# CHARACTERISING POWDER FLOW IN DYNAMIC PROCESSES

*An extended abstract of PhD thesis*

**By**

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# 1 Summary

Flow inconsistency of powders is a major problem in industrial processes, which often leads to product wastage, hence economical losses. Many powder flowability characterisation techniques exist, however, their applicability to predict in-process flow behaviour of a powder is limited by the data relevance to the process conditions. Most industrial processes operate beyond the quasi-static flow regime, where flow is predominantly characterised by frictional and collisional interactions between particles. Therefore, prediction of flow performance in these processes requires the use of dynamic flow characterisation techniques, which provide a more accurate characterisation of material performance under process-relevant conditions. The FT4 powder rheometer is widely used in industry for dynamic flow characterisation, however, the complex shearing flows in the powder bed are not fully understood. There is also lack of understanding on the relationship between the prevailing shear stresses in the powder bed and the measured flow energy, and its dependence on material properties and blade operational conditions.

In this work, the flow energy of model powders is experimentally measured using the FT4 powder rheometer and related to physical powder properties and blade tip speed. The Discrete Element Method (DEM) is used to simulate the dynamic flow in the FT4 powder rheometer, which is validated against the experimental flow energy and particle velocity distributions measured by Positron Emission Particle Tracking (PEPT) and Particle Image Velocimetry (PIV). Initially, the sensitivity of the flow energy to sliding friction is evaluated, and it is demonstrated that a velocity-dependent sliding friction model provides better prediction of the experimental flow energy than the conventional velocity-invariant sliding friction model. The distribution of the prevailing stresses and strain rates and their dependence on material properties and blade operational conditions are numerically analysed. An empirical relationship for predicting the average shear stress in the powder bed based on blade torque is developed. Furthermore, constitutive relationships that describe the dynamic granular rheology as a function of material properties and process parameters are developed. A new experimental protocol for the FT4 powder rheometer is proposed to quantify mixing and segregation behaviour of binary granular mixtures under the dynamic shear conditions. A correlation between mixing index and flow energy is developed via DEM. The proposed experimental methodology together with the developed correlation between mixing index and flow energy demonstrate the great potential of the FT4 powder rheometer as a design tool for prediction of

optimum mixing performance under process-related conditions based on system input energy and material properties.

## 2 State of the art and problem addressed

The primary purpose of measuring powder flowability is to characterize flow properties of bulk powders required for reliable industrial process design and control. Poor powder flowability during processing of granular materials in industry unit operations such as storage, fluidization, filling, pneumatic conveying, milling, mixing and blending can lead to increased downtime, loss of product quality, equipment maintenance problems, and product wastage due to loss of material functionality, which subsequently lead to economical loss (Bell, 2001; Freeman, 2007; Leturia *et al.*, 2014). Therefore, characterization of flow properties of bulk powders is required for reliable prediction of flow performance hence efficient and optimum operation of these industrial processes. There exist several traditional methods that have been used to measure powder flowability, which include angle of repose, Hausner ratio, avalanche technique, uniaxial compression testers and shear cell testers among others. The quick and easy qualitative methods such as angle of repose and avalanching behaviour can be used for quality control to compare and rank different bulk powders whilst quantitative methods such as the shear cell method have been used reliably for appropriate design of hoppers (Schwedes, 2003). The recently developed techniques such as the raining bed method (Formisani *et al.*, 2002), Sevilla powder tester (Castellanos *et al.*, 2004) and the ball indentation method (Hassanpour & Ghadiri, 2007), are capable of accurately evaluating powder flowability at low stress levels, and therefore, can represent the actual state of the system for low stress applications such as filling or powder feeding in tabletting machines, dosing and dry powder dispersion for dry powder inhalers.

The drawback of the aforementioned quasi-static techniques is that they can only predict the onset of powder flow i.e., incipient flow transition from a non-flowing static state to a flowing state.  However, in many industrial processes, characterisation of the powder flow behaviour in the flowing state is required for process control. Industrial processes such as mixing are often limited by the powder shear rate and mixing time due to the variation of shear stress at high strain rates. Various dynamic flow characterisation techniques such as the Couette device, dynamic ball indentation, Stable Micro Systems powder flow analyser and the Freeman FT4 powder rheometer, have been developed to address flow problems beyond the quasi-static regime, where powder flow behaviour is sensitive to the shear flow rate. These techniques measure flowability of powder whilst the powder is in motion, which is useful for determining process-related performance. Although the Couette cell has been used extensively in literature to demonstrate the existence of the different granular flow regimes and to understand the underlying powder mechanics, a large quantity of powder sample is required and the complex shear stress variation in the granular bed due to the anisotropic bed fabric further limits its application to characterising flow properties (Kumar *et al.*, 2013). The dynamic ball indentation method is a relatively new promising technique but requires further study to be established as a suitable dynamic flow characterisation technique, particularly with regards to the dynamics of the indenter and the relationship between the constraint factor and powder properties. The main alternative to the Couette cell and dynamic ball indentation method is the shear deformation of a powder bed using a rotating blade.

The FT4 powder rheometer and the Stable Micro Systems powder flow analyser have similar operating principles, whereby the resistance of a rotating blade is measured as it penetrates a powder bed contained in a cylindrical vessel. However, the FT4 powder rheometer has emerged as a popular instrument in industry for measurement of powder flow properties under dynamic shear conditions. Powder flowability is characterised based on rotational (torque) and translational (axial force) work done by the rotating blades traversing a column of powder sample, referred to as flow energy. Freeman (2007) measured the flow energy of six different cohesive and free flowing powders and showed good correlation of flow energy with shear cell measurements while also demonstrating that dynamic testing is highly differentiating even for powders that show similar shear resistance in a shear cell. Bharadwaj *et al.* (2010) carried out numerical analysis of the FT4 powder rheometer using DEM to investigate the effects of particle shape, size, size distribution and inter-particle static and rolling friction on the force and torque measurements of the blade using non-cohesive spherical glass beads. They showed that the particle size and size distribution had minimal effect on axial force and torque measurements whilst the particle shape and friction coefficient had significant influence on the flow energy. The aforementioned studies demonstrate the capability of the FT4 powder rheometer to characterise dynamic flow but little is understood of the evolution of shear stress within the powder bed with respect to material properties and blade operational conditions. Consequently, the instrument is currently used only for comparative testing of powders rather than process design. Hare *et al.* (2015) analysed the dynamics of the FT4 powder rheometer for cohesive material (silanised glass beads) using DEM and found that the shear stress is approximately constant along the length of the twisted blade (or in radial direction) and increases approximately linearly with penetration depth. Nan *et al.* (2017) simulated the dynamic flow behaviour of non-cohesive polyethylene spherical particles in the FT4 powder rheometer in the presence of air flow at high strain rates using DEM coupled with Computational Fluid Dynamics (CFD) and showed good correlation between simulation results and experimental results of the FT4 permeability test. They further analysed the stresses and strain rates immediately in front of the blade and found that the flow energy and stresses are dependent on the strain rate, which is non-uniform along the length of the blade.

Several studies have also been carried out on the FT4 powder rheometer to demonstrate its potential to characterise mixing and segregation of multi-component mixtures. Yan *et al.* (2016) studied the effects of particle size ratio and volume fraction on segregation of binary mixtures in the FT4 powder rheometer using DEM and found that both larger size ratios and volume fractions of the coarse particles tend to promote faster segregation when the system is initially fully mixed. They attributed the segregation tendency to the sifting mechanism, whereby localised shearing and dilatation allow smaller particles to sift through gaps between larger particles. Forte *et al.* (2018) used ECT to relate the electrical permittivity changes of a binary granular mixture to mixing and segregation in the FT4 powder rheometer. They demonstrated that the evolution of the relative permittivity and flow energy could be related to the mixing and segregation tendency in the FT4 powder rheometer, with mixture homogeneity achieved when the relative permittivity converges to a constant value. The rate of convergence of the relative permittivity of the mixture was found to be proportional to the mixing rate. However, this technique is only applicable for granular materials with distinguishable electrical permittivity. Pasha *et al.* (2020) numerically investigated the effect of size, density and interfacial energy on the flowability of binary and ternary mixtures in the FT4 powder rheometer. They correlated the flow energy of the mixtures to the arithmetic mean of granular Bond number, which provided the best unification of data for a wide range of granular Bond numbers.

While these efforts have elucidated to some extent the shear dynamics of the FT4 powder rheometer and demonstrated its potential to be used for determining the shear stress of granular materials under dynamic conditions, further work is required to link the measured flow energy and shear stress as a function of particle properties and blade operational conditions. Moreover, the operational window i.e., the range of achievable strain rates in the device, as well as the stress and strain rate sensitivity to particle properties, are yet to be fully established. The present study aims to develop constitutive relationships that describe the rheology of powders at high strain rates, and to characterise mixing and segregation behaviour of mixtures under these conditions by employing experimental and computational (DEM) approaches. DEM is utilised to correlate the prevailing shear stress with measured blade torque and material properties over a wide range of strain rates. An experimental FT4 protocol is developed to quantify mixing and segregation of mixtures under dynamic strain-controlled conditions. Understanding the shear dynamics of the FT4 powder rheometer would enable its potential use as a tool for determining the shear stress of granular materials under dynamic conditions and subsequently as a design tool for predicting powder flow and mixing performance under process-relevant conditions.

# 3 Key scientific and technological innovations, applications, implementations and results

## 3.1 Influence of material properties and blade tip speed on dynamic flow properties (Khala *et al.*, 2020)

The effects of material properties and blade operational conditions on flow energy, measured by the FT4 powder rheometer, were investigated for a range of materials. The flow energy increases with decreasing particle size. This can be attributed to the increase in the ratio of cohesive to gravitational forces with decreasing particle size. Moreover, the greater number of particles in the powder bed increases the number of particle-particle and particle-geometry contacts leading to increased frictional resistance, hence higher flow energies for smaller particle sizes. The effect of blade tip speed on flow energy was evaluated. For small particle sizes (< 300 $\mu$m), the flow energy initially decreases with increasing blade tip speed before plateauing. However, for particles with a diameter greater than the blade clearance, the flow energy increased with increasing blade tip speed. This contrasting flow behaviour can be attributed to a transition from quasi-static to intermediate flow regimes. The initial decrease of flow energy with increasing tip speed for small particle sizes is attributed to higher shear forces required to initiate flow in a densely packed powder bed resulting in higher flow energies at very low blade tip speeds. Furthermore, air entrainment becomes more significant at higher blade tip speeds, which reduce the frictional resistance between particles, hence lower flow energies are obtained. The air entrainment effect is largely negligible for large particles due to the presence of relatively larger voids between particles prior to shearing, therefore, the flow behaviour is dominated by the shear stresses imposed by the blade on the powder bed.

Increasing the particle density resulted in an increase in flow energy, owing to the increased inertial and gravitational forces for high particle densities.

### 3.2 Effect of sliding friction coefficient on dynamic flow energy (Khala *et al*., 2021b)

Numerical simulations of the dynamic flow energy test were carried to determine the prevailing stresses in the powder bed and the range of achievable strain rates. Initially, the sliding friction coefficient was experimentally measured at different sliding velocities and normal forces by shearing two monolayers of dry spherical glass particles. The sliding friction coefficient between the glass surfaces exhibits a velocity-strengthening behaviour but is less sensitive to the variation of normal force for both particle-particle and particle-wall contacts. A friction law correlating the sliding friction coefficient to sliding velocity and normal force was established and implemented into a DEM model. Simulations of the experimental flow energy test were carried out using the developed velocity-dependent sliding friction and the conventional velocity-invariant sliding friction over a range of blade tip speeds. PEPT and PIV techniques were employed to further validate the DEM simulations. The constant friction model only provides a good prediction of the experimental flow energy over a very narrow range of blade tip speeds, whereas the velocity-dependent friction model provides better prediction of the experimental flow energy over a wider range of blade tip speeds. This emphasises the need for quantification of frictional behaviour of granular materials for model calibration, which is necessary for accurate prediction of shear flow behaviour.

### 3.3 Shear stress and strain rate distribution in the powder bed (Khala *et al*., 2022)

Using the calibrated DEM model from section 3.2, numerical analysis of the shear stress distribution in the powder bed showed that the shear stress increases with penetration depth due to increasing hydrostatic pressure above the blade. In the shear band, a low stress zone exists above the blade whilst a high stress zone exists below the blade due to the combined effects of increased frictional resistance with bed depth or hydrostatic pressure, as well as the blade pitch compacting the particles downwards. Dissipation of the shear stress in the circumferential direction away from blade indicates that the shear stress is greatest in the region nearest to the front of the blade. Analogously, the strain rate decreases with increasing angular distance away from the blade as the bed becomes less dynamic. Generally, the shear stress is approximately constant in the radial direction along the blade geometry. The effects of blade tip speed, aspect ratio and cohesion on the stress distribution were evaluated. Increasing the blade tip speed, particle aspect ratio or interfacial energy results in an increase in the shear stress, with the effects more pronounced near the tip of the blade. The blade torque, flow energy and the average shear stress imposed on the powder bed are dependent on the blade tip speed, aspect ratio and interfacial energy. An empirical relationship between the shear stress and blade torque was developed, which is dependent on the height of the moving bed equivalent to the shear band thickness. The height of the moving bed is independent of the blade tip speed and interfacial energy for the ranges investigated but decreases slightly with increasing aspect ratio. A good correlation was observed between the shear stress predicted by DEM and the shear stress estimated by the proposed empirical relationship based on blade torque.

### 3.4 Rheological models for dynamic shear flow (Khala *et al*., 2022)

Constitutive relations that provide a unified description of the dynamic rheology of powders based on the shear stress, apparent shear viscosity, macroscopic friction coefficient, inertial number and bond number were explored. A generalised correlation of non-dimensional shear stress against the inertial number for the downward and upward blade traverse unifies the data into a single curve for the range of strain rates investigated. The constitutive $\mu(I)$ rheological law can be applied to describe granular flow in the FT4 powder rheometer. The macroscopic friction coefficient remains constant below the quasi-static limit of the inertial number, beyond which it increases with increasing inertial number. The macroscopic friction coefficient is dependent on the particle aspect ratio as it increases with increasing aspect ratio, however, it is independent of the interfacial energy. The apparent shear viscosity decreases logarithmically with inertial number, which is analogous to non-Newtonian shear thinning fluid behaviour. However, the data collapses into separate curves for different blade tip speeds, which may be due to the influence of granular temperature. The granular bond number correlates well with the macroscopic friction coefficient and provides data unification for the range of strain rates investigated. The developed rheological models give a unified description of the granular flow at high strain rates and paves the way for application of these models to describe and predict material flow in industrial processes.

### 3.5 Prediction of mixing performance and segregation tendency (Khala *et al*., 2021a)

The dynamic nature of the test procedure in the FT4 powder rheometer can induce mixing and/or segregation of the tested material, as such the device has the potential to be used as a prediction tool for mixing performance and segregation tendency of multi-component mixtures. Density and size-induced mixing and segregation of binary mixtures of glass and/or resin particles were investigated experimentally by proposing a new FT4 methodology. Quantification of mixing and segregation of mixture components based on a mixing index can be performed using the proposed methodology. Variations of flow energy and torque were related to the mixing index for materials with distinct differences in size and density, thus demonstrating the potential of the FT4 powder rheometer as a predictive tool for mixture uniformity under process-relevant stress conditions. Numerical analysis by DEM showed that for non-cohesive systems mixing and segregation occur by percolation of fines through inter-particulate gaps formed in the sheared layer, whereas for the cohesive systems, mixing occurs by rupture of cohesive fine clusters when sheared. Increasing the interfacial energy resulted in a reduction of the mixing rate and mitigation of segregation for the highly cohesive mixtures for the number of test cycles investigated. For the non-cohesive binary mixtures, higher blade tip speeds inhibit percolation due to lower ratios of axial to horizontal velocities, thus reducing the rate of mixing and segregation of differently sized components. Contrastingly, for cohesive binary mixtures, the mixing index increases with increasing blade tip speed due to the higher shear stresses imposed on the powder bed, which promote breakage of the agglomerates. A correlation between mixing index and cumulative flow energy showed that for highly cohesive mixtures, higher flow energies are required to achieve the same mixing index as the less cohesive systems. The mixing index was also correlated with a non-dimensional flow energy, normalised by the cohesive energy, providing data unification for all cohesive mixtures investigated. The proposed correlation between mixing index and non-dimensional flow energy is relevant to industrial applications for quantitative prediction and control of homogeneity of multi-component mixtures based on the system energy input and primary material properties.

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