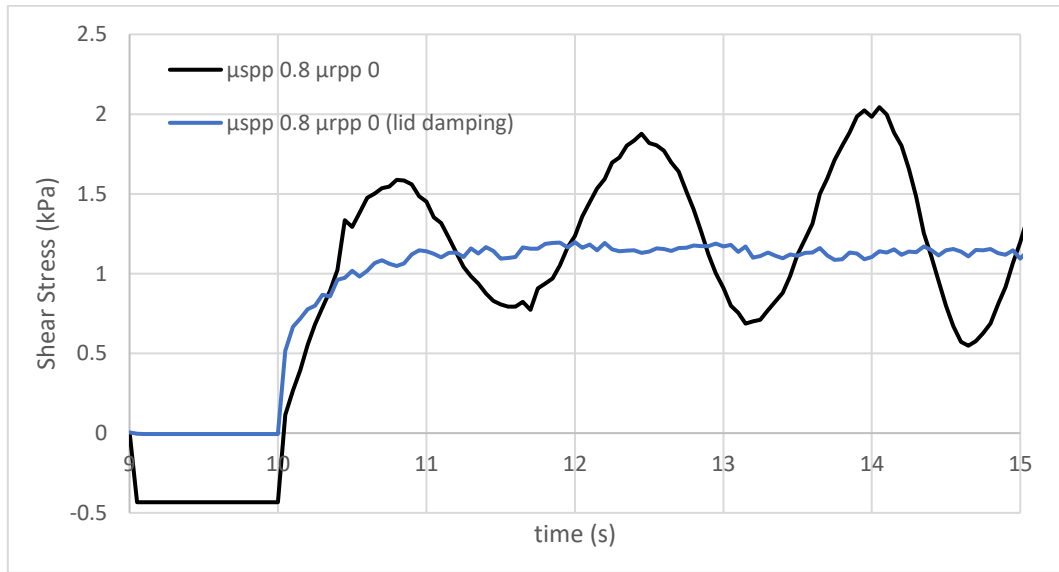


Figure 4-4: Shear stress lid damping study at $E = 10^8$ Pa

Although this numerical instability was only observed at a Young's modulus of 10^8 Pa, lid damping was retained for the 10^6 Pa and 10^7 Pa cases to provide an additional margin of numerical stability. Verification tests for the 10^6 Pa and 10^7 Pa cases demonstrated that lid damping did not affect the DEM model and the output shear stress time series response, as well the values of the average shear stress, with the difference being negligible when compared to the cases where no lid damping was used. Based on these results, the model was considered numerically verified and suitable for subsequent sensitivity and calibration studies. For all the simulations run for the parametric sensitivity analysis, and calibration of the DEM model, the mass of the lid is set to be 1 kg, and a damping coefficient of 20 Hz is used for lid damping for all the steps of the shear test.

4.2.5 Parametric sensitivity study framework

The visco-elasto-plastic adhesion contact model EEPA was used to model the cohesive powder flow. The model parameters that can impact the cohesive powder simulations and thus may need to be calibrated are as follows, particle-particle coefficient of static friction $\mu_{s\ p-p}$, particle-particle coefficient of rolling friction $\mu_{r\ p-p}$, particle-geometry coefficient of static friction $\mu_{s\ p-g}$, particle-geometry coefficient of rolling friction $\mu_{r\ p-g}$, particle virgin loading stiffness k_1 , particle unloading/reloading stiffness k_2 , contact adhesion energy $\Delta\gamma$, constant pull-off force or adhesive strength f_0 , particle-particle coefficient of restitution COR_{p-p} , and the particle-geometry coefficient of restitution COR_{p-g} . The frictional parameters $\mu_{s\ p-p}$, $\mu_{s\ p-g}$ and $\mu_{r\ p-p}$, $\mu_{r\ p-g}$ have significant influence on the particle motion and the shearing between particles. The stiffness parameters k_1 and k_2 influence significantly the compressibility and the stress history dependent behaviour of the powder which impacts the porosity of the powder bed and the number of inter-particle contacts. The adhesion energy $\Delta\gamma$ represents the energy required to separate the particles, $\Delta\gamma$ is used in calculation of the load dependent adhesive force. The adhesive strength f_0 can be used to represent forces such as the van der Waals forces, f_0 and $\Delta\gamma$ impact the porosity and compressibility of the powder bed. The restitution coefficients COR_{p-p} and COR_{p-g} determine the energy dissipation between the particle-particle and particle-geometry contacts respectively and are expected to have only a limited influence on the shearing behaviour of the material. A parametric sensitivity study was carried out to determine the influence of these parameters on the simulation results. The average shear stress during the pre-shearing stage of the shear test was the output simulation parameter which was used to evaluate the sensitivity of the model parameters, the average pre-shear shear stress was chosen as it generally depends on the cohesivity of the powder and greatly impacts the powder failure strength in the subsequent shear to failure step. A pre-shear normal load of 1.5 kg which translates to a normal stress of approximately 1.858 kPa was applied to the sample during pre-shearing. The shear stress τ acting on the sample is calculated by the torque acting on the shearing lid as follows: $\tau = \frac{M_D}{r_m A_D}$, where M_D is the torque experienced by the shearing lid during shearing, r_m is the lever arm or moment arm of the force calculated as $r_m = \frac{2}{3} \frac{r_o^3 - r_{in}^3}{r_o^2 - r_{in}^2}$, with r_o and r_{in} being the outer and inner diameter of the lid respectively, A_D is the cross sectional area of the lid.

The particle virgin loading stiffness k_1 depends on the Young's modulus and the radius of the particles and is calculated by $k_1 = 2E^*R^*$, where E^* and R^* are the equivalent Young's modulus and equivalent radius calculated by $\frac{1}{E^*} = \frac{(1-\nu_i^2)}{E_i} + \frac{(1-\nu_j^2)}{E_j}$ and $R^* = \frac{(r_i \times r_j)}{(r_i + r_j)}$ respectively. E_i , E_j ; ν_i , ν_j ; and r_i , r_j are the Young's modulus, Poisson's ratio and radii respectively of the particles i and j in contact. The plasticity ratio, $\lambda_p = 1 - \frac{k_1}{k_2} = \frac{\delta_p}{\delta}$ represents the contact plasticity, where δ_p is the maximum plastic deformation and δ is the total deformation at the contact. As k_1 is dependent on the Young's modulus of the particles and the interplay

between k_1 and k_2 can be represented by λ_p , therefore for calibrating k_1 and k_2 , E and λ_p can be calibrated instead.

One factor at a time (OFAT) study of $\mu_{s\ p-g}$, $\mu_{r\ p-g}$, COR_{p-p} , COR_{p-g} , λ_p , $\Delta\gamma$ and f_0 was conducted to gauge their sensitivity on the pre-shear shear stress. The OFAT study varies only one parameter at a time, keeping all other system parameters fixed, allowing ascertaining the effect of that parameter in isolation. The properties mentioned in Table 4-1 were used for this study unless stated otherwise. The average shear stress at the steady state during the pre-shear step was determined to ascertain the influence of the parameters.

The DEM simulations in this study used the Euler time integration method which is an explicit time integration method where the properties of the next time step are calculated by the properties at the current time step. A time step of 7.5% of the Rayleigh time step T_R (Equation (2-13)) was used in the simulation in this study as this time step provided time-step independent and stable results.

The modulus of elasticity E significantly influences the simulation time through its non-linear relationship with T_R , a higher value of E would lead to a lower T_R and thus longer simulation time. Also, E may impact the shear behaviour of the particles as a “softer” or “harder” particle leads to varying degrees of shearing resistance in the shear cell. The interplay of particle-particle coefficient of static friction $\mu_{s\ p-p}$ and particle-particle coefficient of rolling friction $\mu_{r\ p-p}$ was suspected to be influencing the net shear effect. Therefore, a full factorial parametric sensitivity study was conducted to understand the influence and interplay of the particle-particle static and rolling frictions together with the Young’s modulus of the particles. The full factorial study of these parameters was conducted across three levels: $\mu_{s\ p-p}$ (0.2, 0.5, 0.8), $\mu_{r\ p-p}$ (0, 0.25, 0.5) and E (10^6 Pa, 10^7 Pa, 10^8 Pa), resulting in a total of 27 runs. These parameters can possibly impact the packing of the powder particles in the shear cell; in order to isolate the effect of these parameters on the pre-shear step, the same powder bed or initial packing was used to run the 27 simulations with the parameters changed before the initial loading of the powder bed which precedes the pre-shear step. This was done to minimize the possible impact of packing on the pre-shear step. The time step chosen for all the runs in this study was 7.5% of T_R , with the other properties used as listed in Table 4-1.

4.2.6 DOE runs of sensitive parameters and calibration framework

The sensitive model parameters were identified from the OFAT and full-factorial studies. The calibration of these parameters was initiated by exploring the design space by carrying out the shear cell simulations across two pre-shear normal loads. Modified Extensible Lattice Sequence (MELS) DOE (Altair HyperStudy, 2023a; Hickernell et al., 2000) was used for this purpose, a lattice sequence is a quasi-random sequence, or low discrepancy sequence, designed to spread-out points in a space by minimizing clumps and empty spaces. The shear test typically tests the powder under 4 pre-shear normal load stress levels/regimes. In this study, the powder model was calibrated by using two of these four pre-shear normal loads. This was done to check the

efficacy of the calibration process by checking if the model could predict the behaviour at the other pre-shear normal loads and produce the resulting flow function by using data only from 2 pre-shear normal loads. This study utilized the data of the first pre-shear and a single shearing point for each of the two pre-shear normal load levels used for calibration. As seen from Figure 4-5, the experimental yield locus which can be obtained by fitting the points of incipient failure is approximately a straight line and therefore the yield locus can be considered linear for ESKAL 500 in this study. For this purpose, a single point from the yield locus (0.3 kg for 1.5 kg (1.858 kPa) pre-shear normal load and 0.15 kg for 0.7 kg (0.867 kPa) pre-shear normal load), together with the corresponding pre-shear point was utilized for calibration. Both these points were a fairly good representation of the linear yield locus and thus the full shear test with four shear points was not necessary, the calibration of the system was thus carried out with only the first pre-shear and shearing points across the two pre-shear normal loads.

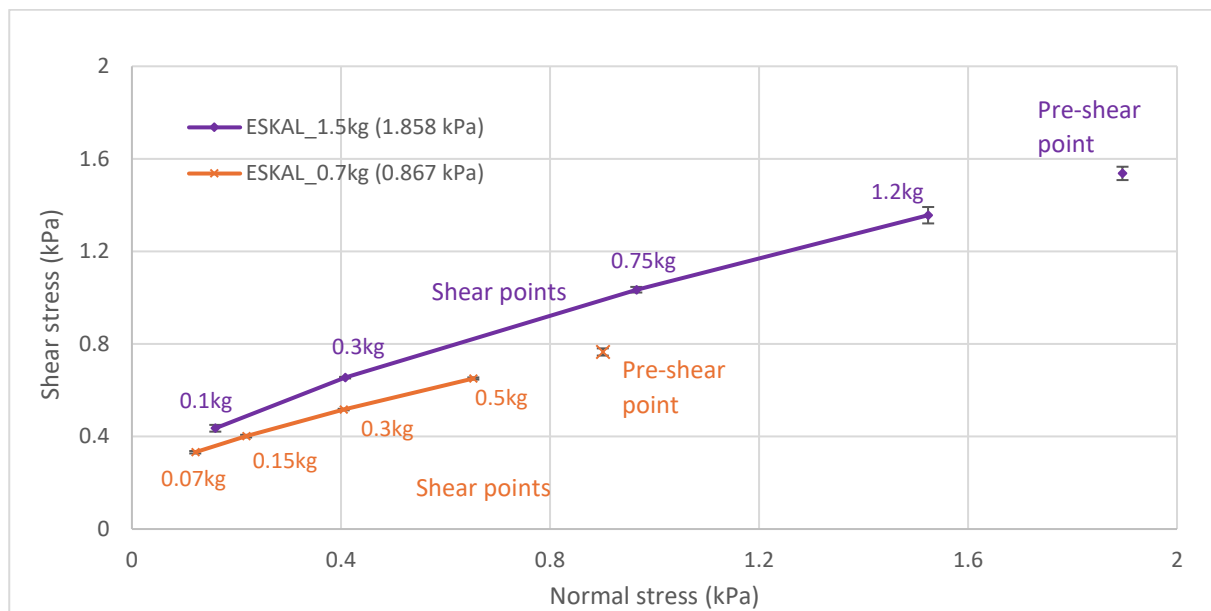


Figure 4-5: Shear points and pre-shear point of ESKAL 500 from experiments at 0.7 kg (0.867 kPa) & 1.5 kg (1.858 kPa) pre-shear normal load.

The DOE runs yielded the output responses w.r.t the input variables which were used in the objective functions for optimisation. A multi-objective genetic algorithm was used to minimize the objective functions and to calibrate the DEM model. The objective functions were formulated as the error functions between the experimental and the modelling response. Altair HyperStudy was utilized for model calibration and the Multi-Objective Genetic Algorithm (MOGA) (Altair HyperStudy, 2023b) was run for the specified population size and generations, the output responses were fed into HyperStudy and the Fit Automatically Selected by Training (FAST) method (Altair HyperStudy, 2023c) was used to fit the data from the DOE runs. MOGA was run to minimize the objective functions, and its convergence produced the optimal Pareto front.

MOGA is an evolutionary optimization method that can simultaneously optimize multiple objective functions, these functions can be complementary or competing in nature. The algorithm begins with an initial population of candidates, where each individual represents a unique combination of the input model parameters, the DOE runs were included in this initial population. The population size determines the number of candidate solutions evaluated in each generation.

During each generation, the fitness of each individual is evaluated according to the specified objective functions. Fittest individuals from each generation are selected for producing offspring through crossover and mutation, allowing the search space to be explored while progressively improving solution quality. Individuals with highest fitness values are identified as elite individuals and passed directly to the next generation, this elite population was set as 10% of the total population size in this study. The fitness of each of these individuals in the new generation is evaluated, which is followed by reproduction and elite population being passed to the next generation. The number of generations thus defines how many times this evolutionary process is repeated.

When optimizing multiple objectives simultaneously, MOGA identifies a set of non-dominated solutions known as the Pareto front. A solution is considered non-dominated if no other solution can improve one objective without simultaneously worsening at least one other objective. The Pareto front therefore represents the set of optimal solutions with respect to all objectives considered.

The DEM model developed was tested for its efficacy by replicating the experimental shear test, for this purpose, shear tests with four incipient failure points were run across varying pre-shear normal loads, the pre-shear normal loads were 0.7 kg (0.867 kPa), 1kg (1.239 kPa), 1.2 kg (1.486 kPa) and 1.5 kg (1.858 kPa). These were chosen as the experiments were also conducted at these pre-shear normal loads. The yield loci were plotted, and the major principal stress and unconfined yield strength were derived from the data to plot the flow function.

4.3 Parametric sensitivity analysis and DEM model calibration

4.3.1 One factor at a time (OFAT) study

The results of the OFAT study conducted for ascertaining the sensitivity of $\mu_{s\ p-g}$, $\mu_{r\ p-g}$, COR_{p-g} , $\Delta\gamma$ and COR_{p-p} can be found in Table 4-2. The particle geometry static friction $\mu_{s\ p-g}$ up to 0.5 had negligible influence on the shear stress, indicating that the bulk response was primarily governed by particle-particle interactions, therefore a value of 0.5 was selected for the MELS DOE set of simulations, $\mu_{s\ p-g}$ of 0.8 caused the shear stress to be unstable as seen from Figure 4-6, likely due to excessive wall friction promoting stick-slip behaviour. The shear stress increased with increasing values of $\mu_{r\ p-g}$, a value of 0.2 was chosen for the MELS DOE set of simulations as the runs showed that it influences shear stress, and it was not suitable to have it switched off. COR_{p-g} was found to be not impacting the shear stress, therefore a value of 0.175 was

selected. Similarly, as seen from Table 4-2, COR_{p-p} had little impact on the shear stress, the instability in the shear stress at COR_{p-p} of 0.1 as seen in Figure 4-7 was due to the larger time step of 7.5% of T_R , as reducing the time step to 5% of T_R , stabilised the system. , a value of 0.175 for COR_{p-p} at a time step of 7.5% of T_R was thus chosen for the MELS DOE simulations.

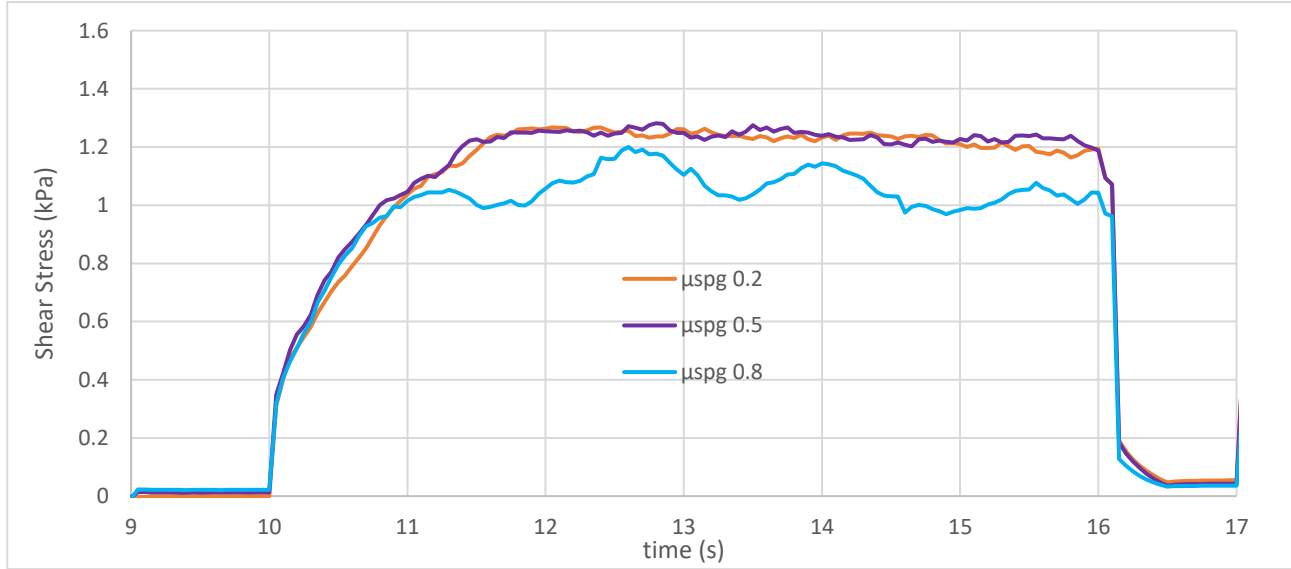


Figure 4-6: Particle-geometry static friction influence on shear stress.

The duration of collision t_c for two colliding spheres modelled with a linear spring-dashpot system is related to the coefficient of normal restitution e_n as $e_n = \exp\left(-\frac{\gamma_n}{2m^*} t_c\right)$ (Kruggel-Emden et al., 2007; Schäfer et al., 1996), where γ_n is damping constant and m^* is the effective or equivalent mass of bodies in contact. Consequently, the duration of collision can be expressed as $t_c = -\frac{2m^*}{\gamma_n} \ln e_n$, which means a lower restitution coefficient leads to a longer collision duration. Therefore, a smaller simulation time-step is required for resolving the particle contact/collision over several time-steps. This explains the stability in the simulations for COR_{p-p} of 0.1, when a time step of 5% of T_R is used. A value of 0.175 for COR_{p-p} at a time step of 7.5% of T_R is thus chosen for the simulations in this study, as this provides for numerically stable yet faster simulations.

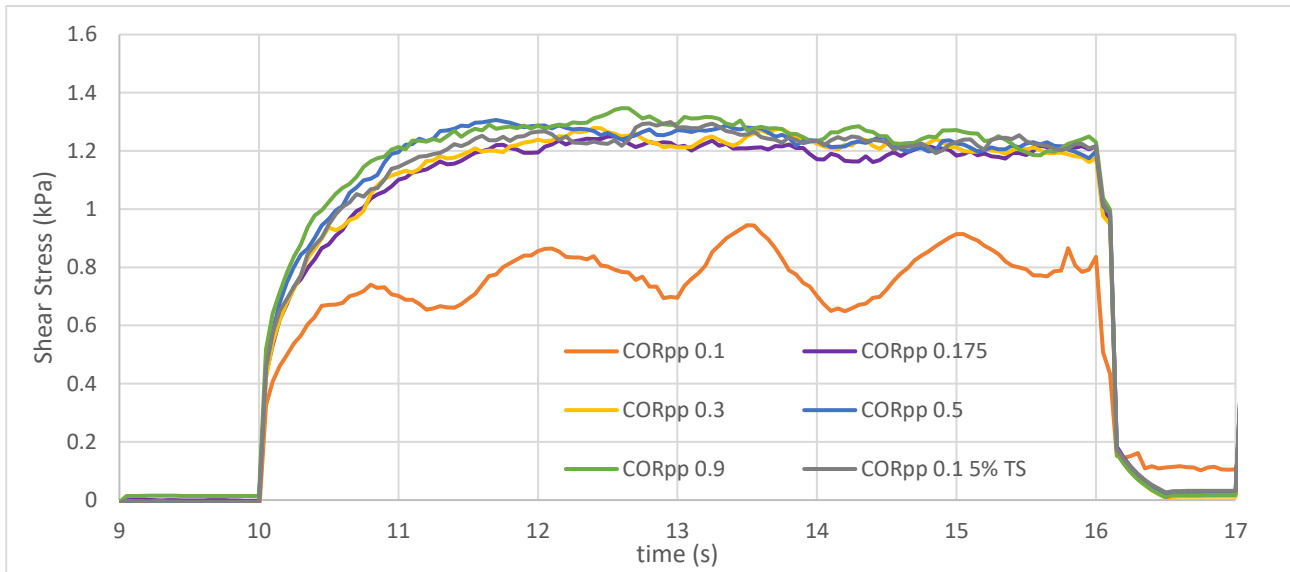


Figure 4-7: Particle-particle coefficient of restitution influence on shear stress.

Table 4-2: OFAT study results.

Properties	
$\mu_{s\ p-g}$	Shear Stress (kPa)
0.2	1.236
0.5	1.236
0.8	1.060
$\mu_{r\ p-g}$	Shear Stress (kPa)
0	1.236
0.25	1.319
0.5	1.397
COR_{p-g}	Shear Stress (kPa)
0.175	1.195
0.2	1.236
0.5	1.241
0.8	1.233
$\Delta\gamma$ (J/m ²)	Shear Stress (kPa)
0.1 ($f_o = 0.0002$ N)	1.059
1 ($f_o = 0.0002$ N)	1.110
0.1 ($f_o = 0.002$ N)	1.212
1 ($f_o = 0.002$ N)	1.224
0.1 ($f_o = 0.01$ N)	1.649
1 ($f_o = 0.01$ N)	1.668
COR_{p-p}	Shear Stress (kPa)
0.1	0.795
0.1 (5% TS)	1.243
0.175	1.200
0.3	1.232
0.5	1.242

0.9	1.270
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Table 4-3 lists the constant pull off force and the surface energy values and their respective contributions to the Bond number Bo_g , as can be seen from Table 4-3, f_o is the major contributor to the cohesive forces and hence to Bo_g . The shear stress variation when the surface energy was changed from 0.1 to 1 across the constant f_o values was not significant as seen in Table 4-2 and thus a constant $\Delta\gamma$ value of 0.1 was chosen. On the other hand, f_o had a significant impact on the shear stress values and thus was chosen as one of the parameters requiring calibration.

Table 4-3: Constant pull off force f_o and surface energy $\Delta\gamma$ contributions to Bond number.

f_o (N)	$\Delta\gamma$ (J/m ²)	$\Delta\gamma$ contr.	f_o contr.	Bo_g
0.0002	0.1	0.28	2.22	2.51
0.0002	1	2.80	2.22	5.02
0.002	0.1	0.28	22.24	22.52
0.002	1	2.80	22.24	25.04
0.01	0.1	0.28	111.21	111.49
0.01	1	2.80	111.21	114.01

4.3.2 Full factorial parametric sensitivity study

The shearing resistance of the granular assembly is governed by the combined effects of particle stiffness, static friction, and rolling friction, each influencing friction mobilization at the contact level. The Young's modulus of the particles impacts its deformation, a stiffer particle with higher E would deform lesser compared to a softer particle when under a similar applied force. Softer particles can deform excessively at the contacts preventing the normal contact forces and force chains to develop fully. This impacts the development of the tangential contact forces as they are limited by the Coulombic limit, as a result the full shearing resistance of the particles is not realized. Lommen et al. (2014) found that particles having stiffness below a threshold limit produced excessive normal overlaps which impacted the angle of repose and bulk density of the system. The static friction impacts the Coulombic limit of the tangential contact forces and determines the limit at which the particles begin to slide. The static friction is thus critical to simulate the experimental shearing resistance as the particles begin to slide once the friction is fully mobilized. Rolling friction helps to simulate the particle shape in DEM and allows for the simplification of the real particle shape (Wensrich and Katterfeld, 2012). Rolling friction provides a resisting torque to the particles to limit their rolling and rearrangement, which enhances particle interlocking and stabilizes force chains. This can increase the mobilization of tangential contact forces and consequently the overall shearing resistance of the granular assembly.

Table 4-4 shows the variation of shear stress across the different levels of the parameters, $\mu_{s\ p-p}$, $\mu_{r\ p-p}$ and E . For the cases where E was 10^6 Pa, the shear stress values were lower than the other cases, indicating the particles to be soft and imposing lower force or torque on the shearing lid. The shear stress varied with varying values of static and rolling frictions at $E = 10^7$ & 10^8 Pa, but they mostly fluctuated or varied around a mean of 1200 Pa. The particle Young's modulus of 10^7 Pa gives significant simulation run time savings over the 10^8 Pa cases. The shear stress depends on the interaction of the static and rolling friction coefficients. The E values of 10^7 and 10^8 Pa provide stable systems for the various combinations of static and rolling frictions with the systems with 10^6 Pa being highly sensitive to the friction coefficients. For $E = 10^7$ Pa, the interplay between the static and rolling friction coefficients was largely predictable and on expected grounds, with the shear stress increasing with the increasing rolling friction coefficient across the constant static friction.

Table 4-4: Full factorial study of $\mu_{s\ p-p}$, $\mu_{r\ p-p}$ and E across three levels.

Factor	A ($\mu_{s\ p-p}$)	B ($\mu_{r\ p-p}$)	C (E) (Pa)	Shear Stress (kPa)
Level	0.2	0	10^6	0.774
	0.2	0.25	10^6	0.794
	0.2	0.5	10^6	0.836
	0.5	0	10^6	0.563
	0.5	0.25	10^6	0.587
	0.5	0.5	10^6	0.582
	0.8	0	10^6	0.277
	0.8	0.25	10^6	0.281
	0.8	0.5	10^6	0.277
Factor	A ($\mu_{s\ p-p}$)	B ($\mu_{r\ p-p}$)	C (E) (Pa)	Shear Stress (kPa)
Level	0.2	0	10^7	1.085
	0.2	0.25	10^7	1.172
	0.2	0.5	10^7	1.256
	0.5	0	10^7	1.195
	0.5	0.25	10^7	1.212
	0.5	0.5	10^7	1.361
	0.8	0	10^7	1.185
	0.8	0.25	10^7	1.241
	0.8	0.5	10^7	1.331
Factor	A ($\mu_{s\ p-p}$)	B ($\mu_{r\ p-p}$)	C (E) (Pa)	Shear Stress (kPa)
Level	0.2	0	10^8	1.106
	0.2	0.25	10^8	1.162
	0.2	0.5	10^8	1.296
	0.5	0	10^8	1.150
	0.5	0.25	10^8	1.193
	0.5	0.5	10^8	1.164
	0.8	0	10^8	1.171
	0.8	0.25	10^8	1.264
	0.8	0.5	10^8	1.197

The stiffness (E) of the particles across the 9 runs seen in Figure 4-8 was comparatively low (10^6 Pa), meaning the particles were softer when compared to the cases where stiffness (E) of the particles was 10^7 or 10^8 Pa. The shear stress is calculated by the torque experienced by the shearing lid due to the particles. The shear stress values in this case were quite low compared to the cases where E was 10^7 or 10^8 Pa. This can probably be attributed to the “softer” particles as they would undergo greater deformation during contact, resulting in lower contact force transmission throughout the particle assembly. Consequently, lesser shearing resistance is generated, leading to lower shearing resistance torque recorded by the lid, and therefore lower calculated shear stress in the sample. The impact of static friction (SF) was quite significant on the values of shear stress recorded as seen in Figure 4-8, with the SF of 0.2 leading to shear stress values almost thrice that off SF of 0.8. The rolling friction did not play a significant role for static friction cases of 0.5 & 0.8, with it showing some impact when the static friction was 0.2. The average particle overlap during the pre-shear was around 1% for the 10^6 Pa cases whereas it was around 0.16% for the 10^7 Pa cases, which might be the reason for their unexpected behaviour of increased shear stress at lower static friction values. Lommen et al. (2014) noted that an average normal overlap of 0.3% provided stable systems with Cleary (2010) suggesting an overlap range of 0.1-0.5% for simulating flow behaviour independent of spring stiffness. The average particle overlap (%) can be calculated as; (Average normal particle overlap / Major dimension of dual-sphere particle) * 100.

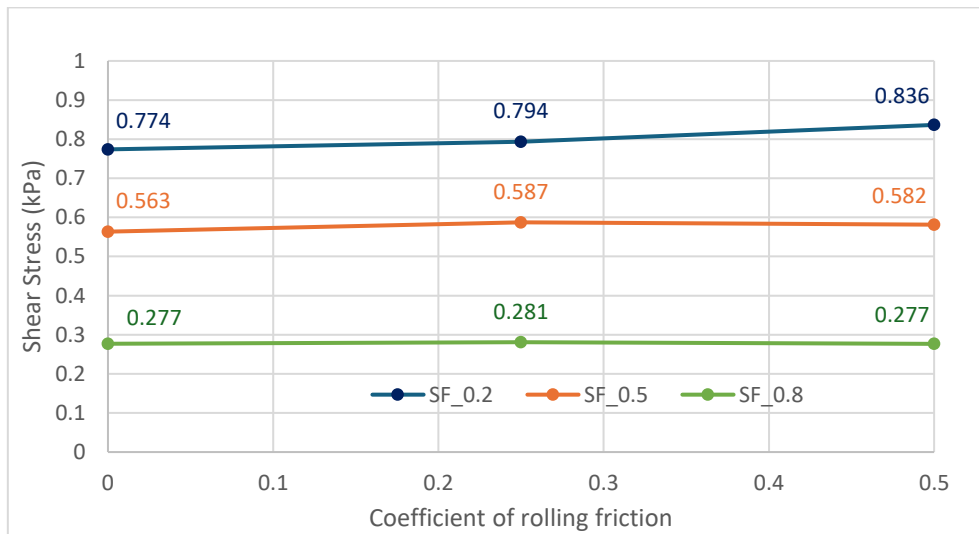


Figure 4-8: Full factorial parametric study at $E = 10^6$ Pa

The variation of shear stress for the 9 cases where E was 10^7 Pa can be seen in Figure 4-9, the shear stress values were maximum for the cases where the rolling friction coefficient was 0.5 at constant values of static friction, indicating interaction of the static and rolling friction coefficients responsible for the shear stress. The shear stress was lowest when the static and rolling friction were at their lowest values of 0.2 & 0 respectively. The mean value of the shear stress was around 1.2 kPa and the values varied between 1.085 kPa (SF = 0.2 & RF = 0) and 1.361 kPa (SF = 0.5 & RF = 0.5) indicating an interplay between the static and rolling friction. The

increase in the rolling friction values at a particular static friction value led to an increase in the shear stress values.

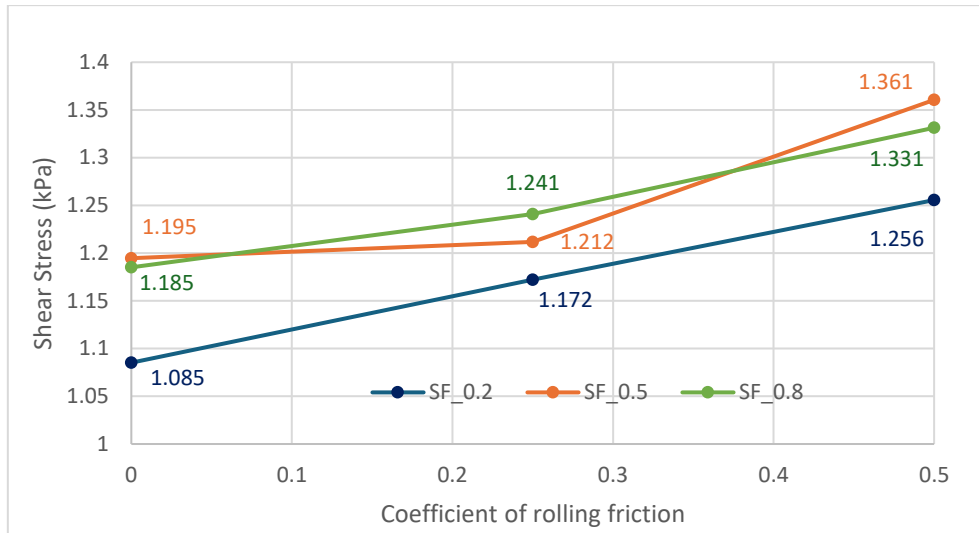


Figure 4-9: Full factorial parametric study at $E = 10^7$ Pa

As can be inferred from Table 4-4 there was a small difference (less than 5% on average) in the shear stress for the cases when E was 10^7 Pa and 10^8 Pa (Figure 4-10), but the simulation was much faster when E was 10^7 Pa. The modulus of elasticity of 10^6 Pa was not able to produce the shear stress near the experimental value of 1.5 kPa for the varying combinations of static and rolling frictions, therefore a value of 10^7 Pa was chosen as the modulus of elasticity for the next set of simulations. The full factorial sensitivity study shows that the particle-particle static and rolling friction coefficients significantly influence the shear stress and therefore require calibration.

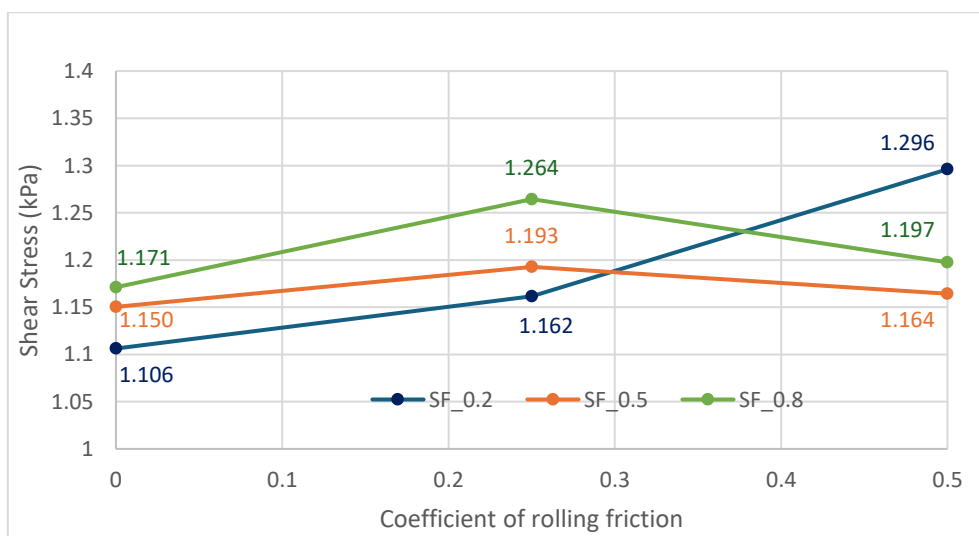


Figure 4-10: Full factorial parametric study at $E = 10^8$ Pa

The plasticity ratio λ_p was varied across two levels, 0.5 & 0.9; $\mu_{r\ p-p}$ was selected as 0.25, $\mu_{s\ p-p}$ as 0.5 and E as 10^7 Pa (the median values of the full factorial study), with the other parameters listed in Table 4-1. The plasticity ratio, $\lambda_p = 1 - \frac{k_1}{k_2} = \frac{\delta_p}{\delta}$ represents the contact plasticity and can be used to calculate the plastic deformation δ_p of the particles which remains after the powder bed is partially unloaded. A higher plasticity ratio of 0.9 means that there is a lower elastic recovery or a higher deformation of particles remaining after the normal load is lowered for the shearing step, as compared to the case where it is 0.5. The higher plasticity ratio leads to a higher contact area which leads to higher cohesive forces between the particles. This effect can be clearly seen in the shearing strength at the varying normal loads, the cases where λ_p is 0.9, the failure strength of the sample is higher as seen in Figure 4-11, meaning higher cohesive forces due to increased plastic deformations; therefore, to match the powder flow behaviour λ_p needs to be calibrated.

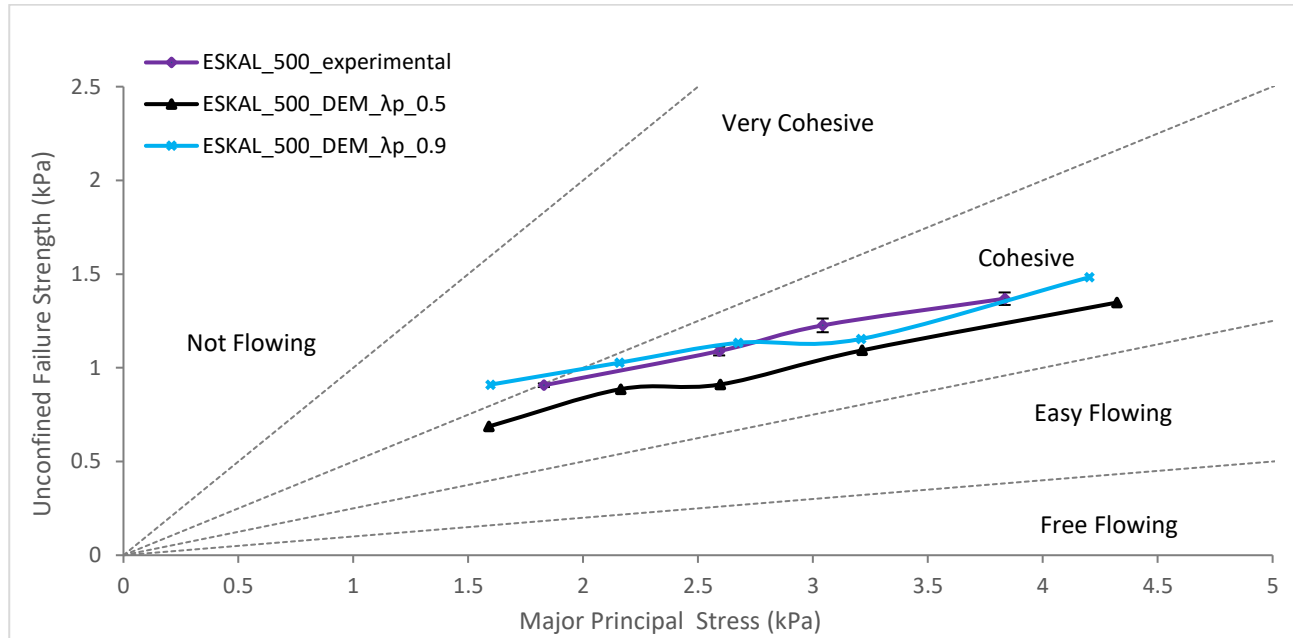


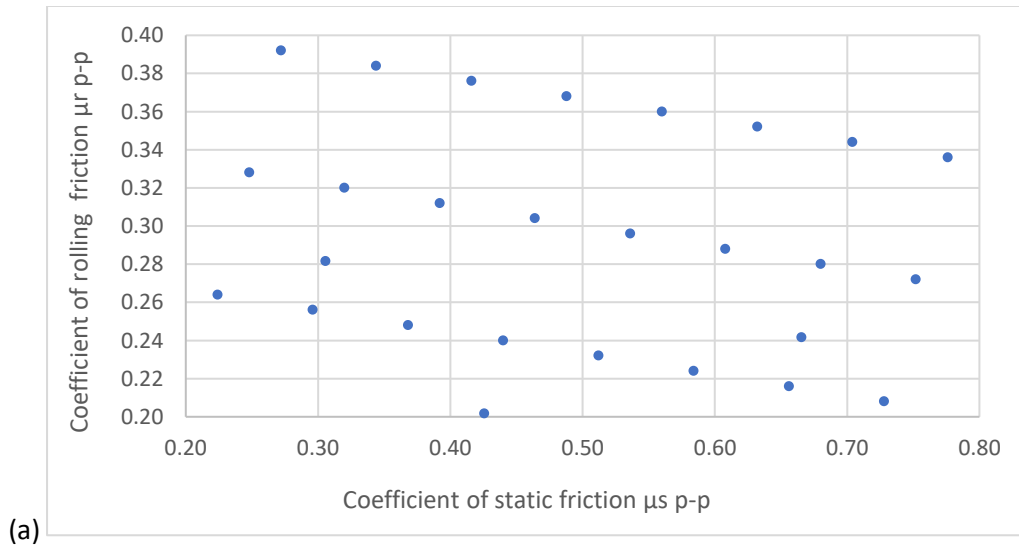
Figure 4-11: Experimental and DEM flow function of ESKAL 500 at varying λ_p .

4.3.3 Model calibration by using responses from DOE runs

The OFAT and full factorial studies helped determine the key parameters for calibration, which are $\mu_{s\ p-p}$, $\mu_{r\ p-p}$, λ_p and f_o . Pre-shear shear stress was highly dependent on static friction, rolling friction & van der Waals forces, the failure strength was highly dependent on plasticity ratio. For the simulations used for calibration, particle polydispersity was introduced with a narrow normal particle size distribution. The normalized standard deviation of 0.1 was used and the minimum and maximum particle sizes were capped at 0.8 and 1.2 of the mean particle radius respectively, to reduce the excessively fine and coarse particles. A dual-sphere particle with an aspect ratio of 1.2, comprised of two overlapping spheres of 1.4 mm each was used to model the particles in this part of the study. The properties determined in the previously discussed OFAT and full factorial studies were used with these being; $\mu_{s\ p-g} = 0.5$, $\mu_{r\ p-g} = 0.2$, $E = 10^7$ Pa, $COR_{p-p} =$

$COR_{p-g} = 0.175$ and $\Delta\gamma = 0.1 \text{ J/m}^2$. The particle density of the dual sphere particle was 4000 kg/m^3 , with that of steel used to represent the equipment material having particle density of 7000 kg/m^3 and modulus of elasticity as $2.1 \times 10^{11} \text{ Pa}$. Poisson's ratio for both the bulk material and steel was 0.25. EEPA model as shown in Figure 4-2 was used with the tangential stiffness multiplier of 0.667.

Modified Extensible Lattice Sequence (MELS) DOE (Figure 4-12) was used to explore the design space as a full-factorial three level study of 4 parameters with 81 runs each at a single pre-shear normal load level was computationally expensive and may not be necessary, moreover the full-factorial study might not be able to explore the empty areas of the design space. 27 runs each at two pre-shear normal loads were run with MELS DOE to explore the design space. The pre-shear normal loads of 1.5 kg (1.858 kPa) and 0.7 kg (0.867 kPa) were chosen as to represent the maximum and minimum pre-shear load used in the experiments and to increase the robustness of the calibration process. Selecting the upper and lower bounds of the parameters is as important as selection of the parameters requiring calibration as the design exploration depends on this and narrowing or widening of these bounds can cause insufficient or unnecessary runs. The study conducted before which produced the flow function as seen in Figure 4-11, helped determine these bounds, with them being as follows; $\mu_{s \text{ p-p}}$ [0.2, 0.8], $\mu_{r \text{ p-p}}$ [0.2, 0.4], λ_p [0.5, 0.9] and f_o (N) [-0.005, -0.001]. The design space was thus explored with these bounds and 27 runs with varying values of these 4 parameters. Each run started from the start, as f_o primarily and the other parameters can impact the particle packing in the powder bed. The sample preparation along with the shear testing procedure was similar to the one described earlier.



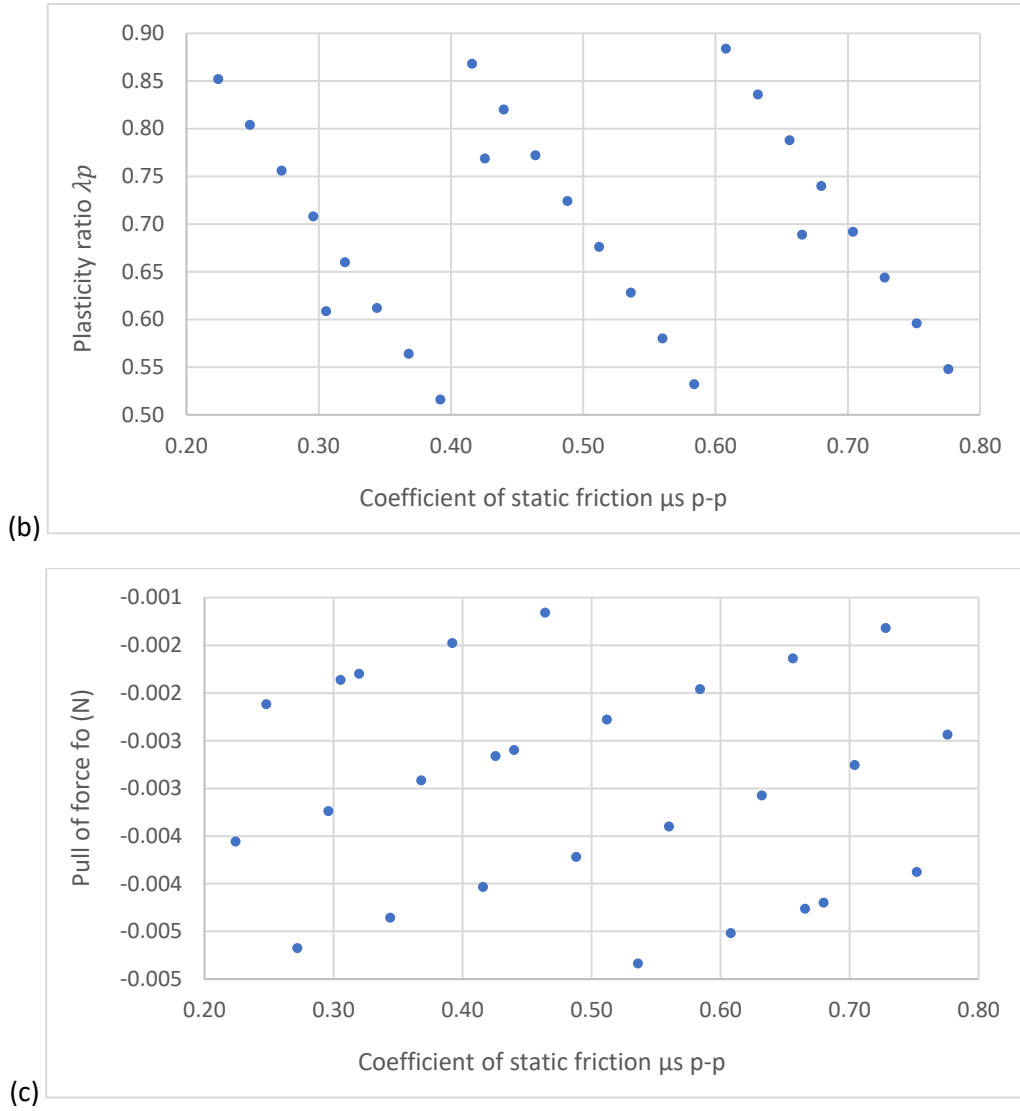


Figure 4-12: DEM model parametric values of MELS DOE runs to explore the design space, for a) $\mu_{s p-p}$, $\mu_{r p-p}$, b) $\mu_{s p-p}$, λ_p and c) $\mu_{s p-p}$, f_o .

$$O_1 = |\tau_{pre(1.5),sim} - \tau_{pre(1.5),exp}| + |\tau_{sh(0.3:1.5),sim} - \tau_{sh(0.3:1.5),exp}|$$

$$O_2 = |\tau_{pre(0.7),sim} - \tau_{pre(0.7),exp}| + |\tau_{sh(0.15:0.7),sim} - \tau_{sh(0.15:0.7),exp}|$$

$$O_3 = |\varepsilon_{x,pre(1.5),sim} - \varepsilon_{x,pre(1.5),exp}|$$

$$O_4 = |\varepsilon_{x,pre(0.7),sim} - \varepsilon_{x,pre(0.7),exp}|$$

The objective functions O_1 and O_2 , represent the combined error in calculating the pre-shear shear stress and the corresponding shear to failure shear stress as seen in Figure 4-13, with O_1 representing the values determined at 1.5 kg (1.858 kPa) pre-shear normal load with the normal load at shear to failure as 0.3 kg, O_2 represents the values determined at 0.7 kg (0.867 kPa) pre-shear normal load and normal load at shear to failure as 0.15 kg. The objective functions O_3 and O_4 represent the compressive strain in the sample during the pre-shearing step at pre-shear normal loads of 1.5 kg (0.867 kPa) & 0.7 kg (0.867 kPa) respectively. The *sim* and *exp* prefixes in the objective functions denote the values determined from the simulations and

experiments respectively. τ_{pre} is the average value of the pre-shear stress, τ_{sh} denotes the failure shear stress, $\varepsilon_{x,pre}$ is defined as the ratio of the compression in the powder bed during pre-shearing, measured as the distance travelled by the shearing lid during pre-shearing, to the original bed height at initiation of pre-shearing.

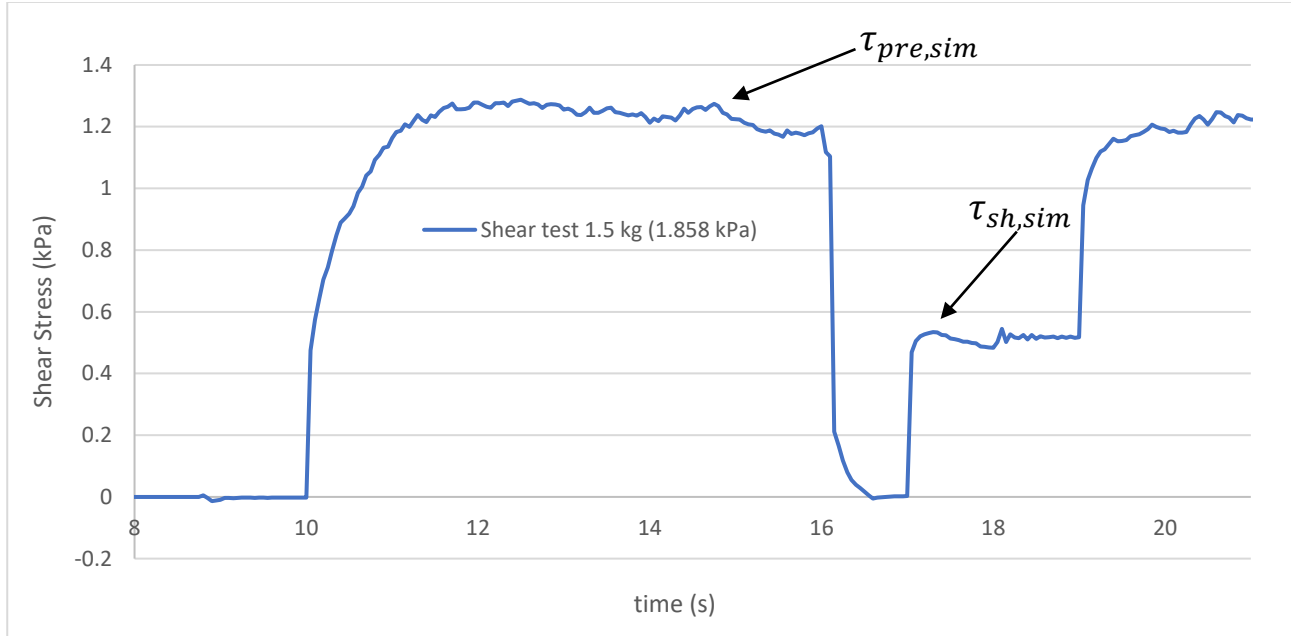
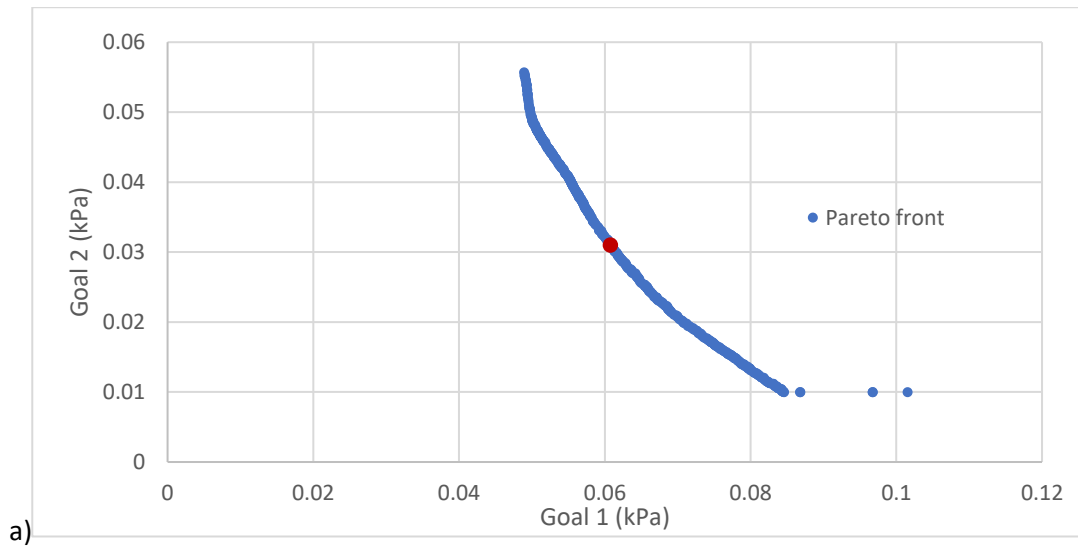


Figure 4-13: Determination of $\tau_{pre,sim}$ and $\tau_{sh,sim}$ from the first pre-shear and shearing point.

Fit Automatically Selected by Training (FAST) method was used to fit the data from the 27 runs, and the values of the objective functions were evaluated from the fit model. MOGA's population size was determined from the relation, $Population\ size = \left\lceil 3 + 37e^{-\left(\frac{P-1}{5.806}\right)^{0.5}} \right\rceil P$, recommended by (Altair HyperStudy, 2023b), where P is the number of input variables. MOGA was run for the specified population size (84) and generations (200) to minimize the objective functions O_1 and O_2 , it converged after 126 generations to give the optimal Pareto front. The optimal parameters had a very high frequency of occurrence, meaning that the optimal parameter set was highly repeatable. The standard deviation in the values of the optimal solutions from the Pareto front is as follows: $\mu_{r\ p-p}$: 0.0089, $\mu_{s\ p-p}$: 0.0307, λ_p : 0.0118, f_o (N): 0.00002931. The Pareto-optimal solutions obtained from MOGA were concentrated within a narrow region of the parameter space, as indicated by the low standard deviations of the optimized parameters. The two objective functions were not competing and therefore produced a cluster of optimal solutions, indicating convergence toward a stable region in the decision space. Consequently, the mean values of the optimal solutions from the Pareto front were considered as representative of the converged solution region. The mean values were as follows: $\mu_{r\ p-p}$: 0.3744, $\mu_{s\ p-p}$: 0.4544, λ_p : 0.6834, f_o (N): -0.002371. The optimal parameter set was selected as the Pareto solution (red dot in Figure 4-14a) lying closest to this mean value or centroid of all Pareto-optimal solutions. This ensures that the chosen point is both feasible and non-dominated, while still being representative of the

convergence region. The optimal parameter set therefore was as follows: $\mu_{r\ p-p}$: 0.3746, $\mu_{s\ p-p}$: 0.4509, λ_p : 0.6849, f_o (N): -0.002375. The optimal parameter set was verified by running the simulations at 1.5 kg (1.858 kPa) and 0.7 kg (0.867 kPa) normal loads for the pre-shear and subsequent shear point and comparing these values with their experimental counterparts. For the 0.7 kg case, the relative error was 1.1% for the pre-shear shear stress, and 4.2% for the subsequent shear point. For the 1.5 kg case, the corresponding errors were 8.9% and 1.9%, respectively. These results indicate that the calibrated parameter set reproduces the experimental behaviour with reasonable accuracy at both calibration points, with slightly higher deviation observed in the pre-shear response at the higher normal load. Following verification, the calibrated parameter set was utilized to compute the full flow function by running simulations for the full shear test (four shear points) across four pre-shear normal load levels.

MOGA was also run with the objective of minimizing the objective functions O_1 , O_2 , O_3 and O_4 with the population size of 84 and 400 generations, it did not converge but gave optimal points on the Pareto front as the solution. Figure 4-14b shows the Pareto front for the optimal solutions for Goal 1 (minimizing O_1) and for Goal 3 (minimizing O_3), as can be seen minimizing the error in O_3 causes the error in O_1 to rise, and vice versa, some solutions can be identified which do not individually minimize the functions O_1 , O_2 , O_3 and O_4 , but provide acceptable levels of errors for each of these.



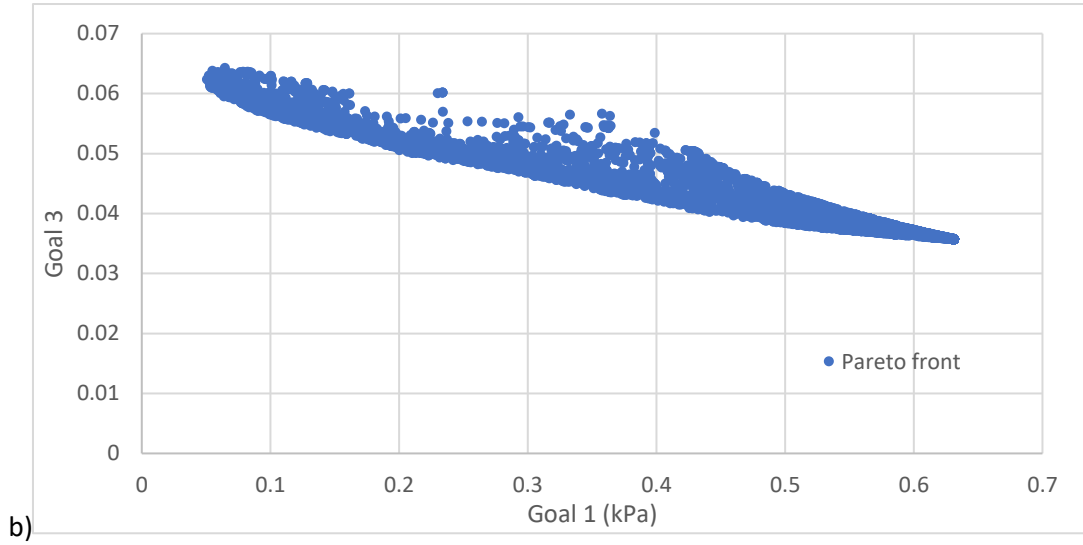


Figure 4-14: Pareto front for optimization of O_1 and O_2 (a), and optimization of O_1 , O_2 , O_3 and O_4 (b).

The reason for the algorithm to converge for the functions O_1 and O_2 and not converge for the functions O_1 , O_2 , O_3 and O_4 is that in the former, both the functions are not competing and are complementary, whereas in the latter, functions O_3 and O_4 are competing with the functions O_1 and O_2 . The functions O_3 and O_4 give the least error at the increased values of f_o , as higher van der Waals forces lead to looser packing of the particle bed and hence more compression of the powder bed during pre-shearing, leading to lesser error in the simulated and experimental axial strains. Higher values of f_o lead to more cohesive behaviour of the particles leading to higher errors in the pre-shear and failure shear stress. The objective of the calibration process is to be able to produce the experimental flow behaviour which would be verified by comparing the experimental and simulation flow function, matching the experimental powder bed compression quantitatively is desirable but not the primary objective, for this purpose, the parameter set obtained from optimizing O_1 and O_2 is considered for the calibration of the system.

An alternative study which combined the pre-shear shear stress & failure strength across two pre-shear normal load levels into a single function and bed compression across two pre-shear normal load levels into a single function was also conducted.

$$O_1 = |\tau_{pre(1.5),sim} - \tau_{pre(1.5),exp}| + |\tau_{sh(0.75:1.5),sim} - \tau_{sh(0.75:1.5),exp}| + |\tau_{pre(0.7),sim} - \tau_{pre(0.7),exp}| + |\tau_{sh(0.15:0.7),sim} - \tau_{sh(0.15:0.7),exp}|$$

$$O_2 = |\Delta_{x,pre(1.5),sim} - \Delta_{x,pre(1.5),exp}| + |\Delta_{x,pre(0.7),sim} - \Delta_{x,pre(0.7),exp}|$$

The optimization study converged but faced the same issue as the last one, which is higher values of f_o led to more cohesive behaviour of the particles leading to higher errors in the pre-shear and the failure shear stress, although the error in O_2 was reduced. The optimal parameter set was not repeatable, and the parameters were spread over the range defined by the parameter bounds.

The DEM model developed was tested for its efficacy by replicating the experimental shear test, for this purpose, shear tests with four incipient failure points were run across varying pre-shear normal loads, the pre-shear normal loads are 0.7 kg (0.867 kPa), 1 kg (1.239 kPa), 1.2 kg (1.486 kPa) and 1.5 kg (1.858 kPa). These are chosen as the experiments are also conducted at these pre-shear normal loads.

The calibrated set of parameters were used to run the full shear test (four shear points) across four pre-shear normal load levels, the efficacy of the parameter set obtained through calibration with a single pre-shear and shear point across two pre-shear normal load levels in predicting the flow behaviour (full shear test) was thus checked. The four normal load levels and the shear to failure normal loads for each of these points were similar to the experimentally used values for the comparison to be robust. Figure 4-15 shows the shear stress time series curves across the varying pre-shear normal loads, the average shear stress during the pre-shear and the subsequent shear stress at failure were determined for plotting the yield loci for the varying pre-shear normal loads. The major principal consolidation stress σ_1 and the unconfined yield strength σ_c were derived from the yield loci and used to plot the flow function curve. The experimentally observed behaviour of shear stress reduction after reaching peak shear stress value during the shear to failure step is characteristic of dilation within the granular assembly. As illustrated in Figure 4-15, this behaviour was well captured by the DEM simulations for all the shear to failure steps across the 4 consolidation stress levels.

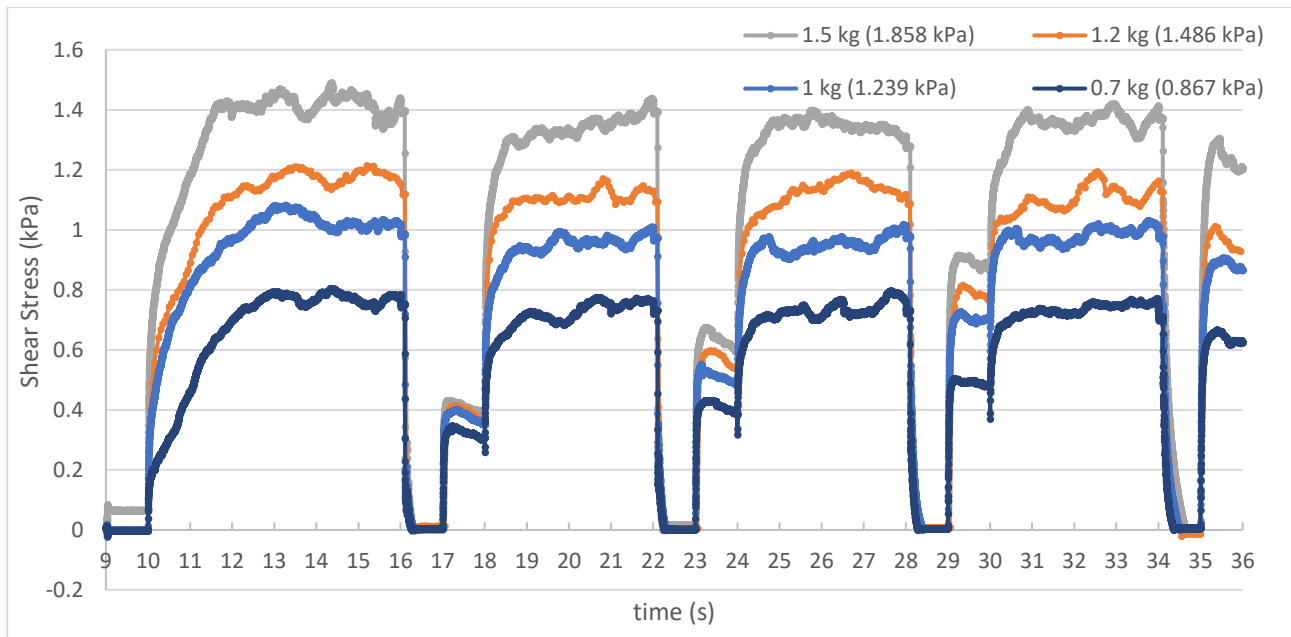
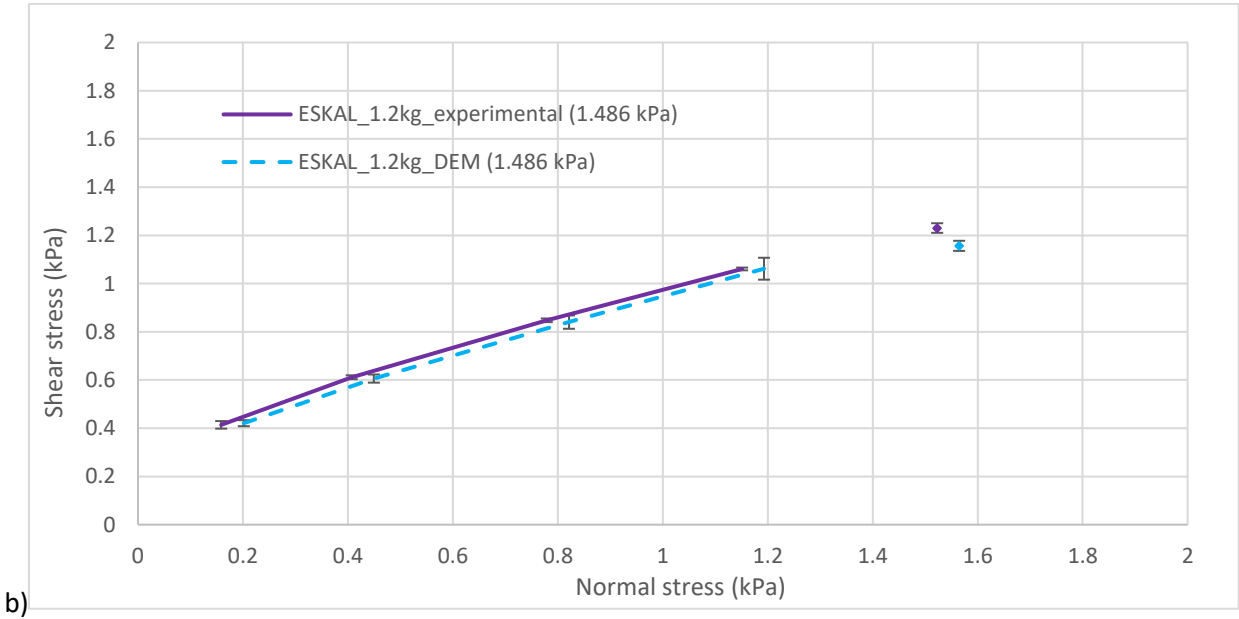
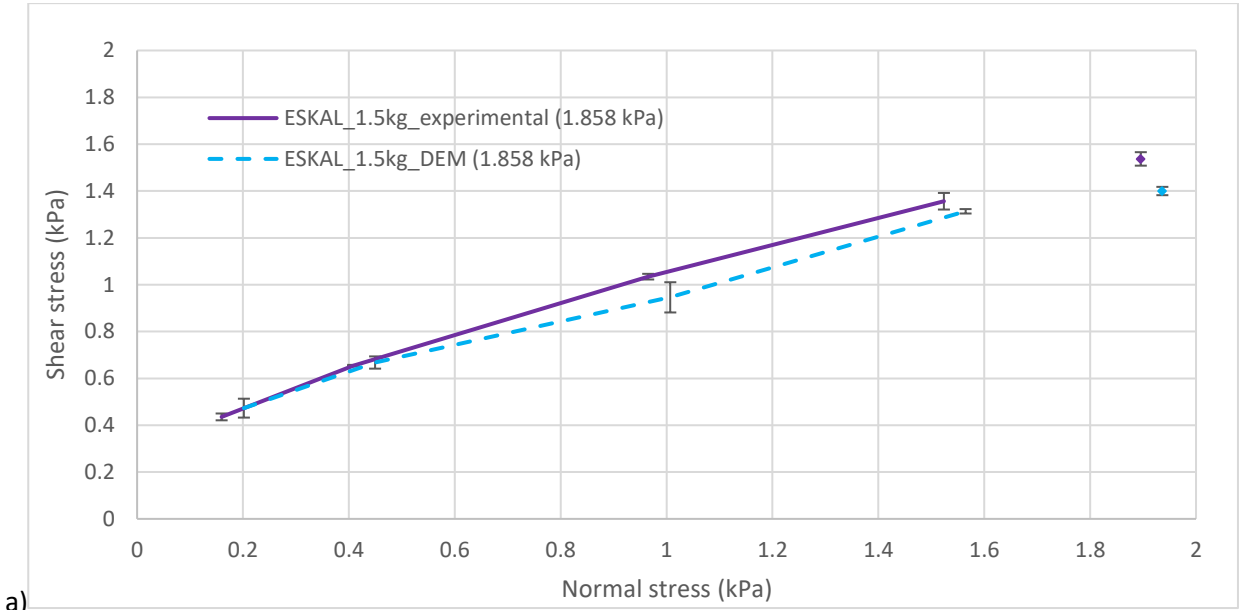


Figure 4-15: DEM shear stress time series curves at varying pre-shear normal loads.

The calibrated DEM model was able to match the experimental yield locus, with Figure 4-16 showing the DEM & experimental yield loci constructed at varying pre-shear normal loads. There was very good agreement between the DEM & experimental failure strengths at the incipient flow points, the pre-shear shear stress is slightly lower than the corresponding experimental value. Flow function and yield locus outputs such as σ_1 ,

σ_c , cohesion and effective angle of internal friction are compared, with these outputs showing good agreement with the corresponding experimental values as seen in Table 4-5.



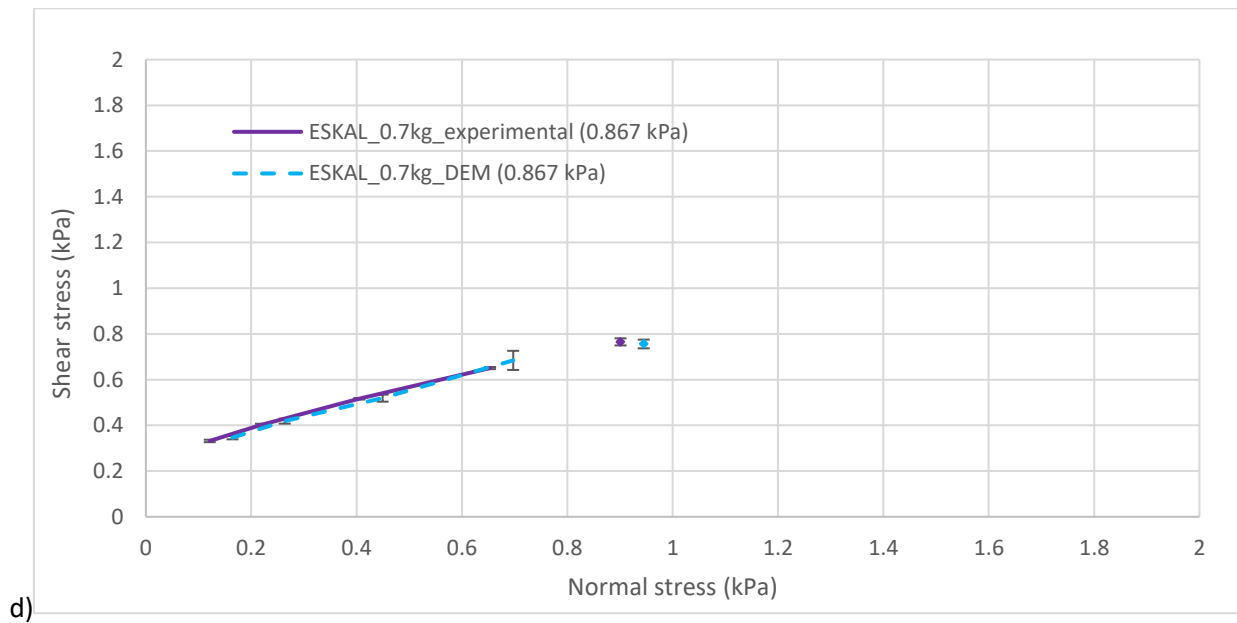
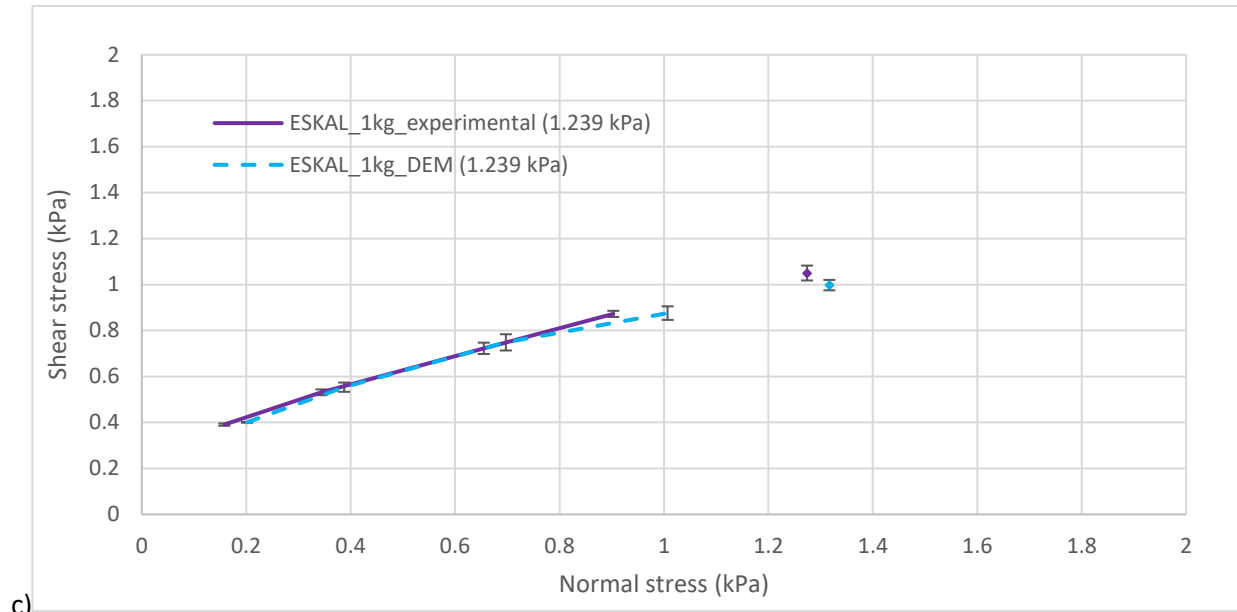
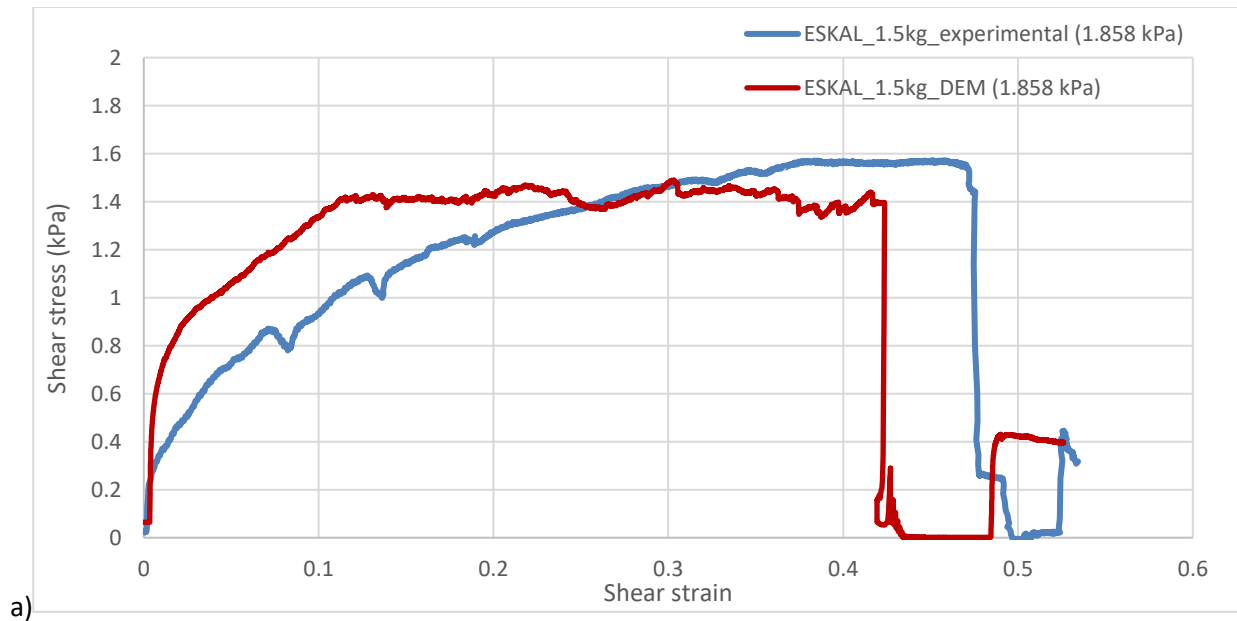


Figure 4-16: DEM & experimental yield loci at varying pre-shear normal loads of a) 1.5 kg (1.858 kPa), b) 1.2 kg (1.486 kPa), c) 1 kg (1.239 kPa), and d) 0.7 kg (0.867 kPa).

Figure 4-17 shows the shear stress evolution with the shear strain for the 1.5 kg (1.858 kPa), 1.2 kg (1.486 kPa) and 1 kg (1.239 kPa) pre-shear normal loading. At small strains the DEM model exhibits a stiffer response, with higher shear stress than the experimental material at equivalent strain levels, indicating an earlier mobilisation of shear resistance and early formation of load-bearing force chains when compared to the experiments. The first pre-shear and first shearing points were plotted for comparison, the DEM powder reached the steady state earlier than the experimental powder, although there was good agreement between the DEM & experimental pre-shear shear stress and failure strength. The upscaled mm range DEM particles likely achieve stable particle arrangement and stable contact network earlier than the micron sized real particles, causing the DEM powder to reach the steady state earlier. The shear strain in the stress-strain plots

of Figure 4-17 was determined from the elapsed simulation time according to $\gamma = \frac{r_{rad}}{L} \omega_{tr} t$, where r_{rad} is the radial distance of the powder element under consideration from the centre of rotation, L is the instantaneous powder bed height, and ω_{tr} is the trough angular velocity, and t is the elapsed simulation time. Consequently, the reported strain increases continuously throughout the simulation, including during the relaxation period following counter rotation of the sample, as seen for example the period from 16.3 – 17s in Figure 4-15. Therefore, the apparent increase in strain during the relaxation period does not correspond to imposed shear deformation, but rather to the continued advancement of simulation time.

The meso-scale DEM model was able to predict the powder flow function as seen in Figure 4-18; the experimental and DEM flow function was derived after averaging the values across three repetitions. The calibration process which used only two points from the shear test (pre-shear point & one shearing point) at the two pre-shear normal loads was able to predict the flow behaviour checked by comparing the flow function (produced at four pre-shear normal loads) with each point of the flow function generated by testing at four shearing points.



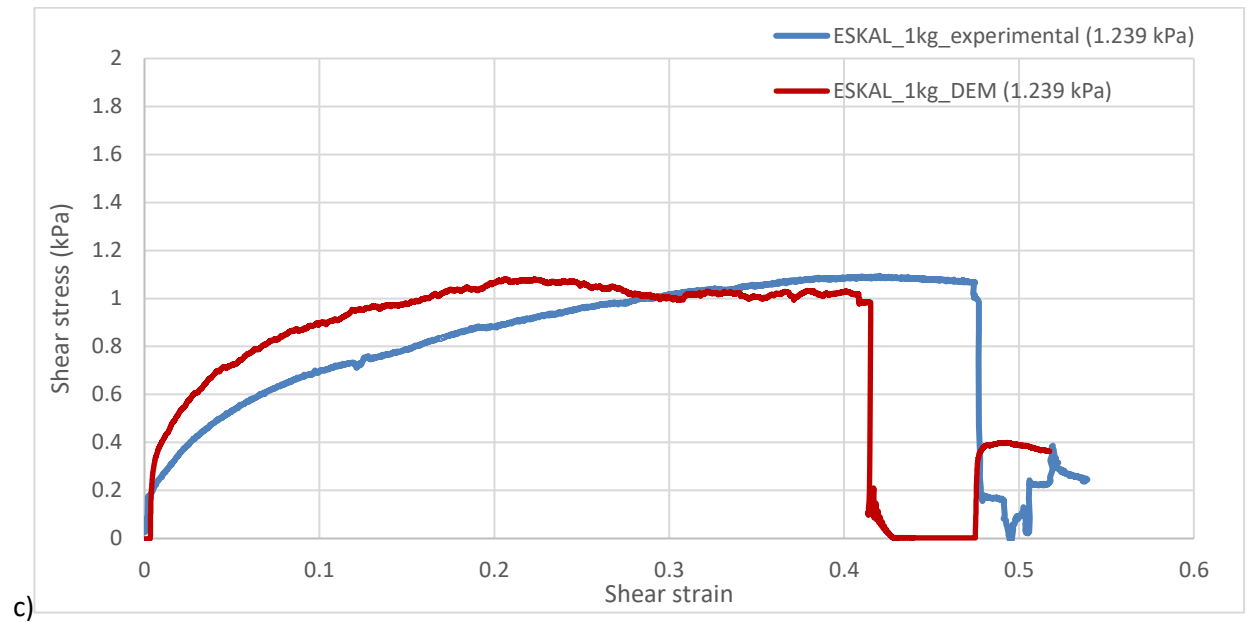
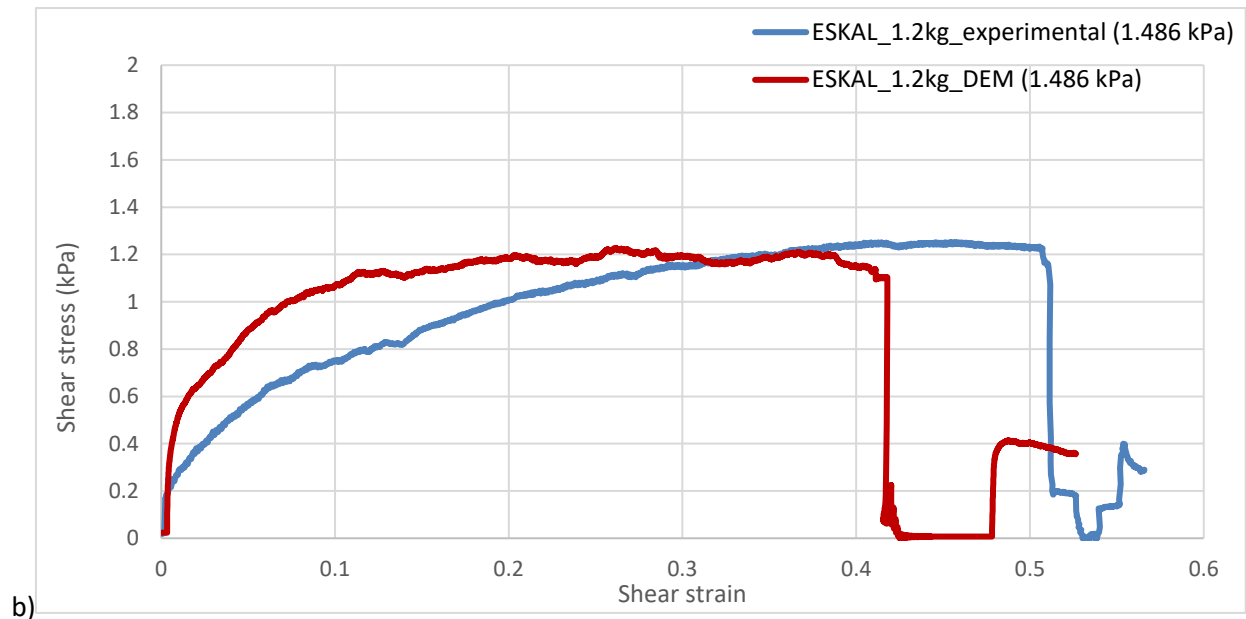


Figure 4-17: Shear stress – shear strain evolution for first pre-shear and first shear point at varying pre-shear normal loads of a) 1.5 kg (1.858 kPa), b) 1.2 kg (1.486 kPa), and c) 1 kg (1.239 kPa).

Table 4-5: a) Experimental and simulation results for ESKAL 500, and b) % error in simulations.

a)

Pre-shear normal load (kPa)	Experimental				Simulation			
	σ_1 (kPa)	σ_c (kPa)	Cohesion (kPa)	φ_e (°)	σ_1 (kPa)	σ_c (kPa)	Cohesion (kPa)	φ_e (°)
1.858	3.834	1.368	0.359	44.3	3.55	1.294	0.366	41.5
1.486	3.042	1.226	0.332	44.6	2.84	1.118	0.306	43.9
1.239	2.591	1.089	0.297	44.9	2.463	1.068	0.303	43.8

0.867	1.829	0.907	0.244	48.1	1.875	0.892	0.253	45
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b)

Pre-shear normal load (kPa)	% error in simulations			
	σ_1	σ_c	Cohesion	φ_e
1.858	-7.41	-5.41	1.94	-6.32
1.486	-6.64	-8.81	-7.83	-1.56
1.239	-4.94	-1.92	2.02	-2.44
0.867	2.51	-1.65	3.68	-6.44

4.3.4 Model validation by simulating 75% partially consolidated state

In order to verify if the calibrated parameter set can predict the flow behaviour in a different experiment, shear tests simulations for the partially consolidated state at 4 pre-shear normal loads were run. In shallow containers and other situations where the internal shear of a powder is limited during storage and flow, the powder may not achieve steady state flow conditions. Handling scenarios where the powder might not achieve the steady state deformation conditions may be classified as powder flow under partial consolidation or the powder to be partially consolidated (Chapter 3). The partial consolidation pre-shear shear stress was taken from experiments; it is necessary as the shearing of the sample during the pre-shear step has to be stopped at a certain fraction of the steady state shear stress as it is considered representative of the state of consolidation. The simulations were carried out for the 75% partial consolidation state where the shear stress during consolidation is 75% of the steady state shear stress value, simulations were repeated thrice for a pre-shear normal load (shear test with 4 shear points).

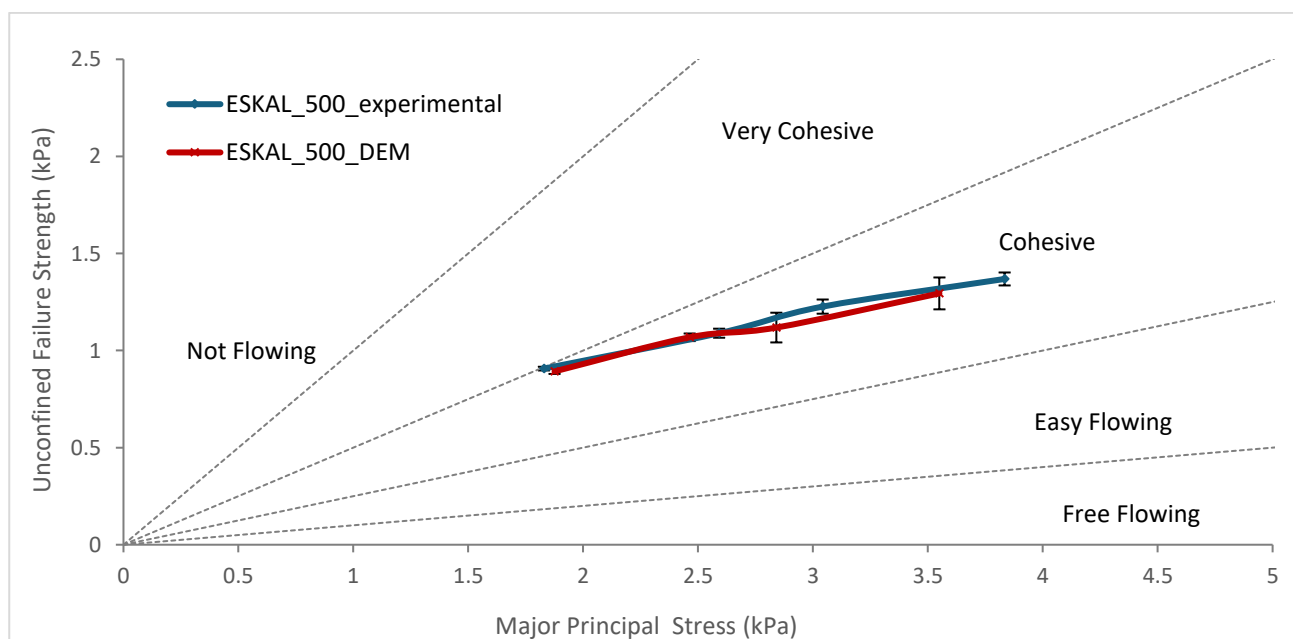


Figure 4-18: Experimental and DEM flow function of ESKAL 500.

Figure 4-19 shows the time series curve for one of the simulation runs at 1.5 kg (1.858 kPa) pre-shear normal load, the pre-shear shear stress during the pre-shearing of the sample was precisely controlled by stopping the rotation of the shear cell trough once the desired pre-shear stress value was reached. This was accomplished by using a Python script (Appendix B) which compared the shear stress value to the desired value and stopped the rotation of the trough, subsequently the trough was counter rotated to relieve the sample of the shear stress, and this counter rotation was stopped by the Python script once the shear stress value during the counter rotation became zero. The script was utilized earlier too for stopping the counter rotation of the trough once the shear stress became zero for the MELS DOE runs and for the full shear test runs.

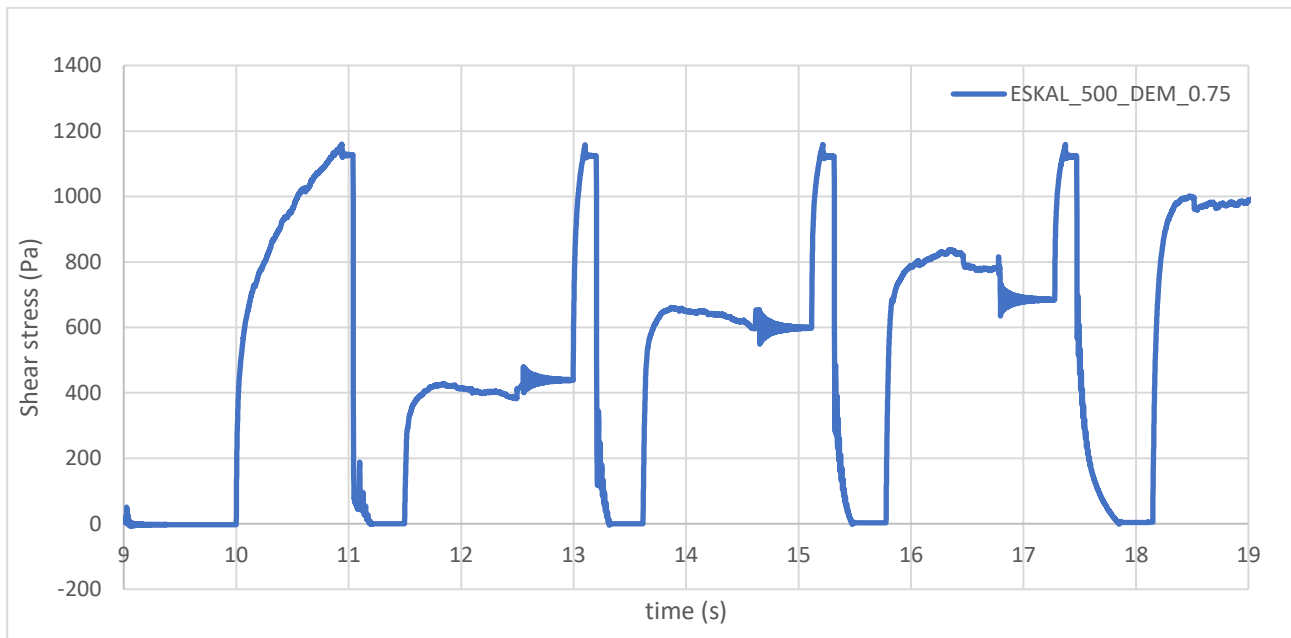


Figure 4-19: DEM shear stress time series curve for 1.5 kg (1.858 kPa) pre-shear normal load at 75% partial consolidation.

The shear stress at the failure points were compared with the experimental values, the calibrated parameter set was able to produce very good agreement with the experiments for the partially consolidated simulations at the varying pre-shear normal loads as seen in Table 4-6. Moreover, the DEM model was able to replicate the experimental phenomena, wherein the strength at incipient failure is less for the partially consolidated state when compared to the strength at steady state at the corresponding normal loads.

Table 4-6: Experimental and simulation results for 75% partial consolidation state.

		1.5 kg Experimental		1.5 kg Simulations		
Pre-shear normal stress (Pa)	Shear normal stress (Pa)	Pre-shear shear stress (Pa)	Failure shear stress (Pa)	Pre-shear shear stress (Pa)	Failure shear stress (Pa)	% error in failure shear stress

867.3	86.7	588.4	299.7	579.4	303.4	1.23
867.3	185.8	591.3	373.7	580.1	372.2	-0.41
867.3	309.7	587.4	461.1	579.5	455.4	-1.23
867.3	495.6	594.8	561.3	581.9	529.9	-5.59
		1.5 kg Experimental		1.5 kg Simulations		
Pre-shear normal stress (Pa)	Shear normal stress (Pa)	Pre-shear shear stress (Pa)	Failure shear stress (Pa)	Pre-shear shear stress (Pa)	Failure shear stress (Pa)	% error in failure shear stress
1239.1	123.9	791.2	335.1	790.9	371.3	10.81
1239.1	309.7	796.1	510.6	790.8	527.3	3.27
1239.1	557.5	807.1	669.4	790.4	658.1	-1.68
1239.1	743.4	819.1	757.4	791.6	739.8	-2.32
		1.5 kg Experimental		1.5 kg Simulations		
Pre-shear normal stress (Pa)	Shear normal stress (Pa)	Pre-shear shear stress (Pa)	Failure shear stress (Pa)	Pre-shear shear stress (Pa)	Failure shear stress (Pa)	% error in failure shear stress
1486.9	123.9	926.3	372.1	927.6	380.3	2.21
1486.9	371.7	928.4	587.7	928.2	610.5	3.87
1486.9	743.4	941.9	805.4	928.4	811.1	0.71
1486.9	929.3	949.3	886.2	928.3	892.2	0.67
		1.5 kg Experimental		1.5 kg Simulations		
Pre-shear normal stress (Pa)	Shear normal stress (Pa)	Pre-shear shear stress (Pa)	Failure shear stress (Pa)	Pre-shear shear stress (Pa)	Failure shear stress (Pa)	% error in failure shear stress
1858.6	123.9	1157.9	391.5	1159.1	400.8	2.37
1858.6	371.7	1165.1	630.2	1158.4	631.7	0.23
1858.6	743.4	1168.9	891.4	1158.4	848.1	-4.85
1858.6	1115.1	1173.9	1083.9	1158.4	1012.7	-6.56

4.4 Summary

This Chapter presented the DEM model development for the cohesive ESKAL 500 powder flow using the shear cell. Critical model parameters were identified from the parametric sensitivity study followed by design of experiments (DOE) study of the identified critical parameters. The DEM model was calibrated using a multi-objective genetic algorithm by minimizing selected objective functions. The calibrated DEM model was able to predict and match the experimental powder flow behaviour for the powders under critical and partial consolidation, although for critical consolidation, the DEM response was stiffer at lower strains, and steady state was achieved earlier than the experimental powder.

The calibrated model successfully reproduced the experimentally observed stress dependent flow behaviour and captured the differences between critical and partial consolidation states. Overall, the DEM development and calibration framework provides a robust and computationally efficient tool for predicting cohesive powder behaviour under varying consolidation states.

Chapter 5 Micro-mechanical analysis of powder flow

5.1 Upscaled particle model

The coarse-grained DEM model for modelling cohesive powder flow was developed and calibrated, as presented in Chapter 4. The micron-sized ESKAL 500 particle was upscaled and represented by a coarse-grain with each coarse-grain representing a collection of the original particles. The sensitive DEM model parameters were identified as particle-particle coefficient of static friction $\mu_{s\ p-p}$, particle-particle coefficient of rolling friction $\mu_{r\ p-p}$, plasticity ratio λ_p and constant pull-off force or adhesive strength f_o (N). These parameters were calibrated by conducting a DOE (design of experiments) study and using MOGA (Multi-Objective Genetic Algorithm) to minimize the objective functions formed as the error function between the experimental and the modelled response. The calibrated DEM model was able to reproduce the experimental powder flow behaviour at the varying states of consolidation. The total number of particles in the system were $\approx 44k$. Appropriate scaling laws can be used to further upscale and coarse-grain the particle system where the DEM model parameters are upscaled when the size of the particle is increased. Conversely, the upscaling laws can also be applied to reduce the size of the coarse-grained particle to increase the total particles in the system for achieving higher fidelity.

The linear version of the EEPA (Edinburgh Elasto-Plastic Adhesive) model was used to develop the coarse-grained cohesive DEM model, the powder is modelled through 4 basic parameters of the EEPA model which are k_1 (virgin loading stiffness), k_2 (unloading/reloading stiffness), k_{adh} (adhesive branch stiffness) and f_o . The virgin loading branch, the unloading/reloading branch and the adhesive branch of the EEPA model are governed by these parameters. The particle-particle interactions during the initial loading, unloading and final separation are governed by these branches. Thakur et al. (2016) and Janda and Ooi (2016) studied the parametric scaling laws for the linear EEPA model for simulations of the uniaxial and cone penetration tests respectively. Janda and Ooi (2016) demonstrated that the upscaled system showed good match with the original system when the stiffness parameters (k_1 , k_2 , k_{adh}) and the constant pull-off force f_o were scaled linearly and quadratically with the particle size respectively. These scaling laws were used for scaling the parameters of the EEPA model in this present study, to increase the total number of particles in the coarse-grained DEM model to obtain a higher fidelity model to study the particle level phenomena at the mesoscopic scale. The subscript “o” presents the original coarse-grained (CG) upscaled particle used in the system having 44k particles, the CG_o particle was a dual sphere particle with an aspect ratio of 1.2 comprising of two overlapping spheres of 1.4 mm diameter each. The scaled CG particle represented as CG_s preserved the shape of the original CG particle with the same aspect-ratio of 1.2 but was composed of two overlapping spheres of 0.65 mm diameter each. The total particles in the scaled system thus increased to around 500,000.

The virgin loading stiffness k_1 depends on the Young's modulus and the radius of the particles, and is determined as $k_1 = 2E^*R^*$, where E^* and R^* are the equivalent Young's modulus and equivalent radius

determined as $\frac{1}{E^*} = \frac{(1-\nu_i^2)}{E_i} + \frac{(1-\nu_j^2)}{E_j}$ and $R^* = \frac{(R_i \times R_j)}{(R_i + R_j)}$ respectively. E_i , E_j ; ν_i , ν_j ; and R_i , R_j are the Young's modulus, Poisson's ratio and radii respectively of the particles i and j in contact. The stiffness of the reloading branch k_2 is dependent on the plasticity ratio λ_p and the virgin loading stiffness k_1 . The plasticity ratio, $\lambda_p = 1 - \frac{k_1}{k_2} = \frac{\delta_p}{\delta}$ represents the contact plasticity, where δ_p is the maximum plastic deformation and δ is the total deformation at the contact. The equivalent Young's modulus E^* for the original and scaled system is considered to be similar, as Young's modulus, which is a particle property, should not be dependent on the particle size. The stiffness k_1 and k_2 for the scaled system thus scales linearly with R^* or particle size. As previously discussed, the constant pull-off force f_o scales quadratically with the particle size, determination of k_1 and k_2 along with k_{adh} for the scaled system helps to model the particle interactions.

The relation between the hysteretic spring normal force f_{hys} and the normal overlap is expressed mathematically by Equations in (5-1).

$$f_{hys} = \begin{cases} f_o + k_1\delta & \text{if } k_2(\delta - \delta_p) \geq k_1\delta \\ f_o + k_2(\delta - \delta_p) & \text{if } k_1\delta > k_2(\delta - \delta_p) > -k_{adh}\delta \\ f_o - k_{adh}\delta & \text{if } -k_{adh}\delta \geq k_2(\delta - \delta_p) \end{cases} \quad (5-1)$$

The total contact normal force f_n is the sum of the hysteretic spring force f_{hys} and the normal damping force f_d^n and is given by Equation (5-2).

$$f_n = (f_{hys} + f_d^n)\mathbf{n} \quad (5-2)$$

where $\mathbf{n}$ is the unit normal vector pointing from the contact point to the centre of the particle.

Altair EDEM 2023 which was used for the DEM simulations in this study lets the user input the values of f_o , E , ν and $\Delta\gamma$ (contact adhesion energy). The particle shape, size, and the particle size distribution can also be defined. EDEM thus calculates k_1 and k_2 from these parameters, with f_o being input directly, it only leaves the question of defining k_{adh} for the scaled system. If the unloading of the particles along the k_2 branch (Figure 2-8) continues below δ_p , it leads to the development of an adhesive force until it reaches the maximum adhesive force f_{min} (Equation (5-3)). If unloading continues further, it leads to a reduction in the adhesive force and the overlap until the particles are separated ($\delta = 0$). The particle overlap at f_{min} can be defined as δ_{min} and can be used to determine k_{adh} according to Equation (5-4).

$$f_{min} = f_o - \frac{3}{2}\pi a_c \Delta\gamma \quad (5-3)$$

$$f_o - f_{min} = \frac{3}{2}\pi a_c \Delta\gamma = k_{adh}\delta_{min} \quad (5-4)$$

$$a_c = \frac{1}{2d} \sqrt{4d^2 R_i^2 - (d^2 - R_j^2 + R_i^2)^2} \quad (5-5)$$

$$d = \begin{cases} (R_i + R_j) - \delta & \text{if } k_1 \text{ branch} \\ (R_i + R_j) - \delta_p & \text{if } k_2 \text{ or } k_{adh} \text{ branch} \end{cases} \quad (5-6)$$

where a_c (5-5) is the contact patch radius (Morrissey, 2013), d is the distance between the centres of the particles with radii R_i and R_j determined from Equation (5-6). For the plastic contact, the patch radius a_c is determined from Equation (5-9) by considering the two particles in contact to have equal radii.

$$\text{If } R_i = R_j, a_c = \sqrt{R_i^2 - \frac{d^2}{4}}, d = 2R_i - \delta_p \quad (5-7)$$

$$a_c = \sqrt{R_i^2 - \frac{(4R_i^2 + \delta_p^2 - 4R_i\delta_p)}{4}} \quad (5-8)$$

$$a_c = \sqrt{\delta_p \left(R_i - \frac{\delta_p}{4} \right)} \quad (5-9)$$

The average normal overlap during the first steady state shearing (Chapter 4) of the sample was used to estimate the average plastic normal overlap δ_p from the relation $\lambda_p = \frac{\delta_p}{\delta}$. The value of $f_o - f_{min}$ can thus be determined and utilized to determine δ_{min} (Equation (5-10)) which is the particle overlap at f_{min} .

$$\delta_{min} = \frac{k_2\delta_p - (f_o - f_{min})}{k_2} \quad (5-10)$$

The adhesive branch stiffness can thus be determined from Equation (5-11).

$$k_{adh} = \frac{f_o - f_{min}}{\delta_{min}} = \frac{\frac{3}{2}\pi a_c \Delta\gamma}{\delta_{min}} \quad (5-11)$$

Coarse-graining ratio l can be defined as the ratio of the original to scaled particle, assuming the particle overlap scales linearly with particle size (Hu et al., 2022), contact patch radius a_c then also scales linearly with the particle size, that results in adhesive stiffness $k_{adh,o}$ to also scale linearly such that $k_{adh,s} = k_{adh,o}l$. The scaled contact adhesion energy $\Delta\gamma_s$ would thus also scale linearly with the particle radius, $\Delta\gamma_s = \Delta\gamma l$ in order for the adhesive branch stiffness to scale linearly.

The scaled values of the adhesion energy (scaled linearly) and the pull-off force (scaled quadratically) for the smaller size meso-particle can now be used to model the high fidelity shear cell model as the four basic EEPA model parameters of k_1 , k_2 , k_{adh} and f_o have been scaled. The resulting upscaled model had $\approx 493k$ particles and will be referred to as the upscaled model in this Chapter, the calibrated model with $\approx 44k$ particles will be referred to as the original model.

The efficacy of the upscaling process was checked by comparing the shear stress at the steady state and the failure strength at the shear to failure points for the upscaled model with the original model. The parameter upscaling technique was found to be effective in reproducing the behaviour of the original model as there

was good agreement between the shear stress at steady state and failure points for the two models as seen in Figure 5-1. Therefore, to study the micromechanical particle phenomena at the mesoscopic scale, the upscaled model can be used. Shear strain in Figure 5-1 is determined similarly as described previously in Figure 4-17.

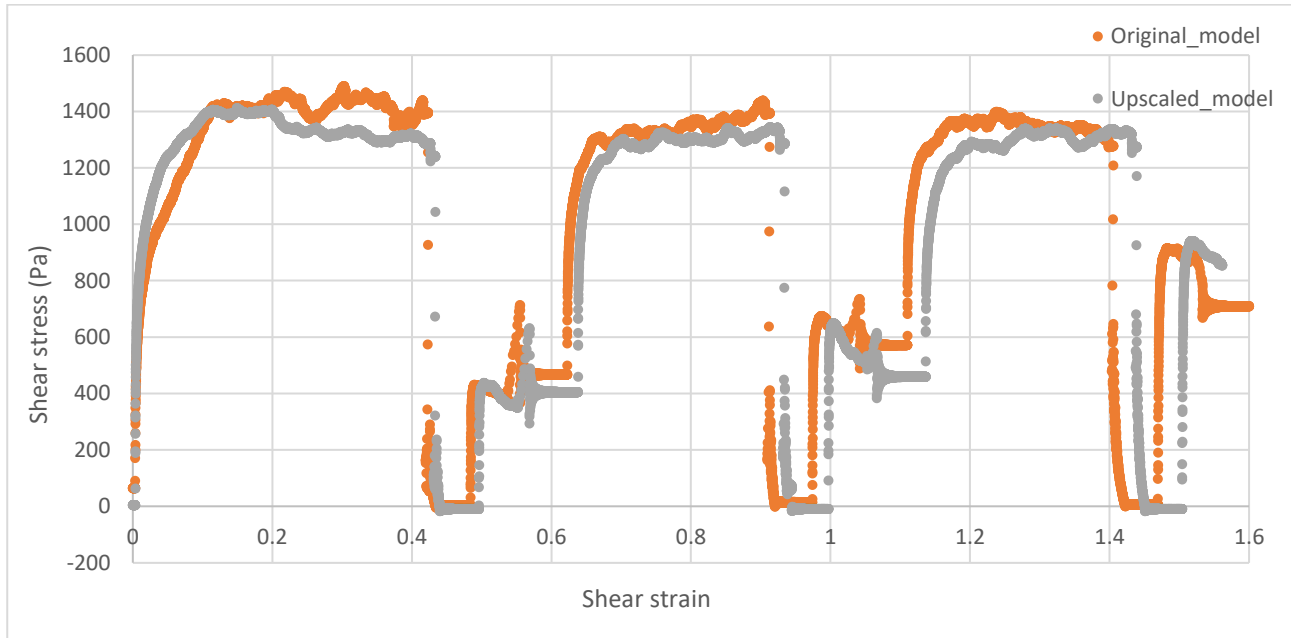


Figure 5-1: Shear stress evolution with shear strain for the original and upscaled model.

5.2 Investigation of variable failure strength at partial and critical consolidation

The failure strength of partially consolidated powders was lower than the critically consolidated powders across varying normal stress, as seen from the yield loci at varying consolidation states presented and discussed in Chapter 3. The reason for this observed phenomenon was hypothesized to be a lower number of particle contacts or coordination number due to lesser bulk density at the partial consolidation states. The upscaled model has been used to investigate this hypothesis along with the investigation of particle overlap and fabric anisotropy as the possible reasons behind the variable failure strength at partial and critical consolidation.

The failure strength at the first shear point (0.1 kg normal load) after the critical and partial consolidation for the upscaled model at 1.5 kg (1.858 kPa) normal load was studied. In the case studied (Figure 5-2), these were 464 Pa and 415 Pa for the critical and partial consolidation states respectively. To avoid any possible differences arising due to different powder packing structure and porosity, the powder bed used for the critical and partial consolidation was the same. The critically consolidated sample was sheared till steady state, the 75% partial consolidation state was achieved by shearing the sample to a shear stress value of 75% of the steady state shear stress. The shear stress time series curves for the partial and critical consolidation are shown in Figure 5-2.

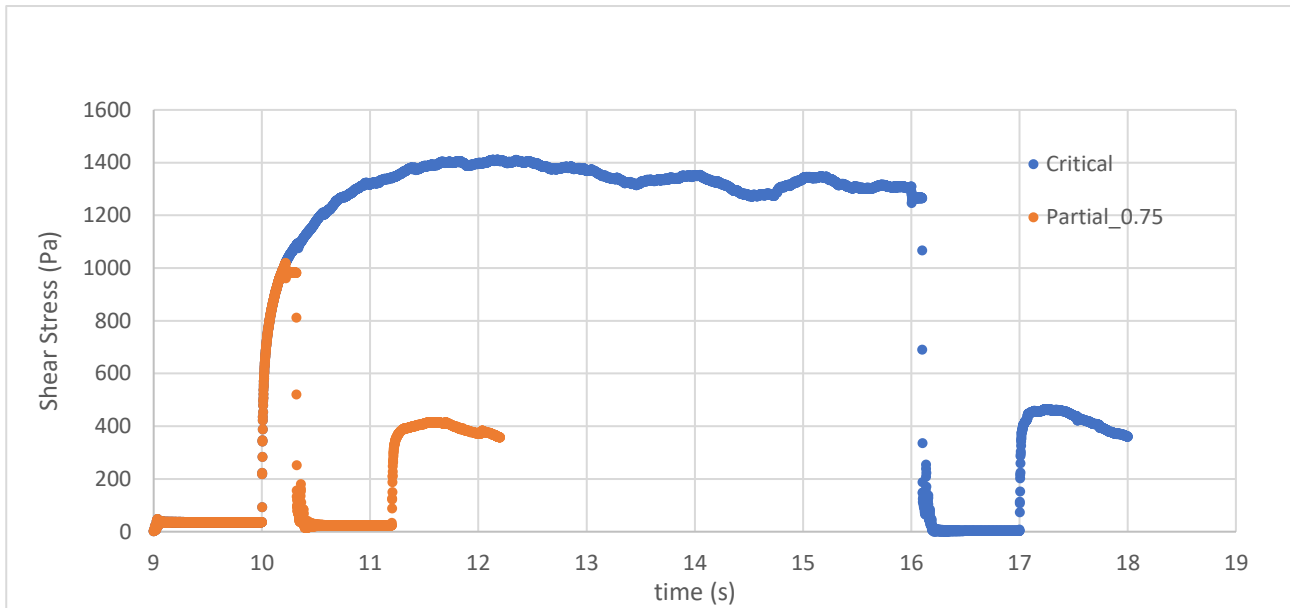


Figure 5-2: Shear stress time series curves for the upscaled model at partial and critical consolidation.

As the shearing of the sample takes place, shear zone localisation beneath the shearing lid vanes takes place (Schulze, 2008). Considering the powder layers along the depth of the shear cell, due to gravity, the normal stress applied to the powder layers increases as we approach the bottom of the shear cell. Therefore, near the top of the shear cell, the normal stress subjected to the powder layers is smaller, which results in a marginally smaller shear stress required for shearing than that required for the layers near the bottom. This causes the shear zone to be located near the top of the shear cell beneath the shearing lid vanes. The rotation of the shear cell takes place from the bottom through a motor, causing a difference in velocity of the particle layers as we go towards the top of the shear cell or near the shearing lid. The velocity of the particles near the bottom of the shear cell is close to the rotational velocity of the shear cell.

To study the effect of the parameters suspected to be impacting the failure strength, zones or bins spread across the depth of the small (S) shear cell of the Schulze Ring Shear Tester (RST) as seen in Figure 5-3 were used. The bins were shaped as annular quadrants with a radial width of 10 mm and a depth of 4 mm, the radial width of 10 mm allowed a clearance of 10 mm each from the inner and outer walls of the shear cell, thus avoiding any possible boundary effects. The bins were linked to the position of the shearing lid, such that the top-most bin, i.e., quadrant 1 was located directly below the shearing lid vanes. This was done to ensure that as the shearing lid progresses into the shear cell with pre-shearing, quadrant 1 remains exactly below the shearing lid vanes. As the shear zone is expected to be located below the shearing lid vanes, it is desirable that the lid vanes do not intrude into the bin, because intrusion by the vanes into the bin can cause reduction in the bin volume, and as a result reduction in the number of particles recorded in the actual shear zone. This can lead to the properties of the shear zone to be not presented correctly. At the start of pre-shear at 10 s, the shearing lid has progressed approximately 1.28 mm into the shear cell trough. This results in the shearing

lid vanes to be approximately 5.28 mm below the top surface of the shear cell. The top surface of the shear cell and the powder surface before any load imposition is considered to be the reference level or at 0 mm vertical level. This places the top-most bin, i.e., quadrant 1 located approximately 5.28 mm below the top surface of the shear cell and directly below the shearing lid vanes (shearing lid vanes height = 4 mm) to capture the properties near the shear zone. This places its centroid to be approximately 7.28 mm below the powder surface or top surface of the shear cell. The total depth of the small (S) shear cell (Figure 4-1) used for the simulations is 24 mm. Quadrants 2, 3 & 4 were also linked to the shearing lid, such that quadrant bin 2 was placed directly below quadrant 1, similarly quadrant 3 was directly below quadrant 2, and quadrant 4 was directly below quadrant 3. The centroid of bottom-most bin, i.e., quadrant 4, as a result, was located approximately 19.28 mm below the powder surface in the shear cell and 2.72 mm above the grooves near the bottom. Each of the annular quadrant bins 1-4 accommodated approximately 8000 particles.

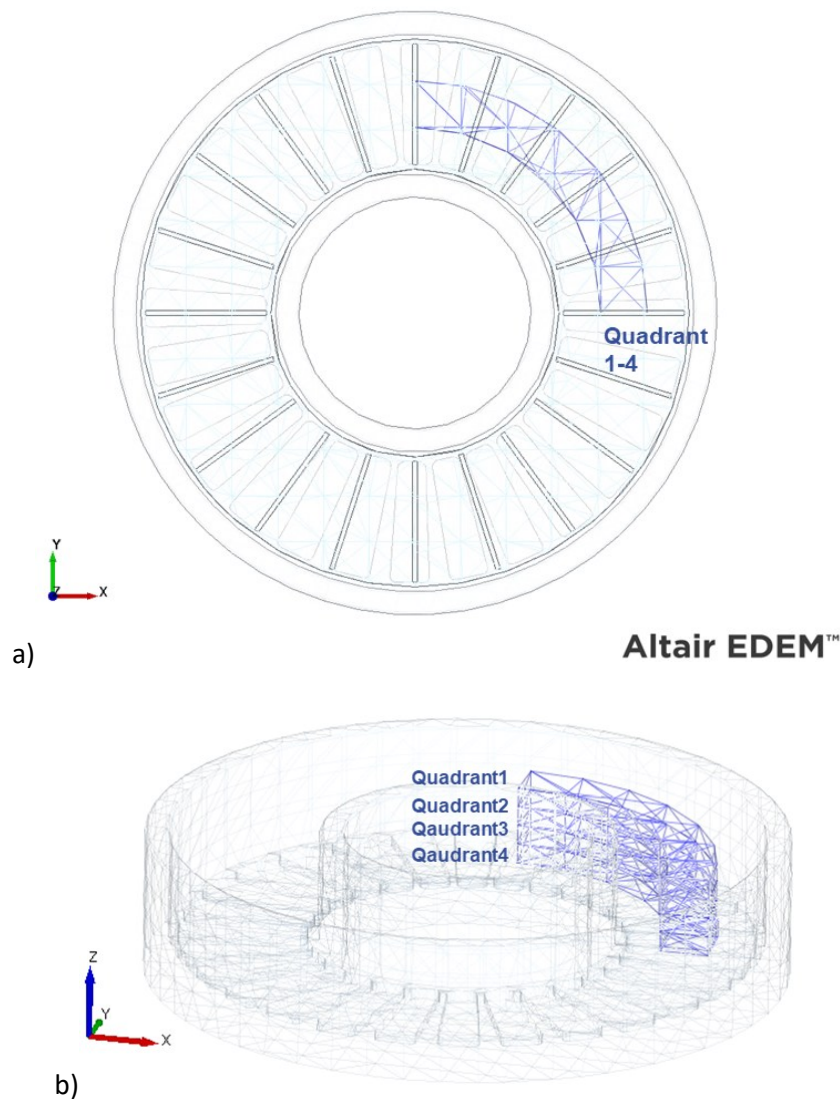
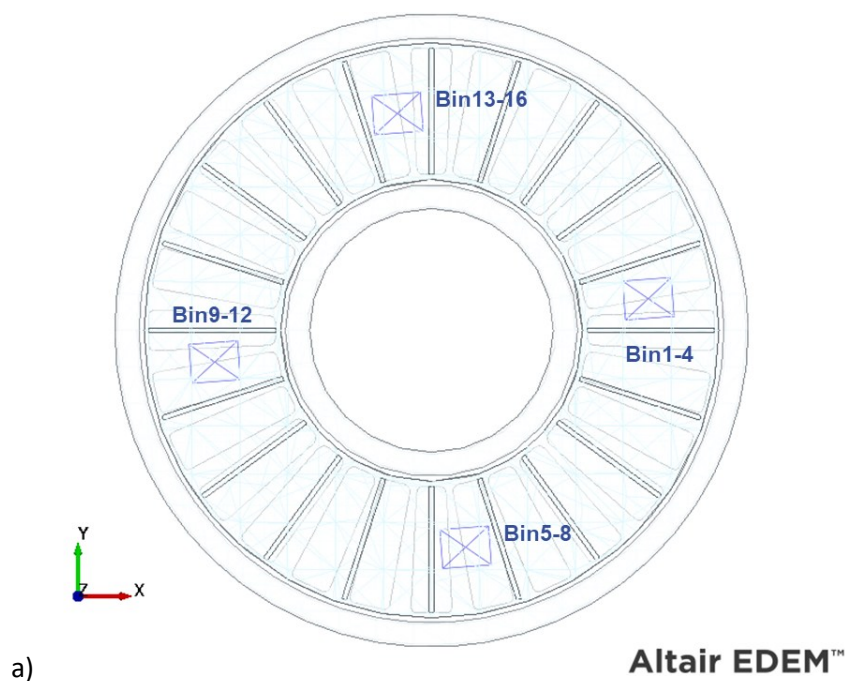


Figure 5-3: Annular quadrant bins across the depth of the shear cell, a) top-view, and b) isometric view.

A secondary study with a set of smaller bins was also conducted to investigate the size of the bins and their radial location on the particle properties suspected to be impacting the failure strength. The zones or bins spread radially and across the depth of the shear cell as seen in Figure 5-4 were used. The bin depth was 4 mm, with the dimensions in x and y directions being 10 mm and 8 mm respectively. Similar to the quadrant bins, the rectangular bins were also linked to the shearing lid's position. At the start of pre-shear at 10 s, the centroid of top-most bins, i.e., bins 1, 5, 9 & 13 were located approximately 7.28 mm below the powder surface or top surface of the shear cell and directly below the shearing lid vanes (shearing lid vanes height = 4 mm) to capture the properties near the shear zone. The centroid of the bottom-most bins, i.e., bins 4, 8, 12 & 16 were located approximately 19.28 mm below the top surface of the shear cell and 2.72 mm above the grooves near the bottom. The Figures in this Chapter which present particle properties with bin location from the powder surface, use the notations for quadrant and rectangular bins 1, 2, 3, & 4 as 7 mm, 11 mm, 15 mm, & 19 mm below the powder surface. The actual centroid locations of these bins at the start of shearing at 10 s is 0.28 mm further than these notation values, further the bin centroid locations change because the bins are linked with the shearing lid position. So as the shearing lid progresses further into the shear cell during pre-shear or when it rises up due to the sample dilation during incipient shearing, the bin centroid locations also change. Therefore, in these Figures, the notation of the bin centroid location from powder surface is to represent the relative locations of these bins w.r.t top of the shear cell and not the exact bin centroid location.



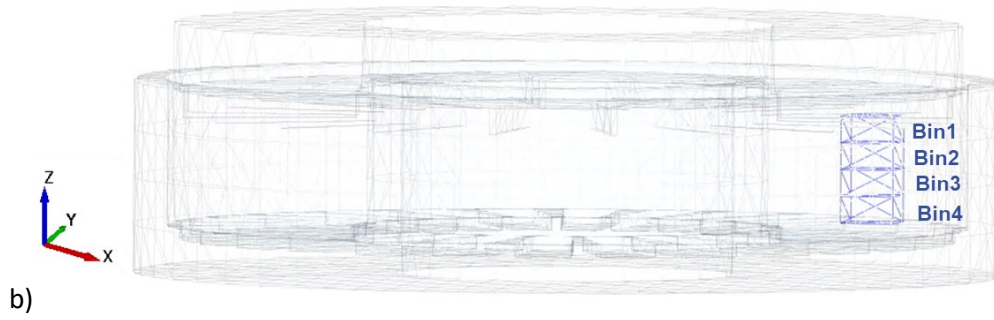


Figure 5-4: Bin locations spread out radially and across the depth of the shear cell, a) top-view, and b) isometric view.

Each of the bins 1-16 accommodated approximately 900 particles. For calculating the velocity of the particles, the smaller bins 1-16 were used as the velocity of the particles can vary with the radial location, and it is appropriate to compute it in a smaller localized zone as the average velocity components (x and y) would also be used to compute the shear strain in the sample. The velocity of all the particles in the bin was averaged. Considering bins 1-4, at the start of pre-shear at 10 s, their centroids were located approximately at 7.28 mm, 11.28 mm, 15.28 mm and 19.28 mm in the vertical (z) direction from the top of the shear cell (powder surface), the bins 5-8, 9-12 and 13-16 were located at similar vertical locations like their corresponding counterparts in bins 1-4. Figure 5-5 shows the velocity variation across the bins 1-4, the velocity for bin 1 is significantly smaller than the other bin velocities. The shearing of the sample starts at 10 s and continues till 16 s, as the shearing progresses, the velocities for bins 3 & 4 become nearly equal to the trough velocity with the velocity for bin 2 approaching this value. Curves similar to Figure 5-5 for the bins 5-8, 9-12 & 13-16 were obtained and therefore are not presented here.

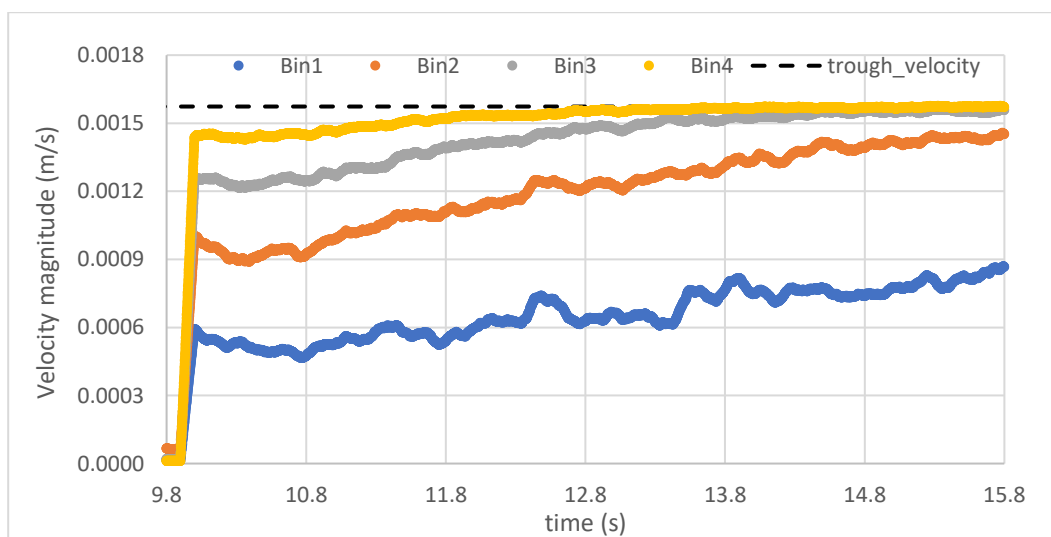


Figure 5-5: Velocity magnitude for bins 1-4.

Figure 5-6 shows the velocity variation across the depth of the powder bed for two time instances during the pre-shearing process, one near the start at 11 s and other at 15 s once the steady state has been established. As can be seen at 11 s, the velocity variation across the depth is approximately linear, but once significant shearing has taken place and particles have rearranged, the velocities for the bottom 3 bins, i.e., bins 2, 3 & 4 approaches the shear cell trough velocity, with the velocity of the bin 1 significantly lower than the other bins. For the powder to be sheared, there should be a velocity difference or relative velocity of sliding between the powder layers, at the steady state (15 s), the relative velocity between bins 1 & 2 is almost 5 times larger than the relative velocity between bins 2 & 3. This indicates that the shearing zone is located near bin 1 which is just beneath the shearing lid vanes.

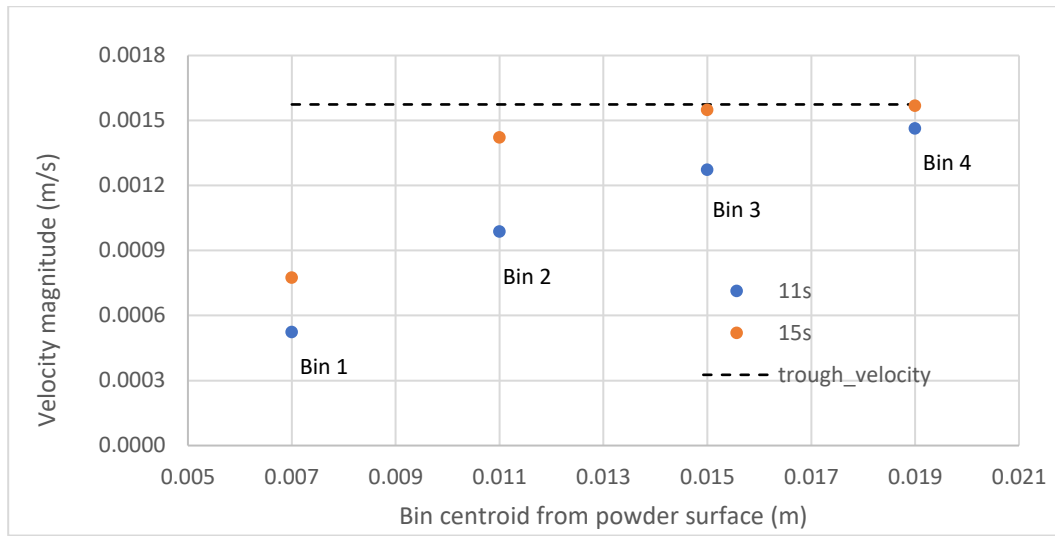


Figure 5-6: Velocity magnitudes for bins 1-4 at the start and end of shearing.

5.2.1 Coordination number

The coordination number Z for the bin can be determined as $Z = 2N_c/N_p$, where N_c are the total particle contacts and N_p represents the total number of particles in the bin. The coordination number is presented for the critically and partially consolidated states for the quadrant (Figure 5-7) and rectangular bins (Figure 5-8). Shearing for both the cases starts at 10 s, it goes on till 16 s for the critically consolidated sample and till 10.218 s for the partial consolidation case. For the critically consolidated sample, to relieve the sample from the shear stress before the shear to failure step, the sample is counter rotated from 16.1-16.21 s with the lowering of the normal stress also taking place at 16.1 s. Subsequently, the sample is sheared to failure from 17-18 s. For the partially consolidated powder, the shear cell is counter rotated from 10.318-10.403 s with the normal stress also lowered at 10.318 s, the sample is then sheared to failure from 11.2-12.2 s.

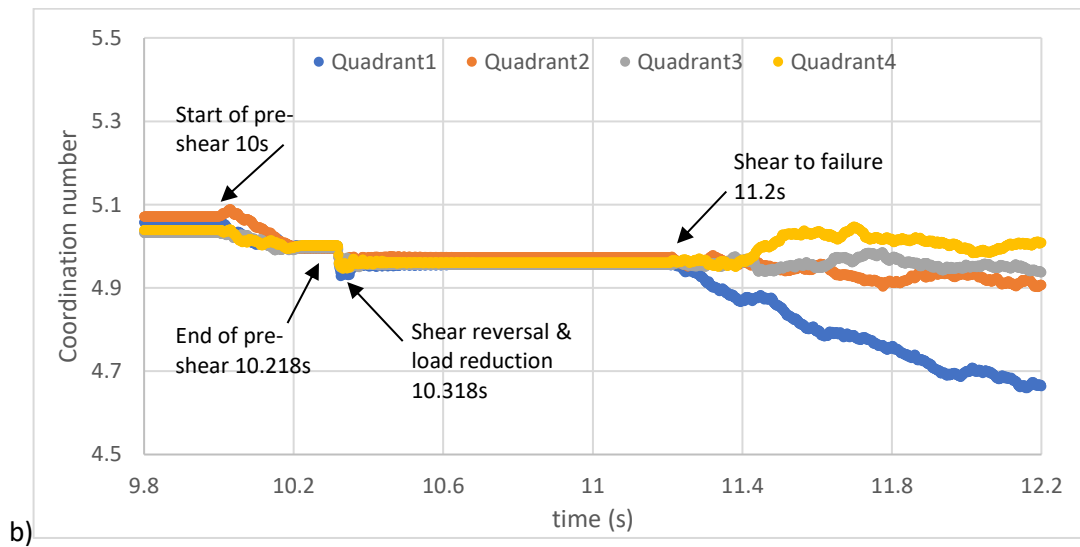
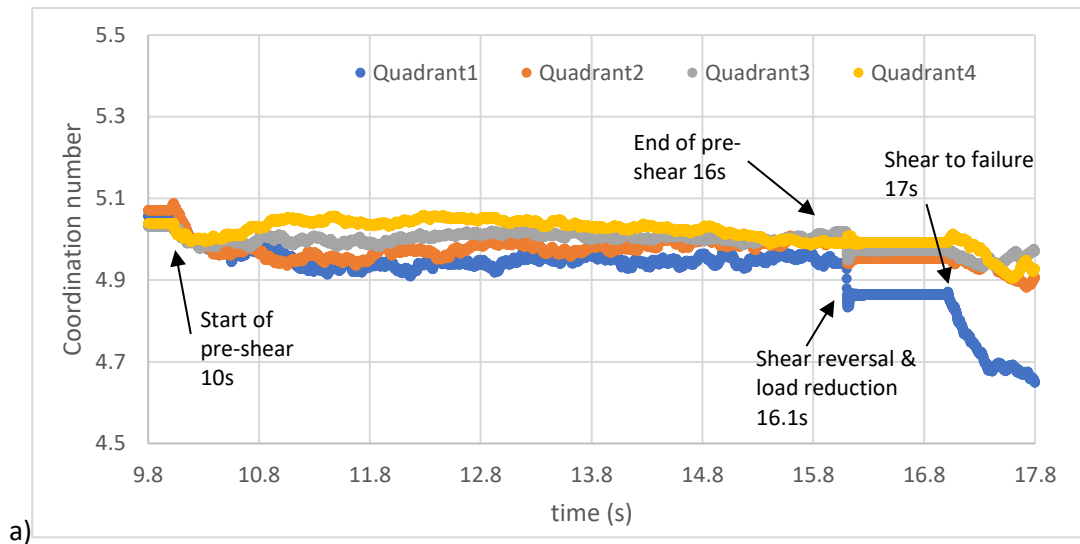
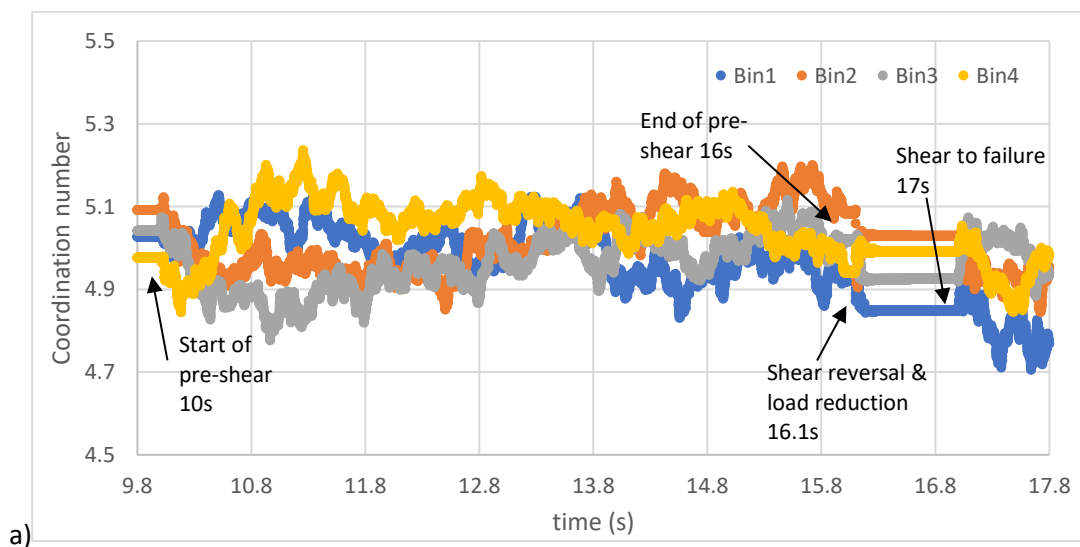


Figure 5-7: Coordination number evolution with shearing for a) critical consolidation, and b) partial consolidation, annular quadrant bins.



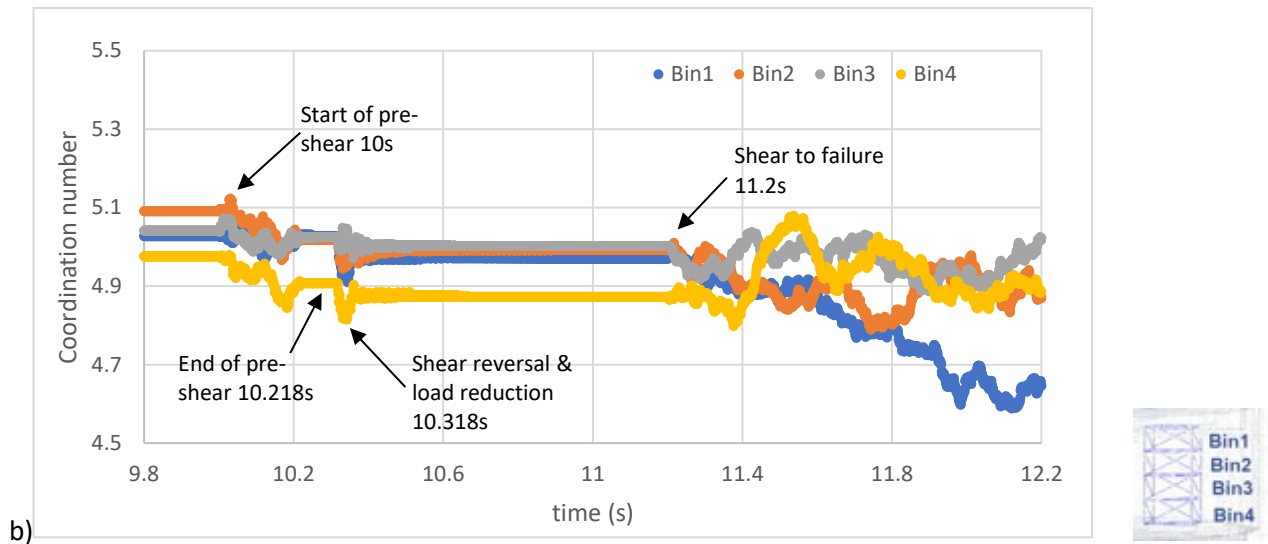


Figure 5-8: Coordination number evolution with shearing for a) critical consolidation, and b) partial consolidation, rectangular bins.

The coordination number Z for the quadrant bins 1-4 and rectangular bins 1-4 for both partial and critical consolidation remains near 5 when the sample is being sheared. There is a slight drop in Z when the pre-shearing starts at 10 s which is likely due to the particle rearrangement caused due to shearing, the coordination number fluctuates during continued shearing but remains around a value of 5. There is significant fluctuation in Z for the rectangular bins with relatively smoother profiles seen for the quadrant bins, this can be explained by the significantly larger number of particles in the quadrant bins. At the initiation of failure at 17 s for the critically consolidated case and 11.2 s for the partial consolidation case, Z is marginally lower for the former at 4.86 than the latter's 4.95 for quadrant 1. The comparatively lower Z for critical consolidation is caused by the rearrangement of particles which takes place due to shearing, Z stabilizes once the particle contacts are established/stabilized once the sample reaches critical shear. The shearing for the partial consolidation case is stopped before the particles rearrange fully and contact stabilization which causes a higher Z value. A higher Z would ideally lead to more cohesive forces developed between the particles and an increase in the cohesive failure strength of the sample, but as Z for the partial consolidation state is higher than the critical state but its failure strength is lower than the critical state, the coordination number does not seem to impact the failure strength at the varying consolidation states.

For both the critical and partial consolidation cases, there is a significant drop in Z for the shear to failure step initiated at 17 s and 11.2 s respectively for the topmost quadrant bin 1 and rectangular bin 1. For quadrant and rectangular bins 2-4, the drop in Z is not as significant as bin 1. This indicates that maximum particle rearrangement takes place in bin 1 pointing to the location of the shear zone in bin 1. The whole of the granular shear cell assembly experiences dilation upon the failure of the over consolidated sample at 14.5 s and 11.2 s for the two cases, but significant dilation is seen only in quadrant bin 1 and rectangular bin 1 as

seen in Figure 5-9 and Figure 5-10, again pointing to the fact that the shear zone is located in top-most bin 1, as the shear zone sees maximized dilation during failure of an over consolidated sample.

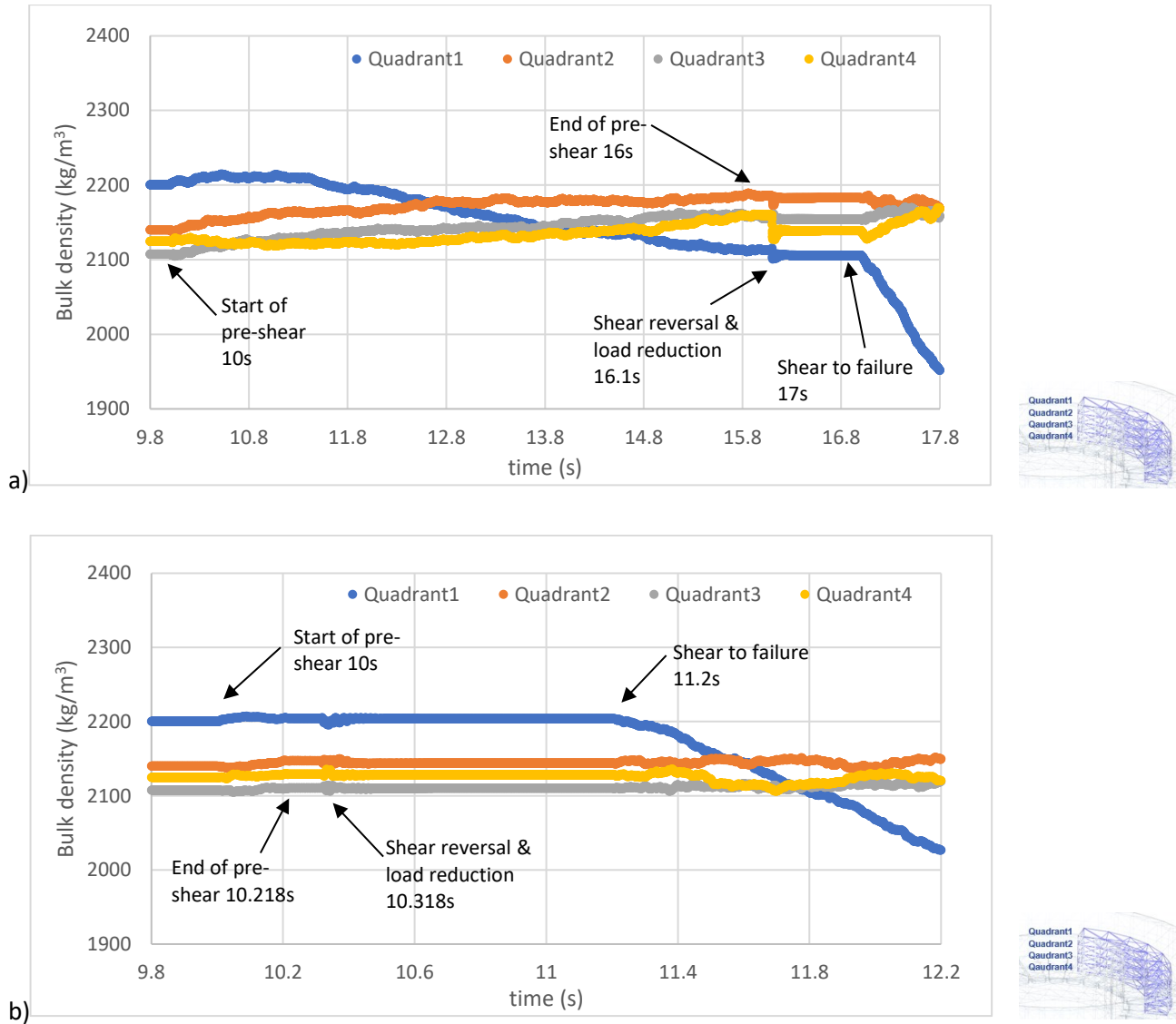


Figure 5-9: Bulk density evolution with shearing for a) critical consolidation, and b) partial consolidation, annular quadrant bins.

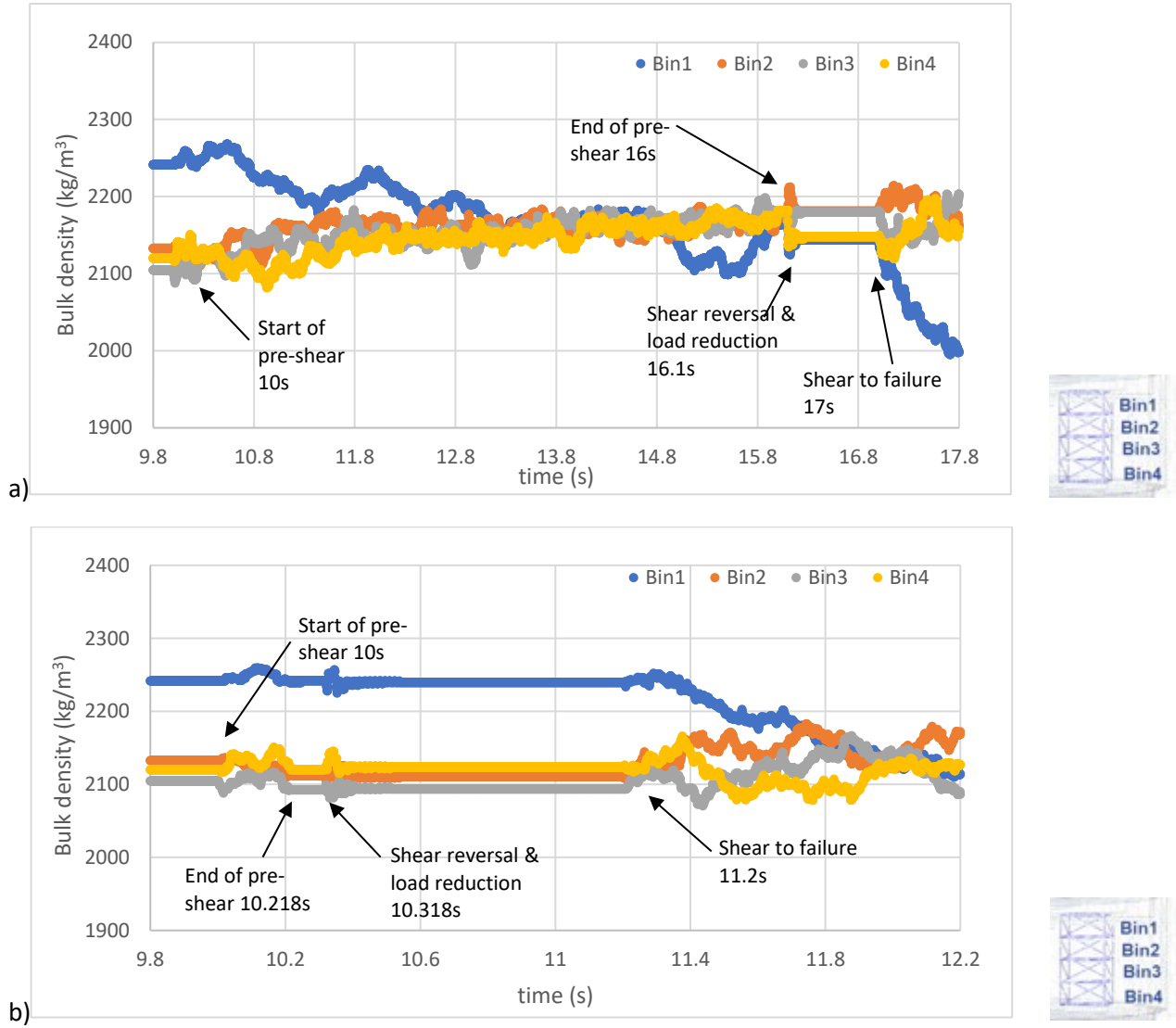


Figure 5-10: Bulk density evolution with shearing for a) critical consolidation, and b) partial consolidation, rectangular bins.

5.2.2 Particle normal overlap

Higher contact normal overlap leads to higher contact normal force F_n . The tangential force F_t is limited by the Coulombic friction limit, such that $F_t \leq \mu F_n$, where μ is the friction coefficient. Upon shearing of the over consolidated sample, the particles begin to slide against each other once this Coulombic friction limit is reached. Thus, higher particle normal overlap could cause a higher tangential force causing the failure strength for the critical state to be larger than the partial consolidation failure strength.

The total particle overlap in the normal direction for particles in a particular bin was determined by adding the normal contact overlap for all the particle contacts in the bin. The average particle overlap was subsequently determined as the ratio of the total particle overlap to the total number of particle contacts in the bin. The average normal overlap was determined for the quadrant bins 1-4 (Figure 5-11) and the rectangular bins 1-4 (Figure 5-12) for the critical and partially consolidated states. The average normal overlap

decreases for all the quadrant bins 1-4 (Figure 5-11) once pre-shearing is initiated at 10 s and continues to decrease till the sample is critically consolidated. The decrease in the normal overlap with shearing and eventual stabilization can be caused by particle rearrangement as the coordination number also follows a similar trend upon shearing (Figure 5-7). As a result, the normal overlap before initiation of shear to failure is greater for the partially consolidated state than for the critical consolidation state, for the top-most quadrant bin 1 it is 1.45×10^{-6} m for partial consolidation and 1.39×10^{-6} m for critical consolidation. This finding suggests that the higher failure strength of the critically consolidated sample cannot be explained solely by the normal overlap, as the failure strength of the partially consolidated sample is lesser despite it having a larger average particle normal overlap.

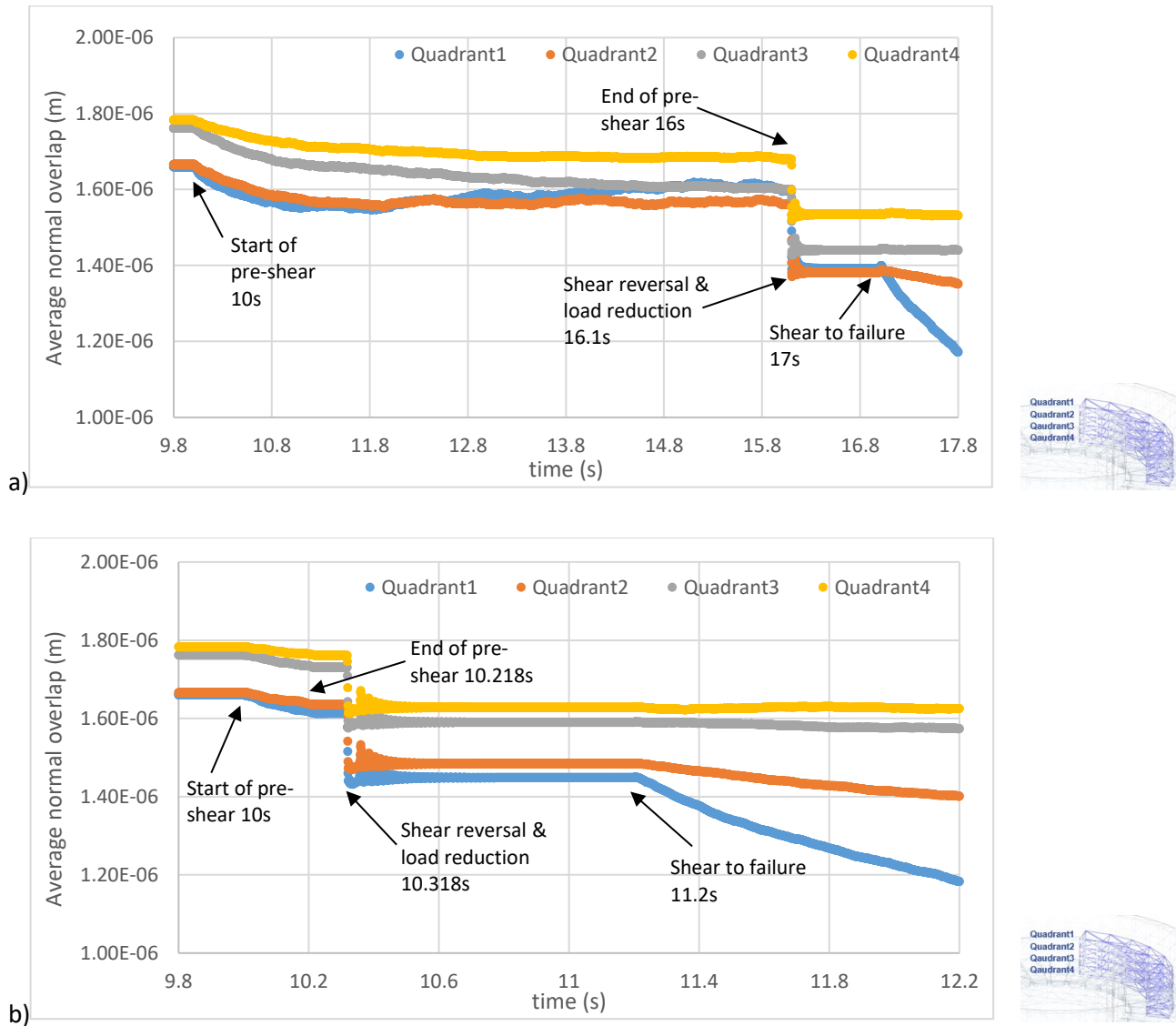


Figure 5-11: Average normal particle overlap evolution with shearing for a) critical consolidation, and b) partial consolidation, annular quadrant bins.

A secondary study with the rectangular bins was conducted to study any localized effects in the shear cell impacting the average normal overlap. As seen from Figure 5-12, the normal overlap increased for the

topmost rectangular bin 1 and decreased for rectangular bins 2-4 upon initiation of pre-shearing at 10 s, before stabilizing to a constant value at critical consolidation. The normal overlap at the initiation of shear to failure for top-most bin 1 at 17 s for critical consolidation (1.46×10^{-6} m) was higher than the corresponding value at 11.2 s for partial consolidation (1.27×10^{-6} m). For both samples, post initiation of the shear to failure step at 17 s & 11.2 s, the particle normal overlap decreases for the top-most bin 1 due to particle rearrangement and dilation.

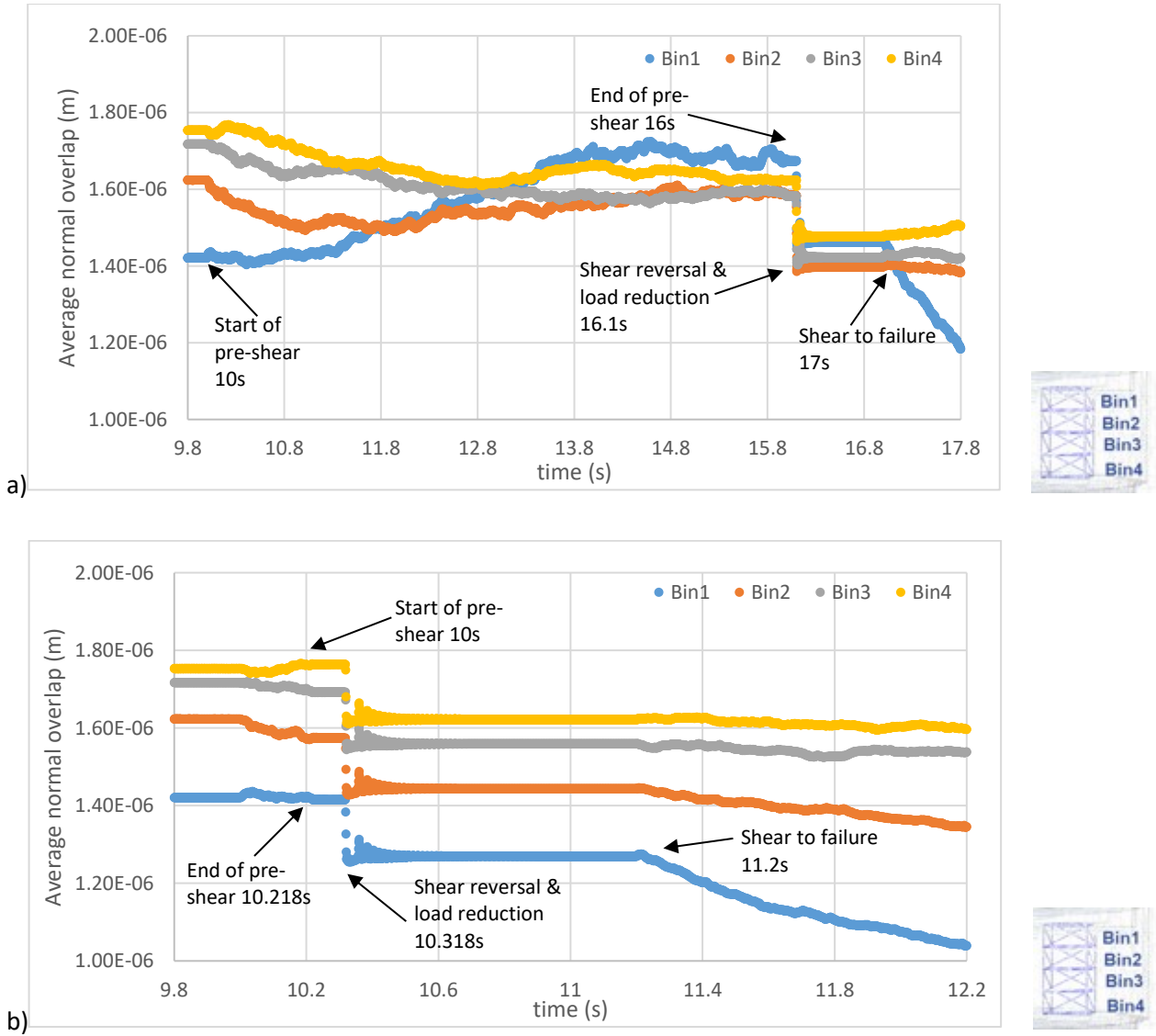


Figure 5-12: Average normal particle overlap evolution with shearing for a) critical consolidation, and b) partial consolidation, rectangular bins.

As the shear zone seems to be located in the top-most bin which is just below the shearing lid vanes, the average particle overlap in the normal direction was computed for the top-most bins 1, 5, 9 & 13 located radially across the shear cell. The contact normal overlap determined for particle contacts in the top-most rectangular bins 1, 5, 9 and 13 during consolidation and incipient failure is shown in Figure 5-13. The particle normal overlap at the end of shearing and at the start of shear to failure step for the critical consolidation

case was higher than the corresponding values for the partial consolidation case. Thus, higher particle normal overlap could cause a higher tangential force causing the failure strength for the critical state to be larger than the partial consolidation failure strength. For both samples, post initiation of the shear to failure step at 17 s & 11.2 s, the particle normal overlap decreases for all the top-most bins due to particle rearrangement and dilation.

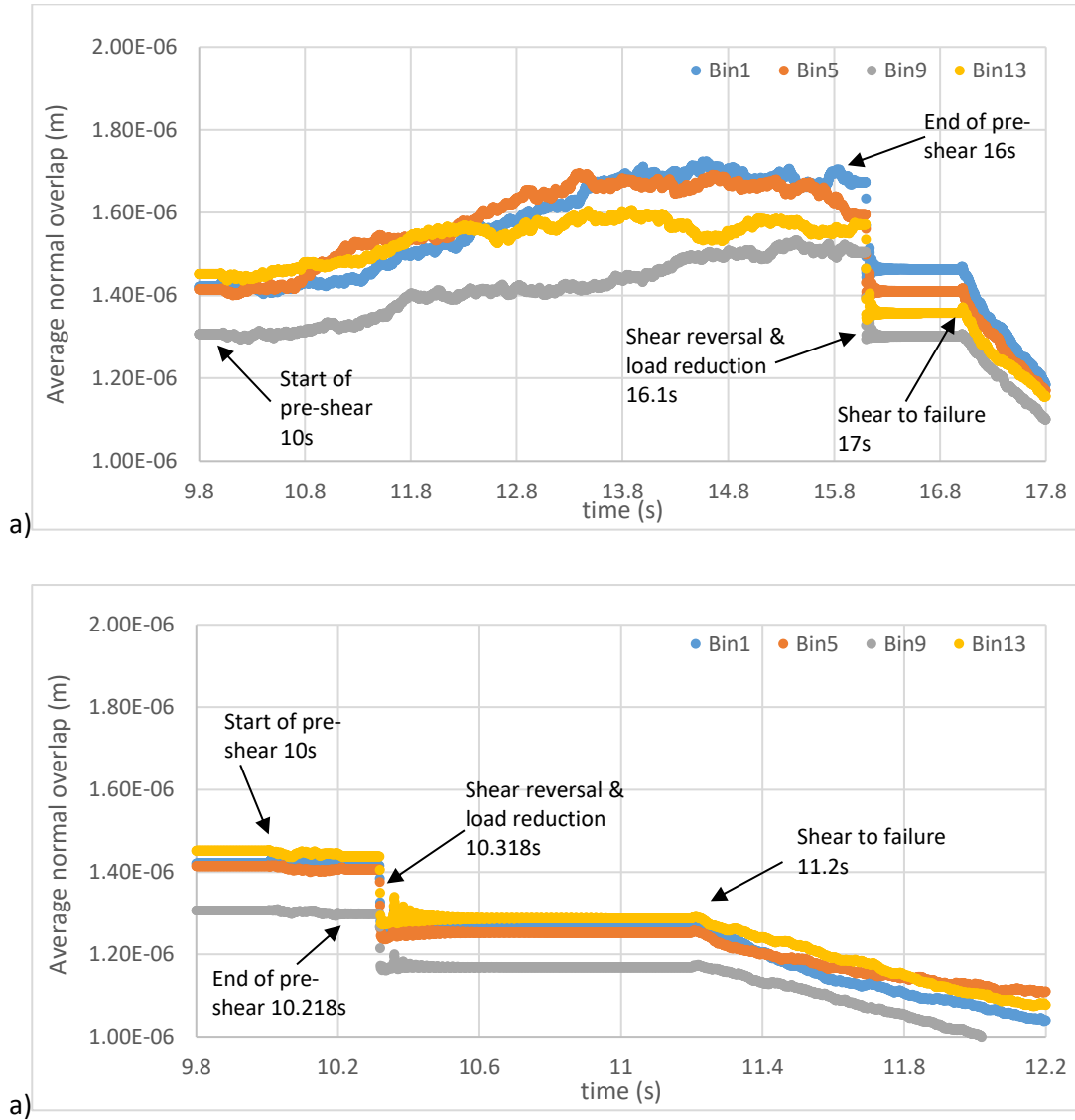


Figure 5-13: Average normal particle overlap evolution with shearing for a) critical consolidation, and b) partial consolidation, rectangular bins 1, 5, 9 and 13.

The normal overlap for the top-most annular quadrant bin 1 (Figure 5-11) and rectangular bin 1 (Figure 5-12) show opposite behaviour upon initiation of pre-shearing at 10 s, with the particle normal overlap decreasing for the former and increasing for the latter with continued shearing. The behaviour exhibited by the larger bin, i.e., the quadrant bin should be considered a better representation of the bulk phenomena in the shear cell than the smaller rectangular bin as the former covers a larger volume of the shear cell and would thus

offer a better representation of the phenomena taking place in the shear cell. The smaller rectangular bin in the case of normal overlap might be capturing some localized effect in the shear cell (albeit it being present in the 4 radial locations of the shear cell) and it cannot be used to overrule the observations made from the larger quadrant bins. Thus, considering the quadrant bins to be more accurately representing the phenomena in the shear cell, the average normal overlap does not seem to be influencing the failure strength of the sample linearly as the normal overlap is larger for partial consolidation than for critical consolidation despite the former having a smaller failure strength than the latter.

5.2.3 Fabric anisotropy

To characterize the structure of the granular fabric, fabric anisotropy calculations can be used and the contact normal based calculation method built on the works of Satake (1982) and Oda (1982) is used in this thesis. The fabric tensor F_{ij} can be used to compute particle contacts orientation, it can be calculated from the contact vector in the normal direction of the particle contacts. The anisotropy of the contact normals in a granular assembly can be determined by computing F_{ij} (Kodicherla et al., 2024; Zhao and Guo, 2013) from Equation (5-12).

$$F_{ij} = \frac{1}{N_c} \sum_{c=1}^{N_c} n n = \begin{bmatrix} n_i n_i & n_i n_j & n_i n_k \\ n_j n_i & n_j n_j & n_j n_k \\ n_k n_i & n_k n_j & n_k n_k \end{bmatrix} \quad (5-12)$$

where N_c is the total number of particle contacts, n is the unit contact normal vector, n_i , n_j and n_k represent the components of n in x , y and z directions respectively.

The fabric tensor F_{ij} is a symmetric tensor with $Tr(F_{ij}) = 1$, its deviatoric part F_{ij}^d (Khayyer et al., 2024) can be defined as Equation (5-13),

$$F_{ij}^d = F_{ij} - \frac{1}{3} Tr(F_{ij}) I \quad (5-13)$$

where I is a 3rd order identity matrix. Fabric anisotropy can be quantified by determining the scalar quantity a_n (Khayyer et al., 2024) from the deviatoric part of the fabric tensor from Equation (5-14),

$$a_n = \sqrt{\frac{3}{2} F_{ij}^d : F_{ij}^d} \quad (5-14)$$

Scalar anisotropy a_n helps quantify the degree of anisotropy in the particle contacts and can help understand the evolution of the particle contact orientation with shear strain in shearing of a granular assembly. A higher value of a_n indicates a greater number of particle contacts aligned with the principal stress direction with a relatively lower value indicating more randomness in the particle contact orientation, a value close to zero would indicate that the granular assembly is closer to an isotropic packing. Isotropic packing of particles essentially means that the particle contacts are uniformly distributed in all directions and there is no specific

or preferred direction along which the particle contacts are aligned. Adesina et al. (2024) determine the scalar anisotropy as the difference between the major and minor eigenvalues of the fabric tensor.

The fabric tensor F_{ij} was computed for 8 bins (rectangular bins 1-4 & bins 5-8) located at two radial locations in the shear cell (Figure 5-4) and further used to calculate contact anisotropy from Equation (5-14) for partial and critical consolidation states. The average particle diameter considering the particle as a perfect sphere is 0.71 mm, therefore the rectangular bin dimensions are approximately $14 d_{50} * 11 d_{50} * 5 d_{50}$, which is appropriate for computing the localized particle properties in the shear zone. The smaller rectangular bin size was considered for computing the anisotropy as the principal stress and strain directions can vary in different zones of the shear cell and can influence the contact anisotropy, thus considering a smaller bin size better represents the anisotropy of that localized zone and captures the local orientation of the particle contacts along the principal stress direction. The fabric tensor was calculated at two instances for both the critical and partial consolidation cases, once at the time of consolidation or shearing and once after the sample has been relieved from the shear stress and is about to undergo shear to failure. For both cases, the particle unit normal contact vectors across 5 time-steps located near the time instance was used for calculations, the particle contacts at each of these time-steps averaged to be around 2000. For critical consolidation, unit contact normal vectors for 5 time-steps centered around 14 s and 16.5 s were utilized, for the partial consolidation case, data at 5 time-steps preceding the end of partial consolidation at 10.218 s was used and 5 time-steps centered around 10.8 s for the sample ready for the shear to failure step.

Table 5-1 shows the scalar anisotropy a_n values for the two cases at the two time instances. The shear zone is located near the top bins 1 & 5 (beneath the shearing lid vanes), anisotropy for top-most bins is on average approximately double at critical consolidation than at partial consolidation. Anisotropy increases with the continuation of shearing which is expected as the particles rearrange and the particle contacts align themselves along the principal stress direction. For the powder bed state post counter rotation of sample analyzed at time instances of 16.5 s and 10.8 s for the critical and partial consolidation states respectively, scalar anisotropy is still greater for critical consolidation than partial consolidation possibly justifying the former's higher failure strength. Higher number of particle contacts aligned along a particular direction can lead to a higher failure strength in that particular direction and thus cause the failure strength to be higher than the case of partial consolidation where contact normal orientations are relatively more random.

Table 5-1: Scalar anisotropy a_n for a) critical consolidation, and b) partial consolidation.

a) Critical consolidation					
Time (s)	Bin1	0.1176	Time (s)	Bin1	0.0987
14s	Bin2	0.1031	16.5s	Bin2	0.1003
	Bin3	0.0909		Bin3	0.0896
	Bin4	0.0883		Bin4	0.0855

14s	Bin5	0.1154	16.5s	Bin5	0.1043
	Bin6	0.1042		Bin6	0.1086
	Bin7	0.0895		Bin7	0.0871
	Bin8	0.0801		Bin8	0.0852
b) Partial consolidation					
Time (s)	Bin1	0.0431	Time (s)	Bin1	0.0448
10.21s	Bin2	0.0499	10.8s	Bin2	0.0489
	Bin3	0.0702		Bin3	0.0682
	Bin4	0.0669		Bin4	0.0635
10.21s	Bin5	0.0658	10.8s	Bin5	0.0692
	Bin6	0.0647		Bin6	0.0622
	Bin7	0.0734		Bin7	0.0718
	Bin8	0.0566		Bin8	0.0571

5.3 Bulk stresses, strain, and coaxiality

The particle contact forces determined by DEM can be used to calculate the stresses at the particle level. These particle level properties can be utilized to determine average values of these properties over a defined area or volume, which can be referred to as the average or bulk stresses. There are many approaches available to extract the macroscopic data from the particle level data in DEM, two commonly used approaches are broadly discussed here, a) continuum analysis, and b) bin averaging. In continuum analysis the property is calculated at a point, a three-dimensional sphere with a specified diameter is built around the point. The particle contacts and resulting forces in this sphere of influence are determined. A Gaussian weighing function is used to determine the weightage of each particle's contribution to the property calculated at the point, the farther the particle from the point of interest, lower the contribution of that particle in the property calculated at that point, this technique can be referred to as Gaussian averaging. The second method of bin averaging averages the properties of all the particles present in the bin volume by weighing the particles as per their volume; this will be referred to as the bin averaging technique. The shape and dimensions of the bin can be varied to define the bin volume and the resultant number of particles in the bin.

The stress tensor for a single discrete element or particle can be determined from Equation (5-15) as,

$$(\sigma_{ij})_p = \frac{1}{V_p} \sum_{c=1}^{n_{pc}} F_i^c S_j^c \quad \text{with } i, j = x, y, z \quad (5-15)$$

$$(\sigma_{ij})_{average} = \frac{\sum_{p=1}^{N_p} V_p (\sigma_{ij})_p}{\sum_{p=1}^{N_p} V_p} \quad (5-16)$$

where V_p is the volume of the particle p , n_{pc} is the number of particles in contact with the particle p , S^c is the vector connecting the particle centre with the c^{th} contact point and F^c the c^{th} contact force. The average particle stress $(\sigma_{ij})_{average}$ can be determined from Equation (5-16), where N_p represents the total number of particles being considered for averaging.

The contact force F_i^c can be decomposed into its normal and tangential components. For example, F_x^c (5-17) would comprise of the normal contact force component in the x direction $F_x^{c,n}$ and the tangential contact force component $F_x^{c,t}$ in the x direction. The unit vectors n and t in the normal and tangential contact directions respectively can be used to compute $F_x^{c,n}$ and $F_x^{c,t}$ (Equation (5-18)).

The unit contact normal vector and the unit tangential contact vector can be represented as its directional components as $n = n_x\hat{i} + n_y\hat{j} + n_z\hat{k}$ and $t = t_x\hat{i} + t_y\hat{j} + t_z\hat{k}$.

$$F_x^c = F_x^{c,n} + F_x^{c,t} \quad (5-17)$$

$$F_x^c = f^n n_x + f^t t_x \quad (5-18)$$

where f^n and f^t are the normal and tangential components of the contact force.

σ_{xx} can thus be computed by using F_x^c together with S_x^c which would be the x direction component of the contact vector S^c .

The average stress tensor for a bin is determined by particle volume weighted average of the stress tensor of all the particles present in that bin. The spatial scale influences the number of particles under consideration for both the bin averaging and Gaussian averaging methods. It is thus important to determine the appropriate volume of interest for both the methods. In the previous section, two bin sizes were considered for evaluating the particle properties such as coordination number, normal overlap and fabric anisotropy. The bin averaging technique, where the stress tensor for each particle in the bin was determined and then particle volume weighted averaging was done to obtain the average value for the bin was used to analyze the stress tensor for the annular quadrant and rectangular bins. The bin averaging technique averages the properties over an entire volume of interest and thus is more suitable for analyzing the stresses in the shear zone of a shear cell as the shear zone is a volume element in a 3-dimensional shear cell.

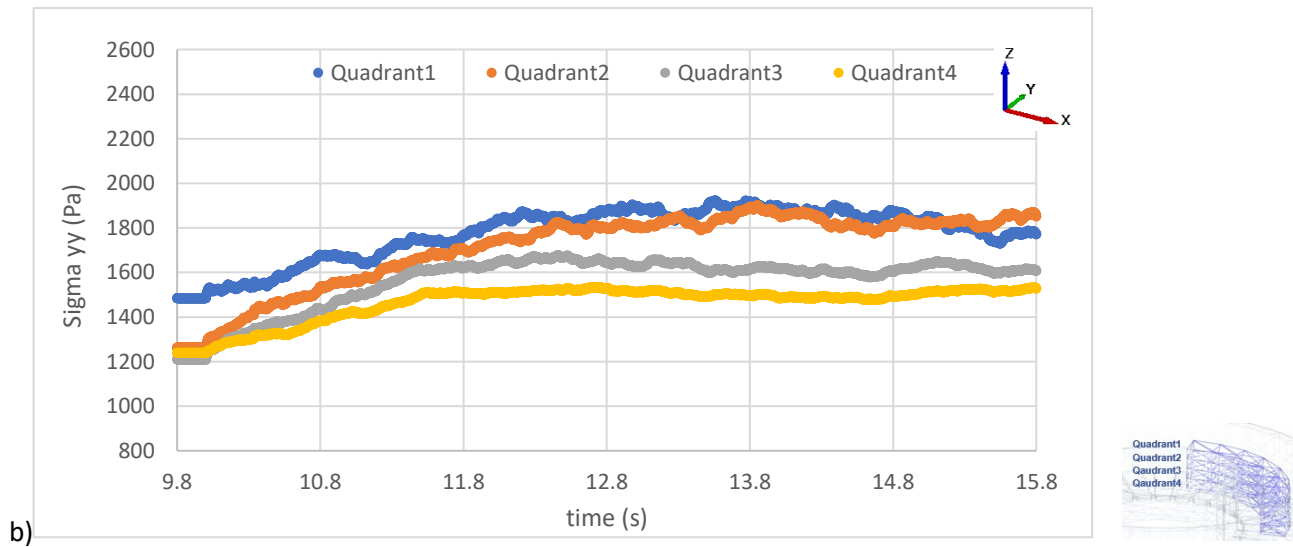
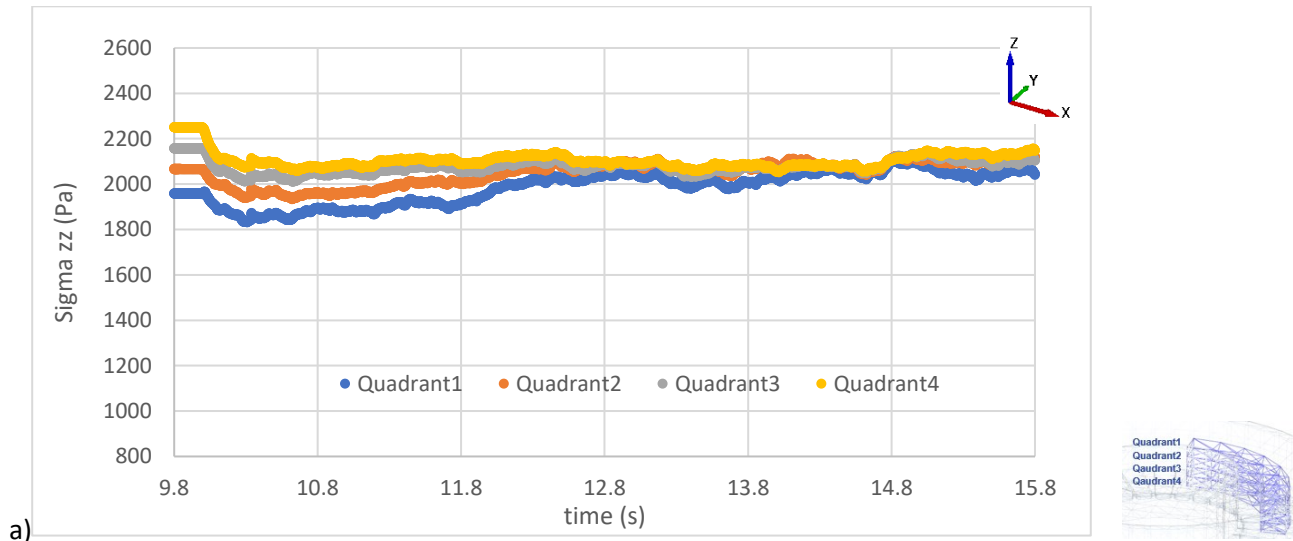
The average stress tensor of a bin $(\sigma_{ij})_{bin}$ is determined by adjusting the average particle stress of the particles (Equation (5-16)) in the bin as Equation (5-19).

$$(\sigma_{ij})_{bin} = \frac{\sum_{p=1}^{N_{p,bin}} V_p (\sigma_{ij})_p}{V_{bin}} \quad (5-19)$$

where V_{bin} is the geometrical volume of the bin.

The normal stress σ_{zz} , σ_{yy} and shear stress τ_{yz} for the quadrant bins (Figure 5-3) were determined from (5-19) and were presented in Figure 5-14. The annular quadrant bins were selected for further analysis as the

rectangular bins showed higher fluctuations in the stress values than their quadrant counterparts. The normal stress in the vertical direction σ_{zz} increased while going from the top bin 1 to the bottom most bin 4, the increase was seen because of the increasing mass of powder compressing the lower bins in addition to the pre-shear normal stress applied through the shearing lid. Shear stress τ_{yz} was highest for the top-most bin 1 and decreased progressively while moving towards the bottom bins, the higher shear stress in bin 1 can likely be attributed to the presence of the shear zone near that bin.



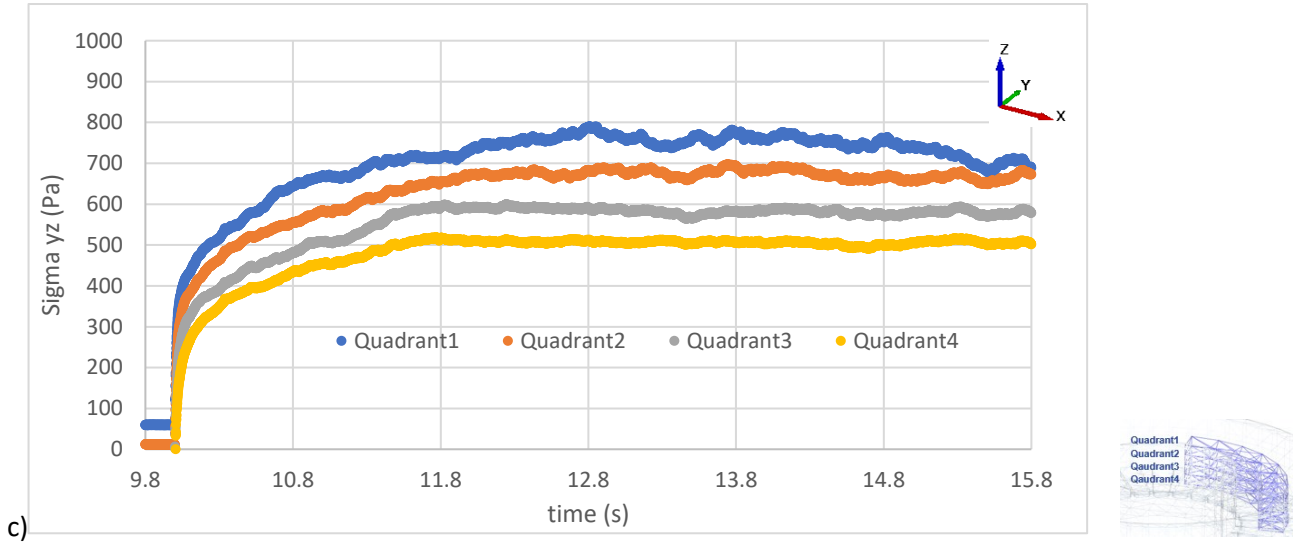


Figure 5-14: Stress evolution with depth, a) normal stress σ_{zz} , b) normal stress σ_{yy} , and c) shear stress τ_{yz} . The inclination of the principal stress plane ψ_{stress} from the vertical plane can be determined from the normal and shear stresses for the 4 quadrant bins spread across the depth of the shear cell by utilizing the relation, $\psi_{stress} = \frac{1}{2} \tan^{-1} \left(\frac{2\tau_{yz}}{\sigma_{zz} - \sigma_{yy}} \right)$. ψ_{stress} is determined for the four quadrants throughout the pre-shear process and thus helps in evaluating the principal stress orientation evolution with shearing across the depth of the shear cell. ψ_{stress} is also analyzed at specific time-steps during pre-shear when the sample is partially consolidated to 50% (10.035 s) and 75% (10.218 s) states, and also at critical consolidation. Before the start of pre-shear at 10 s, the principal stress directions approximately coincide with the horizontal and vertical planes for bin 1 and very closely for bin 2-4 as there is absence of significant shear stress. Figure 5-14 show non-zero shear stress at 9.8 s even before the start of pre-shear because the origin of this shear stress is particle stress which is derived from the contact forces, the non-zero contact forces result in non-zero shear stress which causes the principal stress planes to be inclined or rotated from the horizontal and vertical planes, the principal planes coincide with the horizontal and vertical planes when the shear stress is zero.

Table 5-2a presents the evolution of the principal stress directions with pre-shear process from 10 s - 16 s, moving from the bottom-most bin towards the top-most bin at all the 4 time-steps of consideration showed the rotation of the principal stress direction away from the horizontal and vertical planes. The shear zone is located near bin 1, therefore we consider the orientation of the principal stress planes at the partial consolidation states of 50% (10.035 s), 75% (10.218 s) and critical consolidation (11.7 s & 14 s) in bin 1 for evaluation. The principal stress plane rotates increasingly away from the horizontal and vertical planes and towards 45° as shearing proceeds towards critical consolidation as can be seen from Table 5-2a. Figure 5-15 shows the evolution of the principal stress direction for the 4 bins, it can be seen that the principal stress plane orientation stabilizes as the sample reaches critical consolidation. The horizontal stress σ_{yy} increases as the vertical stress σ_{zz} decreases upon shearing, as this transformation takes place, in the top bin both

values approach each other (Figure 5-14) due to which as shearing continues, the principal stress plane orientation approaches 45° in the top-most bin for the critical consolidation states at 11.7 s & 14 s.

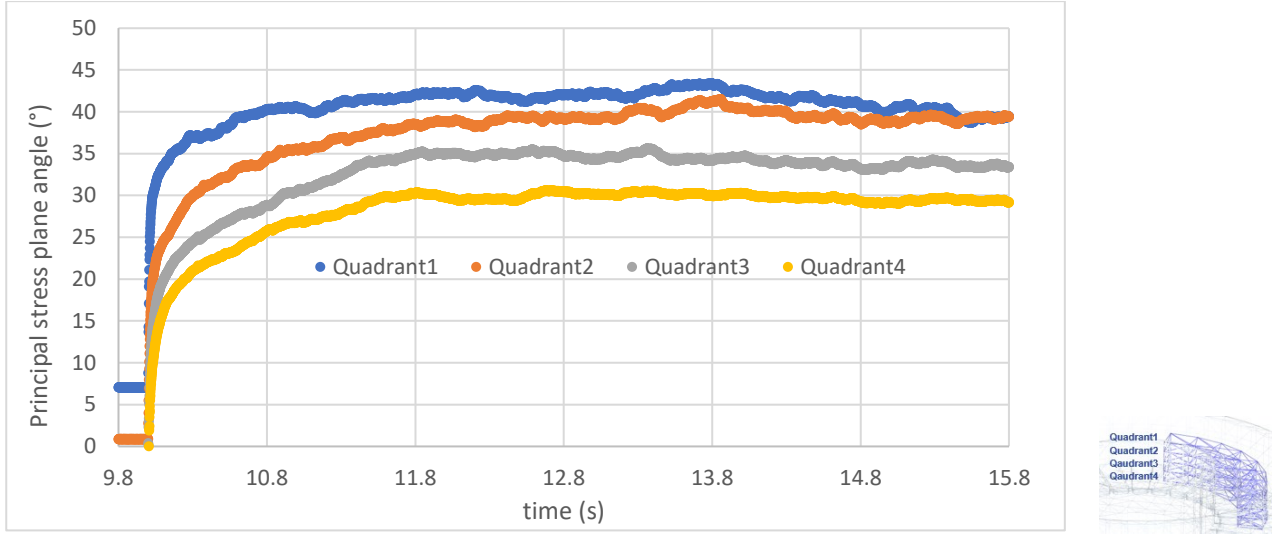


Figure 5-15: Principal stress plane evolution with shearing.

The average particle angular velocity of the bins can be determined by using the average x and y particle velocities in the bins by utilizing the relation $\omega_{bin} = -v_x \sin \theta_r + v_y \cos \theta_r$, where θ_r is the angular location of the bin centroid. The rectangular bins are chosen instead of the annular quadrant bins as in the latter the angular location of the particles varies greatly, and it is not appropriate to calculate the average bin velocity by considering the particle velocities at the quadrant centroid. For computing the principal strain plane inclination, the normal strain of the powder is calculated by considering the bulk density of the 4 rectangular bins. The instantaneous shear $d\gamma_{yz}$ and normal strains $d\varepsilon_{zz}$ are determined by Equations similar to Equation (3-5) and Equation (3-6) respectively. For determining the shear strain (Equation (5-20)), the trough velocity is replaced by $\Delta\omega_{bin}$, the powder bed thickness is replaced by the bin depth (4 mm), and the radial distance r_{rad} from the centre of rotation is taken as the centroid of the bin.

$$d\gamma_{yz} = \frac{r_{rad} \Delta\omega_{bin}}{L} dt \quad (5-20)$$

where $\Delta\omega_{bin}$ for the top bin is determined as $\omega_{bin,2} - \omega_{bin,1}$ and for the bottom most bin as $\omega_{trough} - \omega_{bin,4}$.

The bulk density of the sample is replaced by the bin bulk density for determining the normal strain, dt used for the shear and normal strain calculations is 0.04 s. The angular velocity (Figure 5-16) and bulk density (Figure 5-17) are time-averaged over a rolling window for smoothening the fluctuations in these two quantities for computing the strain values and further the principal strain angle. The data is temporally averaged using a rolling (moving) average with a time window of 0.1 s. The principal strain angle is determined at the specific time stamps when the sample is partially consolidated at 50% (10.035 s) and 75% (10.218 s)

states and further when the sample has reached critical consolidation and is continued to be sheared at 11.7 s and 14 s respectively, these shearing states can be seen from the shear stress time series curve at critical consolidation (Figure 5-2). The principal strain angle is determined from $\psi_{strain} = \frac{1}{2} \tan^{-1} \left(\frac{d\gamma_{yz}}{d\varepsilon_{zz}} \right)$

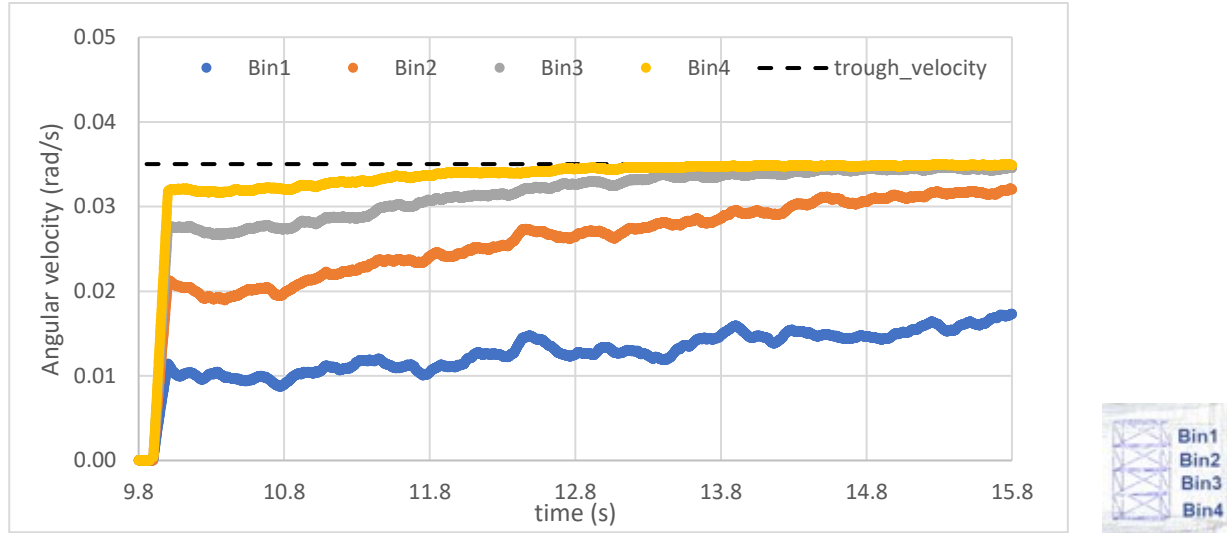


Figure 5-16: Angular velocity evolution with shearing for bins 1-4.

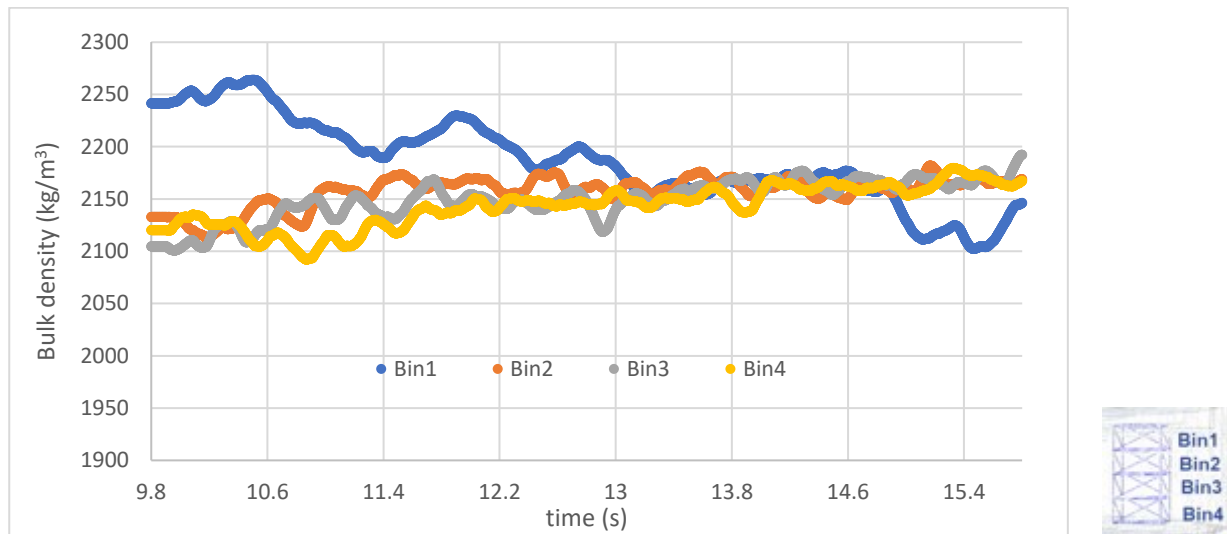


Figure 5-17: Bulk density evolution with shearing for bins 1-4.

Table 5-2: a) Principal stress angle ψ_{stress} , b) principal strain angle ψ_{strain} , and c) non-coaxiality.

a) Principal stress angle (°) ψ_{stress}

Quadrant	Bin centroid location (mm)	50% 10.035 s	75% 10.218 s	100% 11.7 s	100% 14 s
Quadrant 1	0.007	29.26	35.57	41.71	42.57
Quadrant 2	0.011	18.88	27.84	37.94	40.42
Quadrant 3	0.015	13.21	22.97	34.49	34.58
Quadrant 4	0.019	8.89	19.45	29.96	30.21

b) Principal strain angle ($^{\circ}$) ψ_{strain}

Bin	Bin centroid location (mm)	50% 10.035 s	75% 10.218 s	100% 11.7 s	100% 14 s
Bin 1	0.007	31.81	32.96	38.81	42.05
Bin 2	0.011	-22.81	29.13	38.55	21.06
Bin 3	0.015	21.54	9.28	16.41	-4.01
Bin 4	0.019	34.22	-40.35	-14.43	0.76

c) Non-coaxiality ($^{\circ}$)

Bin/ quadrant	Bin centroid location (mm)	50% 10.035s	75% 10.218s	100% 11.7s	100% 14s
1	0.007	2.53	-2.61	-2.88	-0.52
2	0.011	-41.69	1.28	0.61	-19.35
3	0.015	8.33	-13.68	-18.08	-38.59
4	0.019	25.33	-59.81	-44.39	-29.44

Table 5-2b presents the evolution of the principal strain plane orientation for the specified time instances at partial and critical consolidation. The principal strain plane rotates towards 45° for top-most bin 1 as shearing continues from partial to critical states, similar to the principal stress plane direction behaviour. There is decreasing shear strain in bottom most bin 4 (Figure 5-18) as pre-shearing proceeds, negligible shear strain at 14 s causes the principal strain planes to almost incline with the horizontal and vertical planes as seen in Figure 3-3a. Table 5-2c presents the degree of non-coaxiality between the principal strain and stress planes determined as $\psi_{strain} - \psi_{stress}$. For the top-most bin 1, the principal strain and stress planes have a low degree of non-coaxiality for the 50% & 75% partial consolidation state as well the critical states at 11.7 s & 14 s. As the shear zone is located near the top-most bin, it can be said for the partial and critical consolidation states the principal strain and stress planes are nearly coaxial with regards to the top-most bin.

5.4 Shear strain evolution with depth

As discussed earlier in Section 5.2 shear strain localization takes place just below the shear lid vanes as the sample is sheared to critical consolidation. Figure 5-18 presents the shear strain (Equation (5-20)) across the 4 rectangular bins determined at the partial consolidation states of 50% (10.035 s) and 75% (10.218 s) states and further when the sample has reached critical consolidation and is continued to be sheared at 11.7 s and 14 s respectively. As can be seen from Figure 5-18, there is significant shear strain in the bottom most bins 3 & 4 at the partial consolidation states of 50% (10.035 s) and 75% (10.218 s), the shear strain variation is almost linear for the partially consolidated states and is maximum for the top bin 1. As the shearing proceeds and the sample reaches critical consolidation, the shear strain in the bottom most bins approaches zero and the shear zone progressively develops in bin 1 or just below the shearing lid vanes. The shear strain for the top

most bin at 14 s is more compared to the value at 11.7 s, for all other bins the shear strain is lower at 14 s than the values at 11.7 s, which indicates that as the shearing is continued beyond the critical consolidation the shear strain and thus the shear zone is localized even more below the shearing lid vanes.

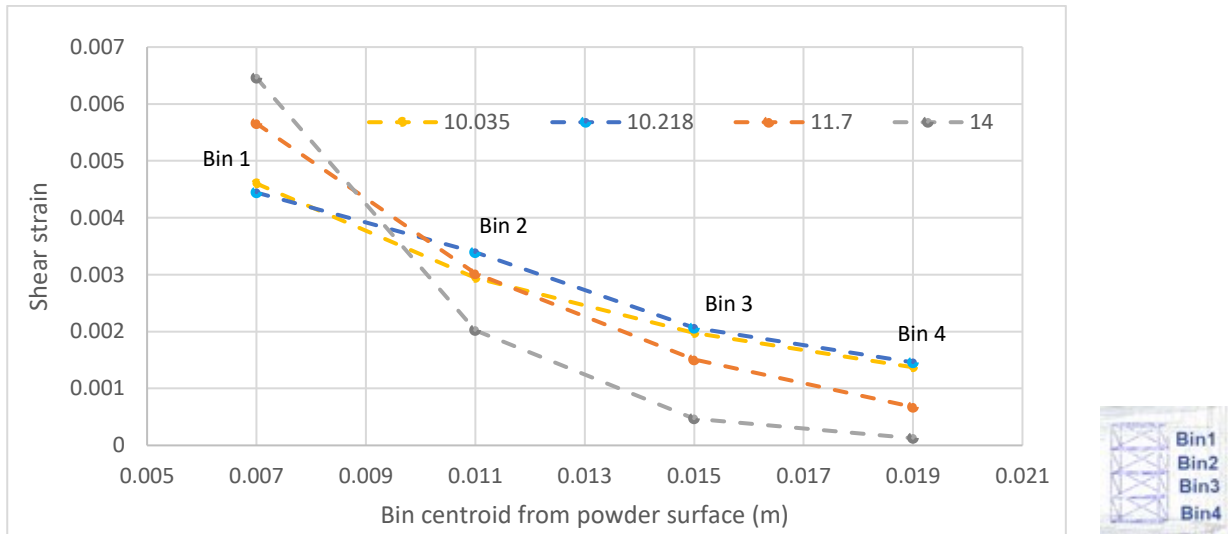


Figure 5-18: Shear strain evolution with depth for shear cell.

Flat bottom silo simulations were run with the calibrated DEM powder (Section 4.3) to study the evolution of shear strain along the depth of the powder bed during shearing initiated from the shearing lid at the top. EEPA model was used to model the powder interactions with Hertz-Mindlin model used for modelling particle-geometry interactions, the modelled silo had approximately 250,000 particles. Figure 5-19 shows the silo apparatus used in the DEM simulations with the sample sheared through the shearing lid whilst also applying a normal load to the sample, 2 bin locations located at 25 mm and 50 mm radially from the centre of rotation were utilized to study the shear strain evolution. Each bin had dimensions of 10 mm*10 mm*10 mm, with 6 bins vertically stacked at the two locations, the depth of the powder bed was approximately 60 mm, silo diameter was 150 mm with the shearing lid having diameter of 147.5 mm, the silo simulations were carried out at the real scale geometrical dimensions of the experimental silo as mentioned in Chapter 3.

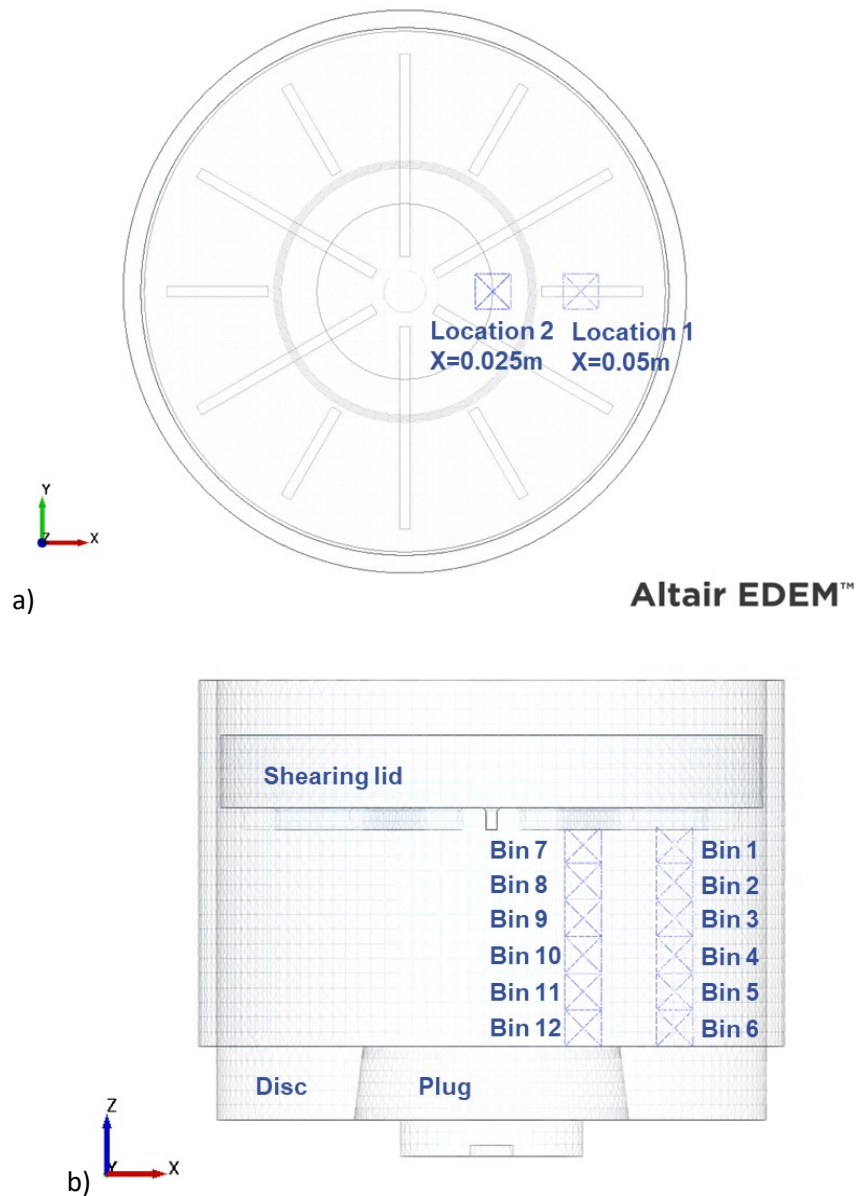


Figure 5-19: Bin locations spread out radially and across the depth of the flat bottom silo, a) top-view, b) isometric view.

Figure 5-20 presents the angular velocity variation with shearing at the two radial locations of 25 mm and 50 mm, as can be seen the angular velocity in the top most bins 1 & 7 approach the shearing lid velocity of 0.035 rad/s, bins 2 & 8 show reduced angular velocities in comparison to the top bins, indicating reduced particle movements in the lower bins. Bins lower than bins 2 & 8 show some particle movement at the start of shearing, but with continued shearing the movement becomes minimal indicated by the low bin velocities for bins 3-6 and bins 9-12. This finding indicates shear localization in the top two bins with continued shearing.

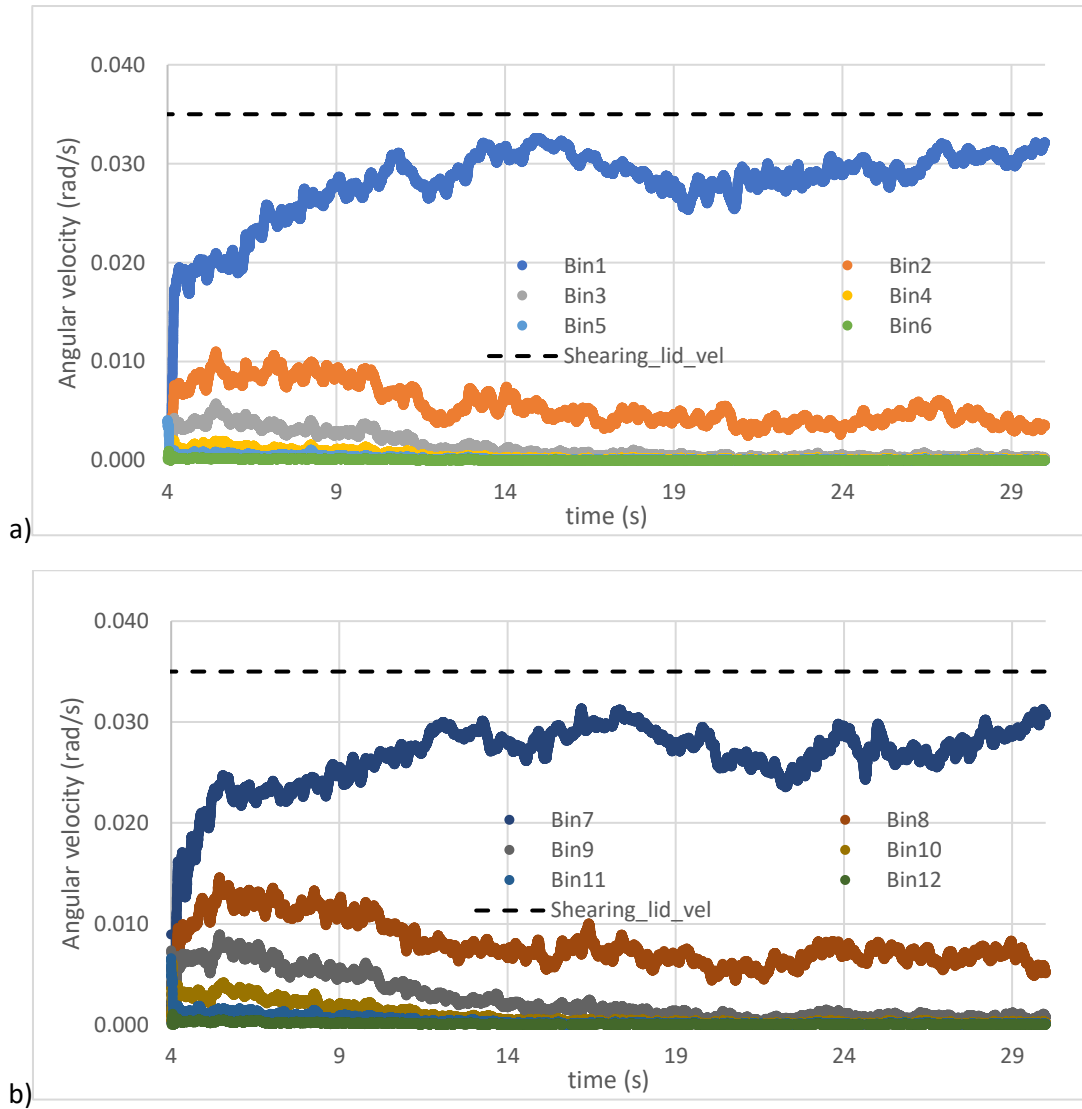


Figure 5-20: Angular velocity evolution with shearing in flat bottom silo for a) bins 1-6, and b) bins 7-12.

Figure 5-21 shows the shear strain evolution with depth across two radial locations at specific time steps of 5 s (50% partial consolidation), 6.5 s (75% partial consolidation) and critical consolidation at 13 s and 28 s. Figure 5-22 presents the shearing lid time series curve for the flat bottom silo, with shearing starting at 4.2 s and continuing till 30 s. Shear strain for the silo simulations was calculated as per the methodology discussed in the previous section. At the more distant radial location of 50 mm where bins 1-6 are located, shear strain was minimal at 5 s & 6.5 s in the lower most bins with bins 3 & 4 showing some shearing, shear strain in the top bins 1 & 2 is significantly larger than the others, indicating the presence of the shear zone. At the critical state of 13 s & 28 s, bottom bins 4-6 showed minimal shearing, with significant shearing taking place in bins 1 & 3 and the shear zone localization in bin 2.

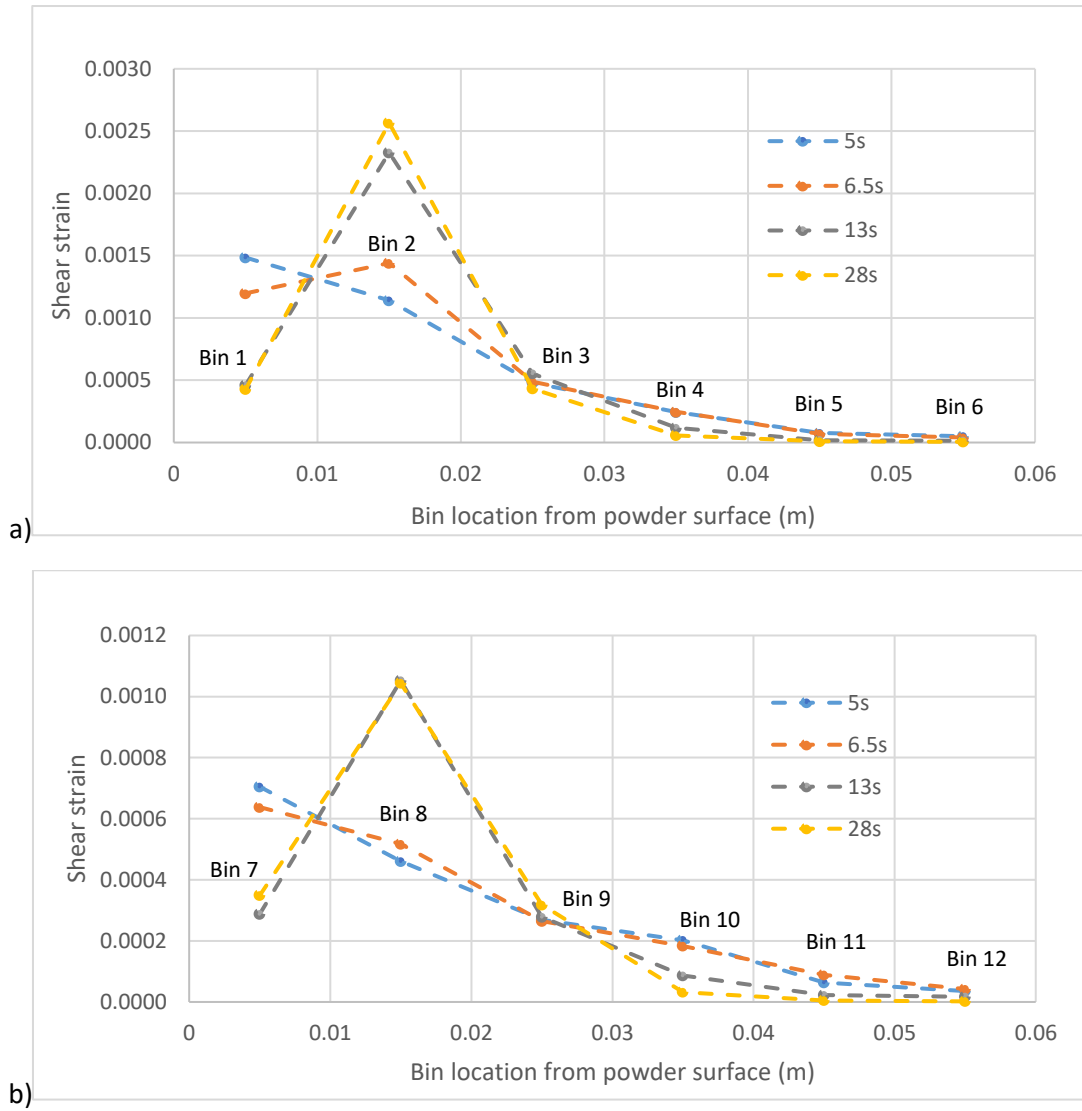


Figure 5-21: Shear strain evolution with shearing in flat bottom silo at specific timesteps for a) bins 1-6, and b) bins 7-12.

At the radial location of 25 mm where bins 7-12 are located, partial consolidation states at 5 s & 6.5 s showed an almost linear increase in the shear strain when going from the bottom bin 12 to the top bin 7. For the critical consolidation states at 13 s & 28 s, just as the case of radial location of 50 mm, shear strain localization took place in bin 8, with significant shearing taking place in bins 7 & 9. Although the bin depth in the silo simulations is 10 mm and hence greater than the bin depth of 4 mm in the shear cell simulations, both set of results show shear strain localization and therefore the presence of shear zone in certain localized zones or areas of the bulk solid. Although the size of the silo bin is greater than the shear cell at 10 mm, but both are approximately 5 particle diameters deep, the silo simulations used the dual sphere particle used in calibration of the DEM model (d_{50} of 1.5 mm assuming the particle to be a perfect sphere).

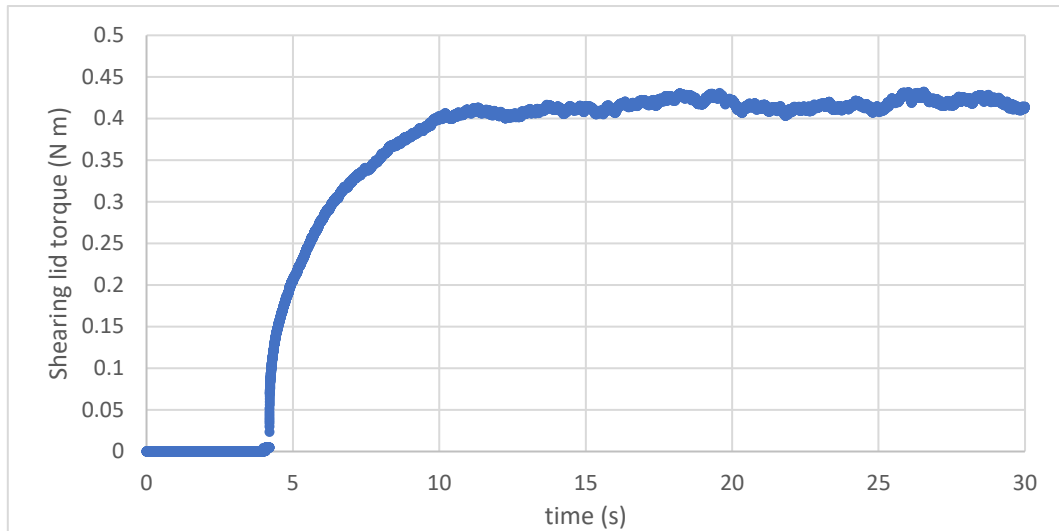


Figure 5-22: Shearing lid torque time series curve for flat bottom silo.

5.5 Summary

The Chapter first presented the upscaling of the calibrated DEM model to obtain a higher fidelity model for studying microscopic particle behaviour at the mesoscopic scale. Next, the coordination number, particle normal overlap and fabric anisotropy for the critical and partially consolidated systems was studied to understand the reasoning behind variable failure strength between the two. The results showed that neither coordination number nor particle normal overlap exhibited a direct correlation with powder strength. On the other hand, the fabric anisotropy at the end of consolidation was significantly greater for critically consolidated powders, indicating a stronger alignment of contacts along the principal stress direction and suggesting a link between contact network anisotropy and increased failure strength.

The Chapter then presented the results of the bulk stresses and strains of the cohesive powder under shearing in the shear cell derived from the particle level data; these results were used to study the principal stress and strain plane orientation and the shear strain evolution with depth for the shear cell. These results demonstrated the capability of the DEM model to connect and study bulk powder behaviour with the underlying particle phenomena at the mesoscopic scale.

Chapter 6 Conclusions and Recommendations for Future Research

6.1 Conclusions

This doctoral research investigated the flow behaviour of cohesive powders under varying consolidation states through a combination of flow characterization experiments, and discrete element modelling (DEM) simulations. The study was motivated by the hypothesis that under certain handling conditions the powder during flow may not achieve critical consolidation and may instead be partially consolidated. The flow behaviour under varying consolidation states was investigated through shear cell and flat-bottom model silo experiments. A DEM model of shear testing was developed and calibrated using shear cell experiments to reproduce experimental flow behaviour, and to study the underlying phenomena behind the variable failure strength (unconfined yield strength) at the critical and partial consolidation states. The following conclusions can be drawn from the work carried out in this doctoral work.

For partially consolidated powders, the powder has not achieved steady state during the consolidation process. The steady state flow is not fully developed; and the partially consolidated state is achieved at a smaller shear deformation than that required for the steady state. An experimental methodology was proposed and applied to study the flowability of partially consolidated powders. The experiments were conducted on a rotational shear tester (Schulze RST) by modifying the consolidation step such that the powder consolidation was stopped at shear stresses that are a defined fraction of the steady state value. The strains to reach the critical state and the partially consolidated states were measured and discussed, as well as the failure strength of the material detected with the shear experiments. The experimental results demonstrated that partially consolidated powders exhibit yield loci with significantly lower cohesion and unconfined yield strength obtained at significantly smaller shear strains.

Different strategies were adopted and discussed to estimate the unconfined yield strength and the major principal stress from data pertaining to critically and partially consolidated powders. Despite the above-mentioned effects of consolidation on the powder strength, the comparison between the flow functions reveals that the powder flow function is only marginally affected by the level of consolidation. This result suggests that the design of the silo's critical outlet dimension at critical consolidation can provide a reasonably accurate prediction of the powder's flow behaviour, across the different degrees of consolidation at which the powder can operate.

To assess the relevance of the state of consolidation in practical applications, a separate set of experiments was carried out on the discharge from flat-bottom silos. The varying states of consolidation in the discharge experiment were recreated by using a rotational shearing lid and a compression lid to subject the powder sample to varying rotational displacements before discharge. The model silo experiments verified the results

obtained from the shear tester, as the deformation or the extent of shearing did not significantly affect the cohesive blockage and the discharge of the powder from the silo.

A coarse-grained cohesive Discrete Element Method (DEM) model was developed and calibrated using experimental shear testing data. The critical model parameters were identified through a parametric sensitivity study, followed by a fractional Design of Experiments (DOE) study of the identified critical parameters to explore the parametric space. A Multi Objective Genetic Algorithm (MOGA) was then employed to calibrate the DEM model by minimizing selected objective functions. The calibrated DEM model for the ESKAL powder successfully predicted key powder flow properties, including cohesion, effective angle of internal friction, and the yield loci across a range of consolidation stresses.

The DEM model is able to reproduce the stress history dependent flow behaviour, and the flow function of the resulting unconfined yield strength as a function of the consolidation stress for the critically consolidated state. The DEM model was also able to match the experimental yield loci across a range of pre-shear normal stresses for the partially consolidated state. The calibrated DEM model was thus able to predict and match the experimental powder flow behaviour for the powders under critical and partial consolidation, although for critical consolidation, the DEM response was stiffer at lower strains, and steady state was achieved earlier than observed experimentally. The coarse-graining technique was found to be a useful modelling tool for conserving or reproducing the key characteristics of the original system whilst decreasing the computational costs.

To gain insights into the micro-scale mechanics governing powder strength, the calibrated model was upscaled using parametric scaling laws to obtain a higher fidelity model. This model was used to study the mesoscopic scale particle level phenomena behind the variable failure strength at the critical and partial consolidation states. The particle scale analysis focused on coordination number, fabric anisotropy and particle normal overlap. These quantities were analyzed across various bins spread out radially and across the depth of the shear cell. The results show that there is no direct correlation between the powder failure strength and either coordination number or particle normal overlap. At the end of consolidation, the scalar anisotropy associated with critical consolidation was on average approximately twice that observed under partial consolidation for top-most bins. This indicates a larger number of particle contacts aligned along the principal stress direction leading possibly to an increased failure strength.

This doctoral research makes the following contributions to the field of solids handling and processing:

1. It develops an experimental methodology for characterising powder flow under partial consolidation states, extending conventional critical-state based shear testing approaches.

2. It demonstrates that although partially consolidated powders exhibit lower cohesion and unconfined yield strength than critically consolidated powders, their flow functions are only marginally affected.
3. It develops and calibrates a coarse-grained cohesive DEM model capable of reproducing both critical and partial consolidation behaviour across a range of consolidation stresses.
4. It provides new insights into the particle-scale origins of powder flow behaviour dependent on its consolidation state.

The following are the practical implications arising from the work.

- Silo design based on critical-state flow functions should remain reliable even when powders operate under partial consolidation states.
- Discrete element modelling can be confidently applied to model micron-scale cohesive powders using larger meso-scale particles, enabling prediction of flow behaviour and providing a practical tool for studying various solids handling processes with careful consideration of appropriate scaling.

Publications based on this thesis:

- Flowability assessment of partially consolidated powders (being finalized for submission to journal)
- Meso-scale DEM modelling of cohesive powders (being finalized for submission to journal)

6.2 Recommendations for future research

This section puts forward some potential areas where carrying out further research can be beneficial.

Flowability characterization at varying consolidation states for a broader range of cohesive powders (other than CaCO_3 and MgCO_3) and at a wider range of pre-shear normal loads can be conducted. This would help in gauging the effect of partial consolidation on the flowability of powders at wider range of consolidation stress levels and help investigate the influence of particle morphology and size distribution on the flow characteristics. Characterizing the flowability at 25% partial consolidation levels, in addition to 50%, 75% and critical consolidation levels, can help to study the flow behaviour at full range of consolidation levels.

The DEM modelling work can be extended towards the development of a more comprehensive model capable of reproducing additional aspects of powder flow behaviour beyond yield loci and flow function characteristics. This enhanced DEM model would aim to quantitatively capture shear-induced densification during consolidation, dilation during shear to failure, and the evolution of shear stress as a function of shear strain. Incorporating these behaviours can allow the DEM model to more accurately represent the evolution of density and fabric tensor, thereby improving its predictive capability for both critical and partial consolidation states.

Future DEM work can focus on further exploring and resolving the micro-mechanical origins of the variable failure strength between critical and partial consolidation. The strong and weak force chains during critical and partial consolidation can be examined to study how the force chain network and stability influences the unconfined yield strength.

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Appendix A

The following section presents and discusses some of the scaling studies undertaken in literature, which are categorized into two primary approaches: parameter scaling and force scaling.

The studies presented and discussed in this Appendix are reproduced from TUSAIL's internal reporting documents.

I. Coarse-graining techniques

a) Parameter-scaling laws

Nakamura et al. (2020) developed a coarse-grained method for granular shear flow (CGSF) for a powder mixing process. The friction damping energy (energy dissipation due to sliding friction), elastic energy, kinetic energy and viscous damping energy were matched for the coarse-grained and original particles and scaling laws for sliding friction coefficient, stiffness and restitution coefficient were derived based on the energy matching principle. The original particles are modeled as a simple cubic lattice structure and the interactions between these particles in the lattice is used for deriving the scaling laws for the representative coarse grain. The coarse grained (CG) particle scales according to the coarse-graining ratio l such that the diameter of the coarse-grained particle $d_c = ld_o$ where d_o is the original particle diameter. The friction damping energy between the original particle group and the CG particle was matched for formulating the scaling law for the sliding friction coefficient with it being scaled according to Equation (A-1),

$$\mu_{C,PP} = \mu_{O,PP} + \mu_{O,PP} \frac{(l-1)\beta(l)d_o(|\omega_i^{CG}n_{ij}^{\wedge}| + |\omega_j^{CG}n_{ij}^{\wedge}|)}{[(v_{t,i}^{CG} - v_{t,j}^{CG}) + \beta(l)(r_c - r_o)(\omega_i^{CG} + \omega_j^{CG})n_{ij}^{\wedge}]}$$
 (A-1)

$$\text{where } \beta(l) = \left\{ \left(\frac{1}{l}\right)^2 + \left(1 - \frac{1}{l}\right)^2 \right\}^{-1/2}$$

$\mu_{C,PP}$ and $\mu_{O,PP}$ are the sliding friction coefficients of the CG and original particles respectively, v_t^{CG} and ω^{CG} are the translational (tangential direction) and angular velocities respectively of the CG particles, $n_{ij}^{\wedge}$ is the unit normal vector connecting the center of CG particles, r_c and r_o are the radii of the CG and original particles respectively.

The scaling law for the stiffness of the CG particle $k_{C,PP}$ was devised such that the elastic energy between the original and the CG particle can be matched, CG particle interactions were modelled by the Linear-spring model. The scaling law for the stiffness between the CG particles was defined as Equation (A-2)

$$k_{C,PP} = l^2 k_{series} = \frac{l^2}{2l-1} k_{O,PP}$$
 (A-2)

where k_{series} is the equivalent stiffness of the springs of the original particle system that the CG particle replaces and $k_{O,PP}$ is the stiffness of the original particle.

The viscous damping energies between the original and CG particles was maintained to derive the scaling law for the restitution coefficient as Equation (A-3).

$$e_{C,PP} = \sqrt{1 - \frac{1}{l}(1 - e_{O,PP}^2)} \quad (\text{A-3})$$

where $e_{C,PP}$ and $e_{O,PP}$ are the restitution coefficients of the CG and original particles respectively.

During the simulations the sliding friction coefficient was updated with the time steps as it depended on the angular and translation velocities of the coarse-grained particle whereas the stiffness and restitution coefficient of the CG particles depended only on the coarse-graining ratio and the values of these parameters of the original particles. The effectiveness of the coarse-graining model was evaluated by comparing the mixing, particle velocity and kinetic energy of the particles in the mixing drum with the results exhibiting that the CGSF method produced results similar to the original case whilst reducing the computational time significantly.

Bierwisch et al. (2009) developed scaling rules for coarse-grained particle systems. For maintaining the gravitational potential energy density of the original and coarse-grained systems, the volume fraction and solid density of the original and up-scaled particle should be equal such that $\rho_s = \rho'_s$. The velocities of the scaled and the original particles are considered equal in order to conserve the kinetic energy density of the system. The collisions per volume and time in the scaled system scaled with $(R/R')^3$ with the mass being scaled with $(R'/R)^3$. The energy dissipated per volume and time of the scaled system is equal to the original system value if the coefficient of restitution of the scaled system remains unaltered by coarse-graining. The normal and tangential forces in a binary collision are given by Equations (A-4) and (A-5) respectively where cohesion is modeled by JKR model with w defined as the cohesive energy per unit contact area.

$$m^* \ddot{\delta} = \frac{2}{3} \tilde{E} \sqrt{R^*} \delta^{3/2} + \sqrt{4\pi w \tilde{E}} R^{*3/4} \delta^{3/4} - \gamma_n \sqrt{R^*} \dot{\delta} \quad (\text{A-4})$$

Where m^* is the reduced mass defined as $m_i m_j / (m_i + m_j)$, δ is the deformation of two spherical particles i and j defined as difference between the sum of their radii, $R_i + R_j$ and the distance of their centers $|r_{ij}|$, effective particle radius $R^* = R_i R_j / (R_i + R_j)$, $\tilde{E} = E / (1 - \nu^2)$ where E and ν are the Young's modulus and Poisson's ratio of the particle respectively, γ_n is the damping parameter with $\dot{h}_{ij}$ representing the normal component of translational velocity, w is the work of adhesion per unit contact area.

$$m^* \ddot{\xi}_{ij} = -\min \left[\mu \left| \frac{2}{3} \tilde{E} \sqrt{R^*} \delta^{3/2} - \gamma_n \sqrt{R^*} \dot{\delta} \right|, k_t \sqrt{\frac{\delta}{R^*}} |\xi_{ij}| \right] \text{sgn}(\xi_{ij}) \quad (\text{A-5})$$

Where ξ_{ij} is the frictional spring elongation (accumulated tangential deformation), μ represents the coefficient of friction and k_t signifies the tangential spring constant.

Dimensional analysis of these equations leads to the dimensionless numbers Π_1 , Π_2 and Π_3 (Equations in (A-6)), if these numbers are kept constant, the energy dissipation rates are not affected by coarse-graining leading to the following scaling rules (Equations in (A-7)) which further help to establish that $w \propto R$, $\gamma_n \propto R$ and $k_t \propto R$. With the forces scaling quadratically, the coarse-graining technique was found to be adequate for reproducing the behaviour of the original system.

$$\Pi_1 = \frac{w}{R^* \tilde{E}}, \quad \Pi_2 = \frac{\gamma_n}{R^* \sqrt{\rho_s \tilde{E}}}, \quad \Pi_3 = \frac{k_t}{R^* \tilde{E}} \quad (\text{A-6})$$

$$\tilde{E} = \tilde{E}', \quad \frac{w}{R^*} = \frac{w'}{R^{*'}}, \quad \frac{\gamma_n}{R^*} = \frac{\gamma_n'}{R^{*'}}, \quad \frac{k_t}{R^*} = \frac{k_t'}{R^{*'}} \quad (\text{A-7})$$

where prime signifies the parameters of the scaled system and quantities without prime represent the parameters of the original system.

Lommen et al. (2019) derived scaling laws for the coarse-graining of particles for large scale bulk handling equipment. The coarse-graining or scaling factor l was defined such that the radius of the coarse-grain $R' = lR$, meaning that the original particles with radius R are replaced by coarse-grains of radius R' . To maintain the same potential energy density between the original and scaled system the particle density of the scaled particles should be equal to the particle density of the original particles together with maintaining the same void ratio. Similarly for maintaining the similarity for kinetic energy density, the velocities of the particles should be similar to the original system. It is assumed that the velocity of the coarse-grained particle represents the average velocity of the original particles that it replaces. For maintaining the energy losses experienced during collisions, the contact stiffness and damping also need to be scaled appropriately. The normal and tangential contact forces were determined by Equations (A-8) and (A-9) respectively.

$$f_n = k_n \delta^{3/2} n + c_n v_n \sqrt{\frac{3}{2} k_n \delta^{1/4}} \quad (\text{A-8})$$

$$f_t = k_t \sqrt{\delta} \delta_t t + c_t v_t \sqrt{k_t \delta^{1/4}} \quad (\text{A-9})$$

where v , k and c represent the particle velocity, stiffness coefficient and damping coefficient, respectively with the subscripts n and t signifying the normal and tangential direction values respectively of these parameters, δ is the normal overlap.

A scaling factor of 2 ($l = 2$) produces a coarse-grain which replaces eight of the original particles in the system, the stiffness and damping of the CG particle should be equal to the equivalent stiffness and damping of the group of unscaled or original particles.

The normal stiffness of the CG particle is $l^{0.5}$ times the original particle stiffness and the normal damping coefficient scales as $c'_n = l^{1.5}c_n$ with the assumption that $\delta_{series} = l\delta$. The tangential stiffness and the tangential damping factors scale similarly to their normal counterparts; this scaling of the normal and tangential parameters ensures that the energy losses due to the inelastic collisions are maintained for the original and CG systems. The scaling laws ensured that the potential energy, kinetic energy and the energy dissipation were maintained for the upscaled system. The simulations of the angle of repose test with the upscaled particles was able to produce results similar to the original system.

Thakur et al. (2016) investigated scaling of the DEM model parameters for quasi-static simulations of the uniaxial compression test. The linear mode of the EEPA model was used to model the particle interactions in this study, the scaling laws of the model parameters were investigated necessary to produce scale independent results. The particle stiffness k_n for the normal contact is given by Equation (A-10),

$$k_n = \frac{16}{15} \sqrt{R^*} E^* \left(\frac{15m^* v^2}{16E^* \sqrt{R^*}} \right)^{\frac{1}{5}} \quad (\text{A-10})$$

where m^* , R^* and E^* are the equivalent mass, radius and Young's modulus respectively and v is the particle impact velocity.

Expressing mass in terms of radius ($m^* = \rho^* \frac{4}{3} \pi R^{*3}$) transforms Equation (A-10) into Equation (A-11) where C is a constant.

$$k_n = CR^* \rho^{*\frac{1}{5}} E^{*\frac{4}{5}} \quad (\text{A-11})$$

The contact normal stiffness should scale linearly with particle radius according to this expression. EEPA model includes a constant pull-off force parameter f_o which can be used to represent forces such as the van der Waals forces. Various models used to represent the pull-off force were studied and it was found that it may scale linearly, quadratically or cubically with the particle radius. Simulations with the upscaled particles were conducted on cohesive and cohesionless systems to identify the appropriate scaling laws. It was found that the normal and tangential contact stiffnesses scaled linearly with the particle size and the adhesion force parameter f_o scaled quadratically with the particle size. Janda and Ooi (2016) also utilized the EEPA model to model cohesive solids and investigated the scaling laws for the contact parameters. It was found that the contact stiffness (for loading, unloading and adhesive branches) and the pull off force f_o scaled linearly and quadratically with the particle size respectively.

Tiscar et al. (2019) provided scaling laws for the coarse-graining of particles and evaluated the dynamic angle of repose from a rotating drum. Linear spring dashpot (LSD) model was selected to model the particle interactions. The study defined a scale factor λ (Equation (A-12)) which helped reduce the number of particles in the upscaled system to N_s from N_r (number of original particles in the system),

$$\lambda = \frac{N_r}{N_s} = \frac{V_s}{V_r} = \left(\frac{R_s}{R_r}\right)^3 \quad (\text{A-12})$$

Where V and R represent the volume and radius of the particle respectively and the subscripts r and s represent the real and scaled variables respectively.

In order to keep the mass of the coarse-grained particle equal to the original particle, the density of the CG particle ρ_s^s scales as $\rho_s^s/\rho_s^r = \lambda^{-1}$. The second scaling law was devised to maintain the average coordination number in the system, the increase in the size of the particle whilst keeping its mass constant will lead to a lower relative overlap (δ/R) which can decrease the coordination number. The relative overlap can be maintained constant if the stiffness coefficient is scaled as per Equation (A-13),

$$\frac{k_{n,s}}{k_{n,r}} = \frac{R_r}{R_s} = \lambda^{-1/3} \quad (\text{A-13})$$

b) Force-scaling laws

Widartiningsih et al. (2020) developed an integrated DEM-CFD method to simulate the powder die-filling systems. The solid phase was modelled through the coarse-graining technique in which the original particles are represented by coarse-grained (CG) particles. The coarse-grained particle is scaled through the coarse-grain ratio l , such that diameter of CG particle d_c is l times the original particle diameter d_o . The mass, momentum and energy of the CG and original particle systems are brought into agreement. The forces acting on the CG particle is calculated based on the energy equivalence of the CG particle and the group of original particles. It is assumed that all of the original particles in the CG particle have the same displacement and translational and angular velocities, binary collision between the CG particles is represented by l^3 pairs of binary collisions between the original particles taking place simultaneously. The velocity and displacement of the CG particle is modelled to match the average values of these parameters of the original particles, in order to satisfy this equivalence the contact force of the CG particle is represented such that the normal and tangential components are represented by the Equations (A-14) and (A-15) respectively.

$$f_{n_{CG}} = l^3(-k\delta_{CG} - \eta v_{n_{CG}}) \quad (\text{A-14})$$

$$f_{t_{CG}} = \begin{cases} l^3(-k\delta_{t_{CG}} - \eta v_{t_{CG}}) & \text{if } |f_{t_{CG}}| < \mu|f_{n_{CG}}| \\ -l^3\mu|f_{n_{CG}}|v_{t_{CG}}/|v_{t_{CG}}| & \text{if } |f_{t_{CG}}| \geq \mu|f_{n_{CG}}| \end{cases} \quad (\text{A-15})$$

where δ_{CG} and $\delta_{t_{CG}}$ are the normal and tangential overlap of the contacting CG particles respectively, k , η and μ are the stiffness, damping coefficient and coefficient of friction respectively and $v_{n_{CG}}$ and $v_{t_{CG}}$ signifies the translational velocity of CG particles in the normal and tangential directions respectively.

The fluid phase was modelled together with the solid phase to simulate die filling; the coarse-graining model was able to produce good qualitative and quantitative agreement with the original particle system. The

particle macroscopic flow pattern was suitably reproduced together with the mass transfer rate and air pressure change being in good agreement with the original particle system.

Washino et al. (2023) presented a Scaled-Up Particle (SUP) model for coarse-graining for simulating various particle processes. The SUP model categorises forces exerted on particles into two categories namely the inter-particle forces and the body forces. The particle size used in the simulations is scaled up by a factor l ($l = (m_s/m_o)^{1/3}$, m represents mass and the same forces and torques are maintained for the original and scaled-up systems acting on control volumes (CVs). The subscripts o and s represent the parameters of the original and scaled particles respectively. The rate of change of momentum in the CVs gives the forces acting on the CVs with this momentum change depending on a) the momentum exchange between the neighbouring CVs due to particle movement, b) the momentum exchange caused by the inter-particle forces between particles of different CVs and c) the momentum which the body forces impart to the particles in the CVs. The translational velocities of the original and scaled-up particles remain the same, the rotational velocity scales by $\omega_o = l \omega_s$ and particle overlap as $\delta_o = \delta_s/l$. In one of the previous studies (Hu et al., 2022) conducted by the authors of this study, the overlap and the separation distance of the scaled-up particles was suggested to scale with the factor l with the possibility for scaling the time step for the scaled-up particle by l times that of the original particle value. As the particle velocities of the original and scaled-up particles are similar, scaling for the momentum exchanged between CVs due to particle movement is not required. The inter-particle forces such as contact force would scale according to $F_{Is} = l^2 F_{Io}$ as the number of particle pairs interacting across the faces of the CVs are reduced by l^2 and so similarly the contact torque scales by $M_{Is} = l^2 M_{Io}$, the body forces are scaled by $F_{Bs} = l^3 F_{Bo}$ as the particles in the CVs in the scaled-up systems are reduced by l^3 . The subscripts I and B represent the inter-particle and body forces respectively. One of the differences between the SUP upscaling approach and the conventional approach is that in the former the particles are considered in a control volume and their interactions considered whereas in the latter the original particles are assumed to be replaced or enveloped by a single coarse-grained particle. The SUP model was validated for non-spherical and poly-dispersed particles for quasi-static and dynamic systems with the results showing the model was capable of replicating the original particle systems.

The following section puts forward some studies conducted in literature where the coarse graining or upscaling approach has been utilized, and the DEM model is calibrated and validated.

II. Coarse-graining calibration & validation studies

Coetzee (2019) broadly classified the approaches to determine the DEM model parameters as the “direct measuring approach” and the “bulk calibration approach”. In the former approach, the input parameters required for the DEM simulations are measured directly at the particle or contact level which can be difficult and even if these properties are calculated, it doesn’t fully ensure that the predicted bulk behaviour would be correct. The bulk calibration approach entails adjusting the parameter values required in the DEM

simulations so that the predicted bulk behaviour matches the behaviour ascertained by experiments. The physical or experimental response can depend on and vary with more than one of the parameter values and it is suggested a unique solution of the parameters predicting the bulk behaviour accurately can be obtained if two or more independent experiments are performed (Coetzee and Els, 2009). In modeling the various industrial applications, computational power constraints quite often make it difficult to model or simulate the size of the particles at the physical or actual scale. Coarse graining is a scale up approach in which the size of the particles is increased while that of the domain/geometry remains same thus a reduction in total number of particles in system/model is achieved which can offset the lack of computational power. It was also suggested to use the “bulk calibration approach” with the scaled particles. The study investigated the dynamic angle of repose of corn grains using a rotating steel drum and used these experimental results to calibrate the DEM model. The shapes of the corn grains could be broadly divided into three shapes as seen in *Figure A-1* with the average dimensions used in building a STL (stereolithography) model for each shape with these being used further to build multi-sphere clump templates to represent the real particles as precisely as possible. These three particle shapes were made use of in equal parts in the simulations with the scaling factors ranging from 1.1 to 9 being applied to the particle diameters. For the simulations used for calculating dynamic angle of repose, the linear contact model was used in the commercial DEM software package PFC3D. Parameters like the particle density, contact damping ratio, the particle-particle and the particle-wall coefficient of friction and the particle-particle contact stiffness were calibrated. The particles were scalable up to a factor of 4.0, with the larger scaling factors leading to inconsistent results. For the particle scaling factor ranging from 1.0-4.0, a unique group of calibrated parameters was identifiable meaning that the dynamic angle of repose (AOR) was not dependent on the scale in this range. It was inferred that the ratio of drum to particle diameter of about 25 or higher was able to predict or measure the dynamic AOR accurately.

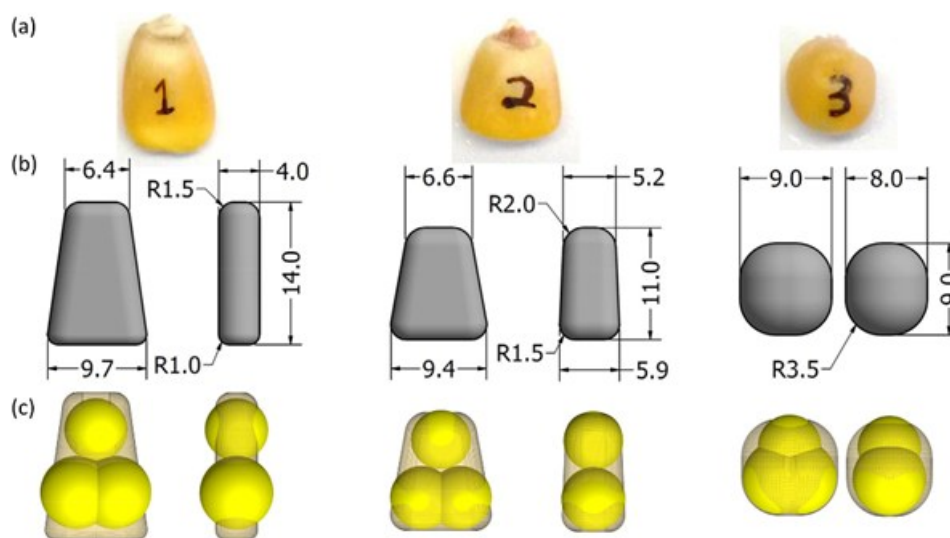


Figure A-1: (a) The identified particle shapes, (b) the CAD/STL models along with dimensions (mm), and (c) the equivalent multi-sphere models (Coetzee, 2019)

For the validation of the calibrated parameter conducted by Coetzee (2019), hopper discharge was modelled, and a flat-bottomed hopper was used to conduct the experiments. The calibrated parameter set accurately predicted the discharge rate for scale factors up to 1.3 for all variations of hopper openings. It indicated that the maximum scaling factor for the particle size was dependent on the application. The rate of discharge obtained from correlations given by Beverloo et al. (1961) and Olivares et al. (2018) were fitted and then compared to the DEM simulation and the experimental results. For a given particle size and three distinct hopper opening sizes, both the correlations were able to accurately predict the rate of discharge while scaling by a factor of two. Vertical velocity profiles were predicted by DEM simulations and were compared to the measurements of particle image velocimetry. A scale factor of less than 2.5 was obtained for all the hopper openings when the maximum rms error between the measured and predicted velocity profiles was set to 10%. The upscaled particles were successfully calibrated and led to a reduction in computing time. Computation time decreased by a factor of 3.2 when the particles were scaled up by a factor of 1.3 while still accurately predicting the discharge rate of hopper and the velocity field with a rms error lesser than 10%.

Zhang et al. (2020) conducted a study on volume-based scaling of rice particles in the DEM simulations and calibrated the input parameters used in the simulations. The rice grain was approximated as an ellipsoidal particle definable by its length, width and depth. A digital calliper was used to measure these three dimensions of about one hundred single rice grains with the mean values further computed. The ellipsoid thickness was approximated as the mean of the rice grain width and depth. Aspect ratio of the rice grains was defined as the ratio of its length (major axis) to its thickness (minor axis). The produced DEM particle had a volume thrice the original value while maintaining the original aspect ratio. The rice particles were formed by clumping seven spheres as seen in Figure A-2.

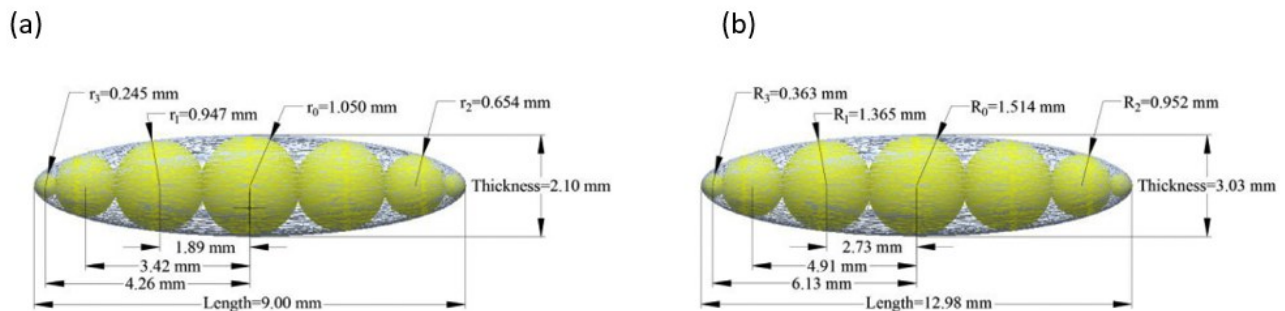


Figure A-2: (a) Multi-sphere DEM rice particle (unscaled), and (b) Multi-sphere DEM rice particle (scaled up) (Zhang et al., 2020)

The angle of repose test and hopper flow were simulated by using EDEM (DEM Solutions Ltd., Edinburgh, UK), with the Hertz-Mindlin (no-slip) (HM) contact model used for particle-particle and particle-geometry calculations. A bulk angle of repose (AOR) box with a sliding gate fabricated from acrylic material was used to

calculate the bulk AOR of rice grains and this test was then simulated. Scaled up rice particles totaling a value of 8400 were used in the DEM simulation. A design of experimentation or DOE study was used to determine the optimal combination of parameters that significantly influenced the angle of repose values. The coefficient of static friction between rice particles, the rice-rice coefficient of restitution and the rice-rice coefficient of rolling friction were determined to be strongly influencing the bulk AOR values of the rice grains. Simulations were run to calibrate these parameters and optimal values of these parameters were obtained which was able to predict the AOR value with a very low error hence emphasizing the successful calibration of the DEM model that used up-scaled particles. The use of scaled-up particles led to a high figure of 76% in computational savings when compared to unscaled particles used in simulations to calculate AOR.

Zhang et al. (2020) validated the calibrated DEM parameters of the upscaled rice particles by comparing the simulated results with the experimental hopper discharge flow rate value and the one obtained from the analytical Equation (A-16) from Beverloo et al. (1961). The optimal DEM parameters obtained from calibration of the upscaled particles were used to simulate rice discharge through the hopper outlet.

$$W = C_b \rho_b \sqrt{g} (D_h - k_b d)^{2.5} \quad (\text{A-16})$$

where, W = average mass discharge rate through the hopper orifice, ρ_b = material bulk density, D_h = orifice hydraulic diameter, d = diameter of particle, g = acceleration due to gravity, C_b and k_b are dimensionless coefficients.

The solids density (Equation (A-17)) of the scaled up particle was adjusted prior to the calculations of flow of rice from the hoppers.

$$\rho_s^s = \frac{3m}{V_s} + (\epsilon_s - \epsilon_r) \rho_s \quad (\text{A-17})$$

Where, ρ_s^s = solids density of the multi-sphere scaled-up DEM particle, the factor 3 represents the volume scaling factor, m is the mean mass of a single rice grain, V_s = volume of multi-sphere scaled-up DEM particle, ϵ_r = porosity of rice grains, and ϵ_s = porosity of the multi-sphere scaled-up DEM rice particle when its solids density is equal to the solids density ρ_s of rice grain.

The discharge flow rate predicted by the DEM simulations, the experiments and the analytical solution in accordance with the equation by Beverloo et al. (1961) showed good agreement with very low error values and confirmed the accuracy of the particle upscaling technique. Computational savings of 60% were seen when the upscaled rice particle was used in the simulations for the hopper flow when compared to the unscaled particle.

Lommen et al. (2019) investigated upscaling of particles for bulk handling equipment where a very large volume of bulk material is handled which makes the computational costs very high. A scaling factor l was

introduced by which the coarse grain or upscaled particle radius R' could be represented by multiplying this factor l with its original radius R , such that $R' = lR$.

The spring damper system in normal direction of the coarse grain with the scaling factor $l = 2$ and the group of original particles can be seen in Figure A-3. The coarse grain replaces eight original particles when the coarse grain doubles in size. To preserve the contact character the stiffness k'_n and the damping c'_n of the coarse grain should be equivalent to the equivalent stiffness k_{eq} and damping c_{eq} of the original particles.

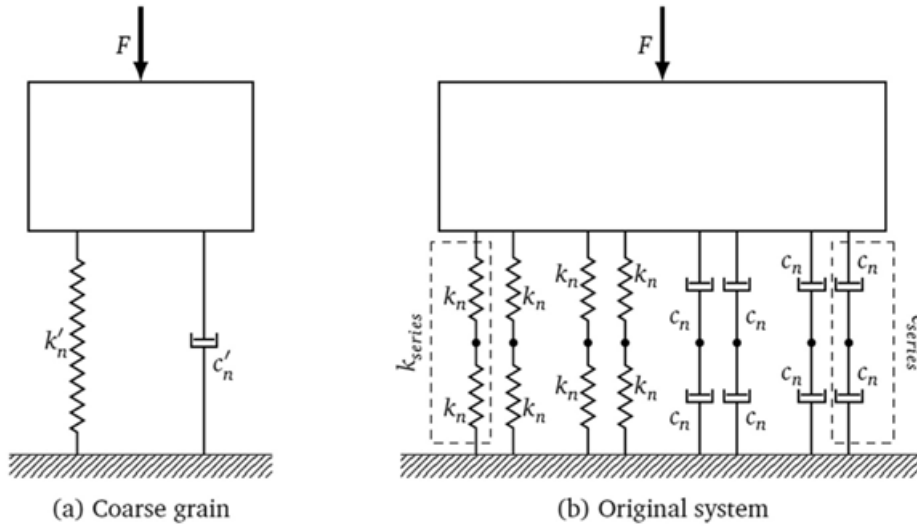


Figure A-3: (a) Coarse grain contact with scaling factor $s=2$, and (b) Equivalent contact of original particle group (Lommen et al., 2019)

Experimental tests such as ledge and cone tests for calculating the angle of repose (AOR) and the penetration test were conducted on the iron ore pellets which were approximately spherical in shape. The Hertz-Mindlin model with restricted angular movements and the Hertz-Mindlin model with rolling enabled were used as the two contact models in the study. The input DEM parameters of particles such as shear modulus, Poisson's ratio, density, coefficient of restitution, static friction, wall friction were used in the restricted angular movement model with rolling friction between particle-particle and rolling friction between wall and particle added for the rolling enabled model. Both these models were calibrated by minimizing the error between the simulated and experimental values of AOR and a set of calibrated parameters were obtained. The effect of coarse graining on AOR and the penetration test were investigated as the latter is key to model the material-equipment interaction during grab unloading. Scaling factor l of 2 and 4 was used to gauge the impact on the AOR simulations, the scaling factor did not significantly impact the simulations and agreed with the unscaled particle results. Scaling factor l ranging from 2 to 5 was used for the penetration studies with the sliding process being scaled more consistently than the penetration process. This was attributed to the fact that for the coarser or upscaled grains it is harder to move as they required to move to the sides prior to the tool passing which leads to elevated normal and frictional forces experienced on the tips. The wall friction coefficient at the tip of penetration tool was adjusted and this produced consistent results when applied to

simulate the industrial scale grab unloader. The coarse graining scheme when used with $l = 5$ and no rotation reduced the simulation time for large scale grab from an estimated 10,000 hours to approximately 18 hours.

Ajmal et al. (2020) used standard draw down experiment to calibrate the parameters for cohesive bulk materials. The authors used the Simplified Johnson Kendall Roberts (SJKR) model and the full Johnson Kendall Roberts (JKR) model with an adhesive hysteretic rolling resistance (AdHys) model to conduct a comparative calibration using the draw down experiment. Hertz-Mindlin contact model was used for the DEM simulations with the JKR type models chosen to simulate the wet sand. A draw down test apparatus (Figure A-4) was used to conduct the draw down test of sand with 10% moisture content. The apparatus consists of two boxes of Perspex. The top box allows material to flow through it by a gate located on its bottom. The opening size of the gate could be varied, and flaps installed beneath the gate could be opened instantaneously to allow material flow into the bottom box. The top box is placed on load cells so as to measure the flow rate of the material flowing through it. A heap is formed in the lower box which indicates the angle of repose of the material while a shear angle forms in the top box as the material flows out from it. The angle of repose β_{DD} and the shear angle Φ_{DD} are measured through image analysis. The test allows to measure the angle of repose, shear angle and the mass flow rate of the material all in a single experiment.

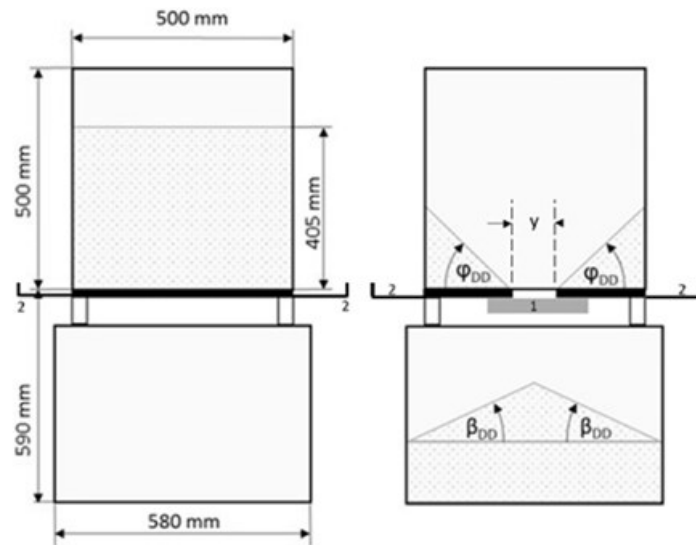


Figure A-4: Draw down apparatus schematic where y is opening size, 1 represents the instantaneous opening flap, and 2 represents the moveable flaps (Ajmal et al., 2020)

It was estimated that the simulation would require 7-8 million particles which necessitated the requirement of upscaling. The particle size distribution was upscaled by a factor of 10. The simulations were performed with approximately 95,000 particles with variation of the opening size of the top box. The particle-particle friction, coefficient of rolling friction and the cohesion energy density (CED) were the parameters calibrated for the SJKR model while particle-particle friction, adhesion hysteresis and surface energy density (SED) were calibrated for the JKR AdHys model. It was found that the SJKR model over predicted the cohesive forces seen by the higher AOR simulation values with the possibility that the breakage of agglomerates was not being

achieved in the model. The AOR values calculated by the JKR AdHys model were in better agreement with the experimental values although the JKR AdHys simulations took 25-30% longer than the SJKR simulations.

Roessler and Katterfeld (2018) emphasized that for different applications, different calibration tests are required which consider the flow regime of the simulation being calibrated. The study conducted focused on rapid flow regimes of non-cohesive powder particles under relatively lower consolidation stress states. Particle scale-up approaches can be broadly divided into 1. exact scaling where the number of particles is kept same and the size of the particle and container are both upscaled, 2. coarse graining where the number of particles is reduced whilst the container size is constant and 3. scalping where particles of certain sizes are removed from the simulations while the container size is kept constant. The coarse graining and scalping approach can require the dimensions of the test rig/equipment to be scaled up so that the test rig size can adapt to the upscaled particles. Need for a size invariant calibration test was felt where results were independent or free of the experiment size. Experiments conducted to calculate AOR on dry sand with the lifting cylinder apparatus showed that the AOR had good reproducibility. At slow or low lifting velocities it was almost independent of the cylinder size, thus it was concluded that the dimensions of the cylinder could be scaled up to fit the upscaled particles. The scalping technique was used to eliminate particles having low sizes, where mass of the particle fractions $> 1\text{mm}$ were considered similar to real specimen and the mass of particle fractions $< 1\text{ mm}$ were merged to the mass of the particle fraction of 1 mm and the average particle size obtained was 1.28mm . The particles were then upscaled up to scaling factors of 12.5 (average particle size of 16mm) depending on the cylinder size to be used in the simulations such that 7500 particles were simulated, Hertz-Mindlin (no slip) model was used to model the contacts. Coefficient of particle-particle friction and coefficient of rolling friction were found to be mainly influencing the AOR values. As the AOR values were found to be independent of the cylinder size for a given lifting velocity in the experiments, AOR values at three different lifting velocities were considered for the calibration purpose. The results showed that the calibration process leads to different choice of parameters depending on the lifting velocity and size invariant results were obtained for the material in static stress state where low lifting velocities were applied. To overcome this shortcoming, shear-box test experiments to calculate the AOR were conducted and then the experiment was simulated. The shear box test provided AOR results which were invariant from the test rig size and could be used for dynamic, fast flowing stress state.

Roessler and Katterfeld (2019) examined the applicability of a simple angle of repose test with lifting of a hollow cylinder for calibrating the cohesive bulk material. Angle of repose for dry sand mixed with water was measured by a lifting cylinder test by keeping the lifting velocity constant and changing the cylinder size, the AOR values varied over a large range of 40.4° to 84.3° due to which it was inferred that the AOR is not suitable to quantitatively characterize and calibrate the DEM parameters. The flow of the wet sand was divided into four phases as seen in Figure A-5, 1. formation of stable bulk material column without bending ($h < 80\text{ mm}$),

2. increasing curvature of the bulk material column and formation of cracks ($h \approx 120$ mm), 3. maximum bending condition of the column ($h \approx 140$ mm) and 4. irregular spreading of material ($h \approx 160$ mm). Experiments indicated good reproducibility of these three phases i.e., build-up of stable bulk material column ($h < 80$ mm), convex bending of the column ($h < 140$ mm) and collapse of the column ($h \approx 160$ mm). These phases were found to be independent of cylinder size and the lifting velocity of the experiments conducted.

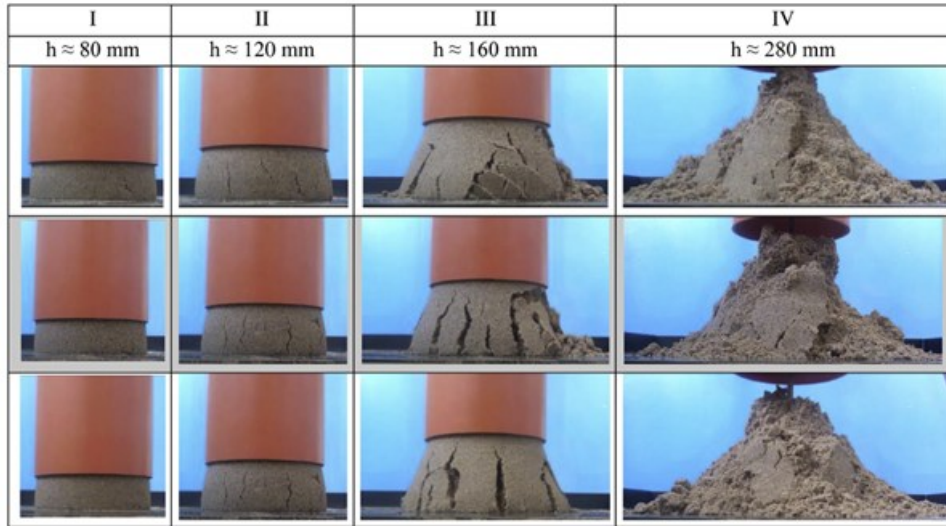


Figure A-5: Phases of the bulk-material column (Roessler and Katterfeld, 2019)

For the DEM simulations Hertz-Mindlin (no slip) was used as the contact model and simplified JKR model utilized for representing the cohesive contact. The particles were upscaled to particle diameters ranging from 5.5 to 12.7 mm. The particle-particle friction, coefficient of rolling friction and the cohesion energy density (CED) were the parameters that were calibrated.

Pachón-Morales et al. (2019) modelled the flow of cohesive lignocellulosic biomass powders with poplar (*Populus euro-americana* 'Koster') selected to represent the biomass. The poplar powder was obtained after milling and was further differentiated into two sieving cuts representing the fine and coarse particles with the PSD of both falling largely in the sub-millimeter range. To gauge the bulk behaviour of the powder and for calibrating the DEM simulation parameters, angle of repose, bulk density and rectangular container or shear box tests were conducted. The rectangular container test is equipped with adjustable walls so that it adapts to the available volume of the sample. Cabin lid of the container is lifted after the sample is filled in the container with the retainment ratio calculated as the ratio of the mass remaining in the container to that of the originally filled. For the DEM simulations Hertz-Mindlin contact model with elastic-plastic spring-dashpot or EPSD2 as the rolling friction model and simplified Johnson Kendall Roberts (SJKR) cohesion model were used. The powder was modelled by the multi sphere method with particles represented by three spheres oriented along a longitudinal axis and overlapping each other. The particles were scaled up by a factor of 4.

The parameters that were calibrated included the cohesion energy density (CED) and the coefficients of sliding and rolling friction which were optimized by using a multi-objective evolutionary algorithm.

The DEM parameters calibrated in the study conducted by Pachón-Morales et al. (2019) were validated by simulating the ring shear test and comparing the results with the experiments. The geometry of the shear tester was also scaled up by a factor of 4. The simulations were conducted by firstly a pre-shear step followed by shearing at a stress lower than the pre-shear step. The shear stresses obtained at these two steps were computed by simulations and compared to the experimental values. The simulation and experimental results generally agreed with some variation and underestimation of the shear stress values seen in some samples. The authors stressed that the calibration of the parameters and the experiments used for it should be cognizant of the actual simulated process. It was suggested that the calibration of parameters achieved by angle of repose, bulk density and the shear box tests may not be sufficient for simulating materials under a consolidated state as seen in case of shear testing.

Sakai et al. (2014) verified and validated the coarse grain model in three dimensional (3-D) fluidized beds. The verification and validation tests are performed for both the fluidized and the fixed bed state by simulating non cohesive particles in a 3-D fluidized bed. A coarse grain ratio l was introduced where the coarse grain particle size was l times larger than the original particle. A particle size of $150\ \mu m$ if converted or upscaled to a coarse grain/particle of $300\ \mu m$ led to a reduction in total number of particles in the simulation from 540,000 to 67,500 with coarse grain ratio l of 3 further increasing the size of particles in the system to $450\ \mu m$ and reducing the number of particles to 20,000. The contact force acting on the coarse grain particle was evaluated using springs, a friction slider and dash-pots for the solid phase and the governing equations used for the gas phase were the fluid continuity and the Navier-Stokes equations. The coarse grain model was verified by simulating the particle system in a 3-D fluidized bed with the coarse graining ratio l set as 2.0 to 3.0. The particle simulated was a glass bead with suitable values of density, coefficient of friction, coefficient of restitution and stiffness set for the simulations, the gas density and viscosity values were also set. The macroscopic flow properties such as bubble velocity, bed height and pressure drop were regarded to be equivalent to the coarse grain model, the coarse grain model was able to simulate the original system or the simulations with the unscaled or original particle diameter by using lesser number of particles than used in the original simulation. The adequacy or suitability of the coarse grain model for the fixed state was also shown. In order to further establish the suitability of the model validation, tests were conducted for the fluidized and fixed state with the help of apparatus as shown in Figure A-6.

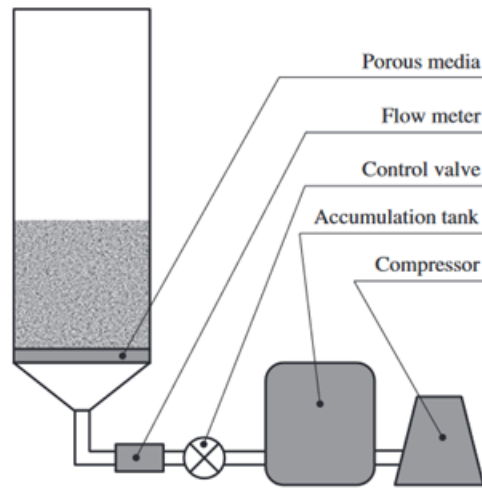


Figure A-6: Experimental device schematic (Sakai et al., 2014).

The coarse grain ratio l was taken as 5 meaning the particle size increased from $150\ \mu\text{m}$ to $750\ \mu\text{m}$ while the total particles in the system reduced from 28,800,000 to 230,400 particles. The weight of the powder bed corresponding to 28,800,000 particles was 0.127 kg for the experiments, and this same value of 0.127 kg was used for the simulations with the 230,400 upscaled particles. The macroscopic flow properties namely pressure drop and bed height were measured experimentally for the fluidized and the fixed state and these values were in quantitative agreement with the corresponding values obtained from simulations. In the fluidized state, qualitative agreement between the experimental and the simulated results was seen by comparing the appearance of the bed. The coarse grain model was found applicable to a real or actual 3-D fluidized bed system.

Appendix B

Script for automating the shear test simulation in EDEM

(requires EDEM's post-processing Python library EDEMPy for execution)

The script compares the target value of the set parameter (shearing lid torque for example) with the value read from the csv file (written concurrently when the simulation is running). The script “kills” or stops the simulation once the value of the parameter in the simulation (last value read from csv file) meets the target set by the user in the script.

Parts of the script were used for simulations at critical consolidation; the shearing of the sample for critically consolidating the sample was carried out for fixed time durations, with the script's “stop” logic being used during the shear reversal step for stopping the trough counter rotation once shear stress drops to zero.

The “stop” logic used in this script was used also for simulating the 75% partial consolidation tests, with the shearing of the sample stopped once the pre-set shear stress (corresponding to 75% partial consolidation) was reached. and for stopping the counter rotation of the sample once the shear stress drops to zero.

The length of the script can be significantly reduced by defining the repeatable functions at the start of the script and then reusing them wherever necessary in the script.

```
import subprocess
import time
import numpy as np
import os
import math
import statistics
from edempy import Deck

lastRecordedTimeValue = "orig1"
lastRecordedTorqueValue = "orig2"
lastRecordedTime = "orig3"
lastRecordedTorque = "orig4"
lastRecordedTime_CR2 = "orig5"
lastRecordedTorque_CR2 = "orig6"
lastRecordedTime_CR3 = "orig7"
lastRecordedTorque_CR3 = "orig8"
lastRecordedTime_CR4 = "orig9"
lastRecordedTorque_CR4 = "orig10"
lastRecordedTime_CR5 = "orig11"
lastRecordedTorque_CR5 = "orig12"
lastRecordedTime_CR6 = "orig13"
lastRecordedTorque_CR6 = "orig14"
lastRecordedTime_CR7 = "orig15"
lastRecordedTorque_CR7 = "orig16"
lastRecordedTime_CR8 = "orig17"
lastRecordedTorque_CR8 = "orig18"
average_torque_PS1 = "orig19"
standard_deviation_PS1 = "orig20"
average_torque_PS2 = "orig21"
standard_deviation_PS2 = "orig22"
average_torque_PS3 = "orig23"
standard_deviation_PS3 = "orig24"
average_torque_PS4 = "orig25"
standard_deviation_PS4 = "orig26"

def start():
    subprocess.Popen('run.bat')

def post_processing_script():
    subprocess.Popen('script_1.5kg.bat')

def kill(deck_name):
    f = open(deck_name+".stop", "x")
    f.close()

def open_file(file_name, frequency):
    while True:
```

```

try:
    file = open(file_name, "r")
    break
except:
    time.sleep(1/frequency)
    continue
return file

def iterate(file, frequency):
    global lastRecordedTimeValue
    global lastRecordedTorqueValue
    global average_torque_PS1
    global standard_deviation_PS1
    print(lastRecordedTimeValue)
    print(lastRecordedTorqueValue)
    N=2
    data = []*N
    timestep = 0
    while True:
        lines = file.readlines()
        if not lines:
            time.sleep(1/frequency)
            continue
        else:
            totalLines = len(lines)
            #print(totalLines)
            lastline = lines[totalLines-1]
            lastRecordedTimeValue = lastline.split(',')[0]
            lastRecordedTorqueValue = lastline.split(',')[1]
            print(lastRecordedTimeValue)
            print(lastRecordedTorqueValue)
            lines = file.readlines()
            last_100_lines_PS1 = lines[-100:]
            average_torque_PS1 = statistics.mean(float(line.split(',')[1]) for line in last_100_lines_PS1)
            standard_deviation_PS1 = statistics.stdev(float(line.split(',')[1]) for line in last_100_lines_PS1)
            dt = lastRecordedTimeValue
            if dt != timestep and len(data)>0:
                print("Entered If Block")
                #Store new timestep value
                timestep = dt
                #Convert accumulated data into numpy array
                values = np.genfromtxt(data, delimiter=',')
                #Check stop criterion
                if stop(values) == True:
                    break
            N=2
            data = []*N
            data.extend(lines)
            print("Appended Data")

def stop(values):
    global lastRecordedTorqueValue
    global average_torque_PS1
    global standard_deviation_PS1
    torque_lower_limit = -0.542432
    torque_upper_limit = -0.505657
    stdev_lower_limit = 0.10
    stdev_upper_limit = 0.20
    print("Attempting Comparison")
    print(average_torque_PS1)
    if torque_lower_limit <= average_torque_PS1 <= torque_upper_limit and stdev_lower_limit <= standard_deviation_PS1 <= stdev_upper_limit:
        return True
    else:
        return False

# 1st counter rotation start
def kill_CR2(deck_name):
    f2 = open(deck_name+".stop", "x")
    f2.close()

def open_file(file_name_2, frequency):
    while True:
        try:
            file2 = open(file_name_2, "r")
            break
        except:
            time.sleep(1/frequency)
            continue
    return file2

def iterate_CR2(file2, frequency):
    global lastRecordedTime_CR2
    global lastRecordedTorque_CR2

```

```

print(lastRecordedTime_CR2)
print(lastRecordedTorque_CR2)
N=2
data2 = []*N
timestep2 = 0
while True:
    lines2 = file2.readlines()
    if not lines2:
        time.sleep(1/frequency)
        continue
    else:
        totalLines2 = len(lines2)
        #print(totalLines1)
        lastline2 = lines2[totalLines2-1]
        lastRecordedTime_CR2 = lastline2.split(',')[0]
        lastRecordedTorque_CR2 = lastline2.split(',')[1]
        print(lastRecordedTime_CR2)
        print(lastRecordedTorque_CR2)
        dt2 = lastRecordedTime_CR2
        if dt2 != timestep2 and len(data2)>0:
            print("Entered If Block")
            timestep2 = dt2
            values2 = np.genfromtxt(data2, delimiter=',')
            if stop_CR2(values2) == True:
                break
            N=2
            data2 = []*N
        data2.extend(lines2)
        print("Appended Data")

def stop_CR2(values2):
    global lastRecordedTorque_CR2
    break_value_CR2 = 0.00010
    print("Attempting Comparison")
    print(lastRecordedTorque_CR2)
    if break_value_CR2 <= float(lastRecordedTorque_CR2):
        return True
    else:
        return False

# 1st counter rotation end

# 2nd pre-shear

def kill_CR3(deck_name):
    f3 = open(deck_name+".stop", "x")
    f3.close()

def open_file(file_name_3, frequency):
    while True:
        try:
            file3 = open(file_name_3,"r")
            break
        except:
            time.sleep(1/frequency)
            continue
    return file3

def iterate_CR3(file3, frequency):
    global lastRecordedTime_CR3
    global lastRecordedTorque_CR3
    global average_torque_PS2
    global standard_deviation_PS2
    print(lastRecordedTime_CR3)
    print(lastRecordedTorque_CR3)
    N=2
    data3 = []*N
    timestep3 = 0
    while True:
        lines3 = file3.readlines()
        if not lines3:
            time.sleep(1/frequency)
            continue
        else:
            totalLines3 = len(lines3)
            lastline3 = lines3[totalLines3-1]
            lastRecordedTime_CR3 = lastline3.split(',')[0]
            lastRecordedTorque_CR3 = lastline3.split(',')[1]
            print(lastRecordedTime_CR3)
            print(lastRecordedTorque_CR3)
            lines3 = file3.readlines()
            last_100_lines_PS2 = lines3[-100:]
            average_torque_PS2 = statistics.mean(float(line.split(',')[1]) for line in last_100_lines_PS2)

```

```

standard_deviation_PS2 = statistics.stdev(float(line.split(',')[1]) for line in last_100_lines_PS2)
dt3 = lastRecordedTime_CR3
if dt3 != timestep3 and len(data3)>0:
    print("Entered If Block")
    timestep3 = dt3
    values3 = np.genfromtxt(data3, delimiter=',')
    #Check stop criterion
    if stop_CR3(values3) == True:
        break
    N=2
    data3 = []*N
    data3.extend(lines3)
    print("Appended Data")

def stop_CR3(values3):
    global lastRecordedTorque_CR3
    global average_torque_PS2
    global standard_deviation_PS2
    torque_lower_limit = -0.542432
    torque_upper_limit = -0.505657
    stdev_lower_limit = 0.10
    stdev_upper_limit = 0.20
    print("Attempting Comparison")
    print(average_torque_PS2)
    if torque_lower_limit <= average_torque_PS2 <= torque_upper_limit and stdev_lower_limit <= standard_deviation_PS2 <= stdev_upper_limit:
        return True
    else:
        return False

# 2nd pre-shear end

# 2nd counter rotation start

def kill_CR4(deck_name):
    f4 = open(deck_name+".stop", "x")
    f4.close()

def open_file(file_name_4, frequency):
    while True:
        try:
            file4 = open(file_name_4,"r")
            break
        except:
            time.sleep(1/frequency)
            continue
    return file4

def iterate_CR4(file4, frequency):
    global lastRecordedTime_CR4
    global lastRecordedTorque_CR4
    print(lastRecordedTime_CR4)
    print(lastRecordedTorque_CR4)
    N=2
    data4 = []*N
    timestep4 = 0
    while True:
        lines4 = file4.readlines()
        if not lines4:
            time.sleep(1/frequency)
            continue
        else:
            totalLines4 = len(lines4)
            lastline4 = lines4[totalLines4-1]
            lastRecordedTime_CR4 = lastline4.split(',')[0]
            lastRecordedTorque_CR4 = lastline4.split(',')[1]
            print(lastRecordedTime_CR4)
            print(lastRecordedTorque_CR4)
            dt4 = lastRecordedTime_CR4
            if dt4 != timestep4 and len(data4)>0:
                print("Entered If Block")
                timestep4 = dt4
                values4 = np.genfromtxt(data4, delimiter=',')
                if stop_CR4(values4) == True:
                    break
                N=2
                data4 = []*N
                data4.extend(lines4)
                print("Appended Data")

def stop_CR4(values4):
    global lastRecordedTorque_CR4
    break_value_CR4 = 0.00010
    print("Attempting Comparison")

```

```

print(lastRecordedTorque_CR4)
if break_value_CR4 <= float(lastRecordedTorque_CR4):
    return True
else:
    return False

# 2nd counter rotation end

# 3rd pre-shear

def kill_CR5(deck_name):
    f5 = open(deck_name+".stop", "x")
    f5.close()

def open_file(file_name_5, frequency):
    while True:
        try:
            file5 = open(file_name_5, "r")
            break
        except:
            time.sleep(1/frequency)
            continue
    return file5

def iterate_CR5(file5, frequency):
    global lastRecordedTime_CR5
    global lastRecordedTorque_CR5
    global average_torque_PS3
    global standard_deviation_PS3
    print(lastRecordedTime_CR5)
    print(lastRecordedTorque_CR5)
    N=2
    data5 = []*N
    timestep5 = 0
    while True:
        lines5 = file5.readlines()
        if not lines5:
            time.sleep(1/frequency)
            continue
        else:
            totalLines5 = len(lines5)
            lastline5 = lines5[totalLines5-1]
            lastRecordedTime_CR5 = lastline5.split(',')[0]
            lastRecordedTorque_CR5 = lastline5.split(',')[1]
            print(lastRecordedTime_CR5)
            print(lastRecordedTorque_CR5)
            lines5 = file5.readlines()
            last_100_lines_PS3 = lines5[-100:]
            average_torque_PS3 = statistics.mean(float(line.split(',')[1]) for line in last_100_lines_PS3)
            standard_deviation_PS3 = statistics.stdev(float(line.split(',')[1]) for line in last_100_lines_PS3)
            dt5 = lastRecordedTime_CR5
            if dt5 != timestep5 and len(data5)>0:
                print("Entered If Block")
                timestep5 = dt5
                values5 = np.genfromtxt(data5, delimiter=',')
                if stop_CR5(values5) == True:
                    break
            N=2
            data5 = []*N
            data5.extend(lines5)
            print("Appended Data")

def stop_CR5(values5):
    global lastRecordedTorque_CR5
    global average_torque_PS3
    global standard_deviation_PS3
    torque_lower_limit = -0.542432
    torque_upper_limit = -0.505657
    stdev_lower_limit = 0.10
    stdev_upper_limit = 0.20
    print("Attempting Comparison")
    print(average_torque_PS3)
    if torque_lower_limit <= average_torque_PS3 <= torque_upper_limit and stdev_lower_limit <= standard_deviation_PS3 <= stdev_upper_limit:
        return True
    else:
        return False

# 3rd pre-shear end

# 3rd counter rotation start

def kill_CR6(deck_name):
    f6 = open(deck_name+".stop", "x")

```

```

f6.close()
def open_file(file_name_6, frequency):
    while True:
        try:
            file6 = open(file_name_6, "r")
            break
        except:
            time.sleep(1/frequency)
            continue
    return file6

def iterate_CR6(file6, frequency):
    global lastRecordedTime_CR6
    global lastRecordedTorque_CR6
    print(lastRecordedTime_CR6)
    print(lastRecordedTorque_CR6)
    N=2
    data6 = []*N
    timestep6 = 0
    while True:
        lines6 = file6.readlines()
        if not lines6:
            time.sleep(1/frequency)
            continue
        else:
            totalLines6 = len(lines6)
            lastline6 = lines6[totalLines6-1]
            lastRecordedTime_CR6 = lastline6.split(',')[0]
            lastRecordedTorque_CR6 = lastline6.split(',')[1]
            print(lastRecordedTime_CR6)
            print(lastRecordedTorque_CR6)
            dt6 = lastRecordedTime_CR6
            if dt6 != timestep6 and len(data6)>0:
                print("Entered If Block")
                timestep6 = dt6
                values6 = np.genfromtxt(data6, delimiter=',')
                if stop_CR6(values6) == True:
                    break
            N=2
            data6 = []*N
            data6.extend(lines6)
            print("Appended Data")

def stop_CR6(values6):
    global lastRecordedTorque_CR6
    break_value_CR6 = 0.00010
    print("Attempting Comparison")
    print(lastRecordedTorque_CR6)
    if break_value_CR6 <= float(lastRecordedTorque_CR6):
        return True
    else:
        return False

# 3rd counter rotation end

# 4th pre-shear

def kill_CR7(deck_name):
    f7 = open(deck_name+".stop", "x")
    f7.close()

def open_file(file_name_7, frequency):
    while True:
        try:
            file7 = open(file_name_7, "r")
            break
        except:
            time.sleep(1/frequency)
            continue
    return file7

def iterate_CR7(file7, frequency):
    global lastRecordedTime_CR7
    global lastRecordedTorque_CR7
    global average_torque_PS4
    global standard_deviation_PS4
    print(lastRecordedTime_CR7)
    print(lastRecordedTorque_CR7)
    N=2
    data7 = []*N
    timestep7 = 0
    while True:
        lines7 = file7.readlines()

```

```

if not lines7:
    time.sleep(1/frequency)
    continue
else:
    totalLines7 = len(lines7)
    lastline7 = lines7[totalLines7-1]
    lastRecordedTime_CR7 = lastline7.split(',')[0]
    lastRecordedTorque_CR7 = lastline7.split(',')[1]
    print(lastRecordedTime_CR7)
    print(lastRecordedTorque_CR7)
    lines7 = file7.readlines()
    last_100_lines_PS4 = lines7[-100:]
    average_torque_PS4 = statistics.mean(float(line.split(',')[1]) for line in last_100_lines_PS4)
    standard_deviation_PS4 = statistics.stdev(float(line.split(',')[1]) for line in last_100_lines_PS4)
    dt7 = lastRecordedTime_CR7
    if dt7 != timestep7 and len(data7)>0:
        print("Entered If Block")
        timestep7 = dt7
        values7 = np.genfromtxt(data7, delimiter=',')
        if stop_CR7(values7) == True:
            break
        N=2
        data7 = []*N
    data7.extend(lines7)
    print("Appended Data")

def stop_CR7(values7):
    global lastRecordedTorque_CR7
    global average_torque_PS4
    global standard_deviation_PS4
    torque_lower_limit = -0.542432
    torque_upper_limit = -0.505657
    stdev_lower_limit = 0.10
    stdev_upper_limit = 0.20
    print("Attempting Comparison")
    print(average_torque_PS4)
    if torque_lower_limit <= average_torque_PS4 <= torque_upper_limit and stdev_lower_limit <= standard_deviation_PS4 <= stdev_upper_limit:
        return True
    else:
        return False

# 4th pre-shear end

# 4th counter rotation start

def kill_CR8(deck_name):
    f8 = open(deck_name+".stop", "x")
    f8.close()

def open_file(file_name_8, frequency):
    while True:
        try:
            file8 = open(file_name_8, "r")
            break
        except:
            time.sleep(1/frequency)
            continue
    return file8

def iterate_CR8(file8, frequency):
    global lastRecordedTime_CR8
    global lastRecordedTorque_CR8
    print(lastRecordedTime_CR8)
    print(lastRecordedTorque_CR8)
    N=2
    data8 = []*N
    timestep8 = 0
    while True:
        lines8 = file8.readlines()
        if not lines8:
            time.sleep(1/frequency)
            continue
        else:
            totalLines8 = len(lines8)
            lastline8 = lines8[totalLines8-1]
            lastRecordedTime_CR8 = lastline8.split(',')[0]
            lastRecordedTorque_CR8 = lastline8.split(',')[1]
            print(lastRecordedTime_CR8)
            print(lastRecordedTorque_CR8)
            dt8 = lastRecordedTime_CR8
            if dt8 != timestep8 and len(data8)>0:
                print("Entered If Block")
                timestep8 = dt8

```



```

        values8 = np.genfromtxt(data8, delimiter=',')
        if stop_CR8(values8) == True:
            break
        N=2
        data8 = []*N
        data8.extend(lines8)
        print("Appended Data")

def stop_CR8(values8):
    global lastRecordedTorque_CR8
    break_value_CR8 = 0.00010
    print("Attempting Comparison")
    print(lastRecordedTorque_CR8)
    if break_value_CR8 <= float(lastRecordedTorque_CR8):
        return True
    else:
        return False

# 4th counter rotation end

# Final stop code start

def killagain(deck_name):
    f1 = open(deck_name+".stop", "x")
    f1.close()

def open_file(file_name_1, frequency):
    while True:
        try:
            file1 = open(file_name_1, "r")
            break
        except:
            time.sleep(1/frequency)
            continue
    return file1

def iterate1(file1, frequency):
    global lastRecordedTime
    global lastRecordedTorque
    print(lastRecordedTime)
    print(lastRecordedTorque)
    N=2
    data1 = []*N
    timestep = 0
    while True:
        lines1 = file1.readlines()
        if not lines1:
            time.sleep(1/frequency)
            continue
        else:
            totalLines1 = len(lines1)
            lastline1 = lines1[totalLines1-1]
            lastRecordedTime = lastline1.split(',')[0]
            lastRecordedTorque = lastline1.split(',')[1]
            print(lastRecordedTime)
            print(lastRecordedTorque)
            dt1 = lastRecordedTime
            if dt1 != timestep and len(data1)>0:
                print("Entered If Block")
                timestep1 = dt1
                values1 = np.genfromtxt(data1, delimiter=',')
                if finalstop(values1) == True:
                    break
                N=2
                data1 = []*N
                data1.extend(lines1)
                print("Appended Data")

def finalstop(values1):
    global lastRecordedTime
    break_value_1 = 35
    print("Attempting Comparison")
    print(lastRecordedTime)
    if break_value_1 <= float(lastRecordedTime):
        return True
    else:
        return False

# Final stop code end

def main():
    file_name = "lid_torque_PS1.csv"
    deck_name = "Shear_stop_1.5kg_auto.dem"

```

```

frequency = 10000
start()
file = open_file(file_name, frequency)
iterate(file, frequency)
file.close()
kill(deck_name)
os.remove(deck_name+".stop")
print(lastRecordedTimeValue)
time = float(lastRecordedTimeValue)
print(time)

#Stopping lid rotation after powder consolidation

deck = Deck("Shear_stop_1.5kg_auto.dem", mode='w')
deck.setKinematicEndTime('Trough', 'First_Pre_Shear', time)
deck.setKinematicEndTime('Shearing_Lid', 'First_Pre_Shear', time)
deck.setKinematicStartTime('Shearing_Lid', 'New Motion 18', time)
deck.setKinematicEndTime('Shearing_Lid', 'New Motion 18', time+0.1)
deck.setKinematicStartTime('Trough', 'First_Pre_Shear_Inversal', time+0.1)
deck.setKinematicStartTime('Shearing_Lid', 'First_Pre_Shear_Inversal', time+0.1)
deck.save()

#First shearing end

#1st counter rotation start

start()
file_name_2 = "lid_torque_CR1.csv"
file2 = open_file(file_name_2, frequency)
iterate_CR2(file2, frequency)
file2.close()
kill_CR2(deck_name)
os.remove(deck_name+".stop")
print(lastRecordedTime_CR2)
time_CR2 = float(lastRecordedTime_CR2)
print(time_CR2)

#First shearing & counter rotation end
deck = Deck("Shear_stop_1.5kg_auto.dem", mode='w')
deck.setKinematicEndTime('Trough', 'First_Pre_Shear_Inversal', time_CR2)
deck.setKinematicEndTime('Shearing_Lid', 'First_Pre_Shear_Inversal', time_CR2)
deck.setKinematicStartTime('Trough', 'First_Shearing', time_CR2+0.3)
deck.setKinematicStartTime('Shearing_Lid', 'First_Shearing', time_CR2+0.3)
deck.setKinematicEndTime('Trough', 'First_Shearing', time_CR2+1.3)
deck.setKinematicEndTime('Shearing_Lid', 'First_Shearing', time_CR2+1.3)
deck.setKinematicStartTime('Shearing_Lid', 'Second_Pre_Shear_Weight_Imposition', time_CR2+1.3)
deck.setKinematicEndTime('Shearing_Lid', 'Second_Pre_Shear_Weight_Imposition', time_CR2+1.8)
deck.setKinematicStartTime('Shearing_Lid', 'Second_Pre_Shear', time_CR2+1.8)
deck.setKinematicStartTime('Trough', 'Second_Pre_Shear', time_CR2+1.8)
deck.save()

#First shearing complete

#Second pre-shear start

start()
file_name_3 = "lid_torque_PS2.csv"
file3 = open_file(file_name_3, frequency)
iterate_CR3(file3, frequency)
file3.close()
kill_CR3(deck_name)
os.remove(deck_name+".stop")
print(lastRecordedTime_CR3)
time_CR3 = float(lastRecordedTime_CR3)
print(time_CR3)

#Stopping lid rotation after powder consolidation
deck = Deck("Shear_stop_1.5kg_auto.dem", mode='w')
deck.setKinematicEndTime('Trough', 'Second_Pre_Shear', time_CR3)
deck.setKinematicEndTime('Shearing_Lid', 'Second_Pre_Shear', time_CR3)
deck.setKinematicStartTime('Shearing_Lid', 'New Motion 19', time_CR3)
deck.setKinematicEndTime('Shearing_Lid', 'New Motion 19', time_CR3+0.1)
deck.setKinematicStartTime('Trough', 'Second_Pre_Shear_Inversal', time_CR3+0.1)
deck.setKinematicStartTime('Shearing_Lid', 'Second_Pre_Shear_Inversal', time_CR3+0.1)
deck.save()

#Second pre-shear end

#Second counter rotation start

start()
file_name_4 = "lid_torque_CR2.csv"
file4 = open_file(file_name_4, frequency)

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iterate_CR4(file4, frequency)
file4.close()
kill_CR4(deck_name)
os.remove(deck_name+".stop")
print(lastRecordedTime_CR4)
time_CR4 = float(lastRecordedTime_CR4)
print(time_CR4)

#Second shearing & counter rotation end

deck = Deck("Shear_stop_1.5kg_auto.dem", mode='w')
deck.setKinematicEndTime('Trough', 'Second_Pre_Shear_Inversal', time_CR4)
deck.setKinematicEndTime('Shearing_Lid', 'Second_Pre_Shear_Inversal', time_CR4)
deck.setKinematicStartTime('Trough', 'Second_Shearing', time_CR4+0.3)
deck.setKinematicStartTime('Shearing_Lid', 'Second_Shearing', time_CR4+0.3)
deck.setKinematicEndTime('Trough', 'Second_Shearing', time_CR4+1.3)
deck.setKinematicEndTime('Shearing_Lid', 'Second_Shearing', time_CR4+1.3)
deck.setKinematicStartTime('Shearing_Lid', 'Third_Pre_Shear_Weight_Imposition', time_CR4+1.3)
deck.setKinematicEndTime('Shearing_Lid', 'Third_Pre_Shear_Weight_Imposition', time_CR4+1.8)
deck.setKinematicStartTime('Shearing_Lid', 'Third_Pre_Shear', time_CR4+1.8)
deck.setKinematicStartTime('Trough', 'Third_Pre_Shear', time_CR4+1.8)
deck.save()

#Second shearing complete

#Third pre-shear start

start()
file_name_5 = "lid_torque_PS3.csv"
file5 = open_file(file_name_5, frequency)
iterate_CR5(file5, frequency)
file5.close()
kill_CR5(deck_name)
os.remove(deck_name+".stop")
print(lastRecordedTime_CR5)
time_CR5 = float(lastRecordedTime_CR5)
print(time_CR5)

#Stopping lid rotation after powder consolidation

deck = Deck("Shear_stop_1.5kg_auto.dem", mode='w')
deck.setKinematicEndTime('Trough', 'Third_Pre_Shear', time_CR5)
deck.setKinematicEndTime('Shearing_Lid', 'Third_Pre_Shear', time_CR5)
deck.setKinematicStartTime('Shearing_Lid', 'New Motion 20', time_CR5)
deck.setKinematicEndTime('Shearing_Lid', 'New Motion 20', time_CR5+0.1)
deck.setKinematicStartTime('Trough', 'Third_Pre_Shear_Inversal', time_CR5+0.1)
deck.setKinematicStartTime('Shearing_Lid', 'Third_Pre_Shear_Inversal', time_CR5+0.1)
deck.save()

#Third pre-shear end

#Third counter rotation start

start()
file_name_6 = "lid_torque_CR3.csv"
file6 = open_file(file_name_6, frequency)
iterate_CR6(file6, frequency)
file6.close()
kill_CR6(deck_name)
os.remove(deck_name+".stop")
print(lastRecordedTime_CR6)
time_CR6 = float(lastRecordedTime_CR6)
print(time_CR6)

#Second shearing & counter rotation end

deck = Deck("Shear_stop_1.5kg_auto.dem", mode='w')
deck.setKinematicEndTime('Trough', 'Third_Pre_Shear_Inversal', time_CR6)
deck.setKinematicEndTime('Shearing_Lid', 'Third_Pre_Shear_Inversal', time_CR6)
deck.setKinematicStartTime('Trough', 'Third_Shearing', time_CR6+0.3)
deck.setKinematicStartTime('Shearing_Lid', 'Third_Shearing', time_CR6+0.3)
deck.setKinematicEndTime('Trough', 'Third_Shearing', time_CR6+1.3)
deck.setKinematicEndTime('Shearing_Lid', 'Third_Shearing', time_CR6+1.3)
deck.setKinematicStartTime('Shearing_Lid', 'Fourth_Pre_Shear_Weight_Imposition', time_CR6+1.3)
deck.setKinematicEndTime('Shearing_Lid', 'Fourth_Pre_Shear_Weight_Imposition', time_CR6+1.8)
deck.setKinematicStartTime('Shearing_Lid', 'Fourth_Pre_Shear', time_CR6+1.8)
deck.setKinematicStartTime('Trough', 'Fourth_Pre_Shear', time_CR6+1.8)
deck.save()

#Third shearing complete

#Fourth pre-shear start

```

```

start()
file_name_7 = "lid_torque_PS4.csv"
file7 = open_file(file_name_7, frequency)
iterate_CR7(file7, frequency)
file7.close()
kill_CR7(deck_name)
os.remove(deck_name+".stop")
print(lastRecordedTime_CR7)
time_CR7 = float(lastRecordedTime_CR7)
print(time_CR7)

#Stopping lid rotation after powder consolidation

deck = Deck("Shear_stop_1.5kg_auto.dem", mode='w')
deck.setKinematicEndTime('Trough', 'Fourth_Pre_Shear', time_CR7)
deck.setKinematicEndTime('Shearing_Lid', 'Fourth_Pre_Shear', time_CR7)
deck.setKinematicStartTime('Shearing_Lid', 'New Motion 21', time_CR7)
deck.setKinematicEndTime('Shearing_Lid', 'New Motion 21', time_CR7+0.1)
deck.setKinematicStartTime('Trough', 'Fourth_Pre_Shear_Inversal', time_CR7+0.1)
deck.setKinematicStartTime('Shearing_Lid', 'Fourth_Pre_Shear_Inversal', time_CR7+0.1)
deck.save()

#Fourth pre-shear end

#Fourth counter rotation start

start()
file_name_8 = "lid_torque_CR4.csv"
file8 = open_file(file_name_8, frequency)
iterate_CR8(file8, frequency)
file8.close()
kill_CR8(deck_name)
os.remove(deck_name+".stop")
print(lastRecordedTime_CR8)
time_CR8 = float(lastRecordedTime_CR8)
print(time_CR8)

#Second shearing & counter rotation end

deck = Deck("Shear_stop_1.5kg_auto.dem", mode='w')
deck.setKinematicEndTime('Trough', 'Fourth_Pre_Shear_Inversal', time_CR8)
deck.setKinematicEndTime('Shearing_Lid', 'Fourth_Pre_Shear_Inversal', time_CR8)
deck.setKinematicStartTime('Trough', 'Fourth_Shearing', time_CR8+0.3)
deck.setKinematicStartTime('Shearing_Lid', 'Fourth_Shearing', time_CR8+0.3)
deck.setKinematicEndTime('Trough', 'Fourth_Shearing', time_CR8+1.3)
deck.setKinematicEndTime('Shearing_Lid', 'Fourth_Shearing', time_CR8+1.3)
deck.save()

#Fourth shearing complete

#Final stop and running analysis script

start()
file_name_1 = "lid_torque_z.csv"
file1 = open_file(file_name_1, frequency)
iterate1(file1, frequency)
file1.close()
killagain(deck_name)
os.remove(deck_name+".stop")
post_processing_script()

main()

```
