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Cohesive Powder Flow: Trends and Challenges in Characterisation and Analysis[†]

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Abstract

Powder processing and manufacturing operations are rate processes for which the bottleneck is cohesive powder flow. Diversity of material properties, particulate form, and sensitivity to environmental conditions, such as humidity and tribo-electric charging, make its prediction very challenging. However, this is highly desirable particularly when addressing a powder material for which only a small quantity is available. Furthermore, in a number of applications powder flow testing at low stress levels is highly desirable.

Characterisation of bulk powder failure for flow initiation (quasi-static) is well established. However, bulk flow parameters are all sensitive to strain rate with which the powder is sheared, but in contrast to quasi-static test methods, there is no shear cell for characterisation of the bulk parameters in the dynamic regime. There are only a handful of instruments available for powder rheometry, in which the bulk resistance to motion can be quantified as a function of the shear strain rate, but the challenge is relating the bulk behaviour to the physical and mechanical properties of constituting particles. A critique of the current state of the art in characterisation and analysis of cohesive powder flow is presented, addressing the effects of cohesion, strain rate, fluid medium drag and particle shape.

Keywords: cohesive powder, bulk flow, characterisation, flowability, spreadability, additive manufacturing

1. Introduction

Particulate solids are ubiquitous in many manufacturing industries, ranging from pharmaceuticals, foods, chemicals and minerals to additive manufacturing, which is the fastest growing sector in high value manufacturing and depends critically on powder spreading. Powder pro-

cessing and manufacturing operations are rate processes for which the bottleneck is cohesive powder flow. Diversity of material properties and sensitivity to environmental conditions, such as humidity and tribo-electric charging, make the prediction of powder flow very challenging. However, such prediction is highly desirable particularly when addressing a powder material for which only a small quantity is available or when flow testing at low stress levels is required.

Characterisation of bulk powder failure for flow initiation (quasi-static) is well established, although empiricism is still the order of the day. There are in fact a large number of commercial devices as well as some developed in-house, which can apply a compressive and shearing or a tensile load to a powder bed and provide information on bulk density, bulk cohesion, internal and wall angles of friction, unconfined yield stress and tensile failure stress, all of which can be obtained as functions of applied load and consolidation time. Examples of such instruments

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include: Schulze Ring Shear Tester (Schulze, 1994), Brookfield Powder Flow Tester (Berry et al., 2014), Jenike and Peschel Powder Testers, as reviewed by Schwedes and Schulze (1990), Salerno Unconfined Compression Tester (Parrella et al., 2008), Edinburgh Powder Tester (Bell, 2007), Environmental Caking Tester (Calvert et al., 2013), Ball Indentation Method (Hassanpour and Ghadiri, 2007), SSSpin Tester of Material Flow Solutions Inc. (Johanson, 2019), the shear cell of FT4 Powder Rheometer (Freeman Technology), Sevilla Powder Tester (Castellanos et al., 2004), and the Calabria Raining Bed method (Girimonte et al., 2018). These testers all produce different quantitative values of the bulk parameters, as the powder response depends on the residual stress history and bulk structure. Cohesive powders readily form clusters, the size and packing density of which depend on the stress history (Li et al., 2018; Ku et al., 2015). Moreover, beyond the quasi-static regime the bulk flow parameters are all sensitive to shear strain rate (Tardos et al., 2003), but in contrast to quasi-static test methods, there is no shear cell that can characterise the bulk parameters at high strain rates, i.e. in the dynamic regime. There are only a handful of instruments available for powder rheometry, the most prominent ones being the FT4 Rheometer of Freeman Technology, the powder cell of Anton Paar Modular Compact Rheometer and a Couette device, as described later below. In these devices the bulk resistance to motion can be quantified as a function of speed of shearing (strain rate) and is reviewed here. However, a challenge remains to relate the bulk rheological characteristics, i.e. bulk friction and viscosity, to the physical and mechanical properties of constituting particles in order to predict the bulk flow behaviour.

For free flowing (cohesionless) granular materials, the dimensionless group inertial number, I , has been proposed to account for the dependence of bulk internal friction coefficient, μ_b , and apparent shear viscosity of flowing powder, η , on the shear strain rate:

$$I = \gamma d_p \sqrt{\frac{\rho_p}{P}} \quad (1)$$

where γ is shear strain rate (1/s), d_p is particle size (m), ρ_p is particle density (kg/m^3), and P is hydrostatic pressure (N/m^2). It is noteworthy that the dimensionless shear strain rate, γ^0 , proposed by Tardos et al. (2003), is a special case of inertial number, where the pressure is approximated by the stress exerted by the weight of a single particle distributed over its projected area:

$$\gamma^0 = \gamma \sqrt{\frac{d_p}{g}} \quad (2)$$

where g is gravitational acceleration (m/s^2). For cohesionless particles, Jop et al. (2006) propose the following form for the bulk internal friction coefficient:

$$\mu_b = \frac{\tau}{P} = \mu_1 + \frac{\mu_2 - \mu_1}{I_0 / I + 1} \quad (3)$$

where τ is the shear stress, μ_1 is the bulk friction coefficient in the quasi-static regime, μ_2 is the asymptotic value corresponding to large inertial numbers and I_0 is a fitting constant. Chialvo et al. (2012) propose a similar functional form with slightly different constants in the above equation.

The apparent shear viscosity, $\eta = \tau/\dot{\gamma}$, generally follows a typical shear-thinning Non-Newtonian trend, but the influence of particle shape, adhesive interactions and medium fluid drag has not yet been extensively analysed.

Rognon et al. (2008) modelled shear deformation of dense cohesive granular material by Discrete Element Method (DEM) using a simple cohesive force, which approximates the DMT theory (Derjaguin et al., 1975), together with a linear spring model for the normal force and Coulombic friction condition for the tangential direction. Various features of the macroscopic bulk behaviour such as bulk friction, microstructure, anisotropy and void fraction were analysed. More recently, Berger et al. (2015) propose an extension for cohesive powders by defining a cohesive inertial number:

$$I_c = \frac{I}{\sqrt{1 + \alpha\beta}} \quad (4)$$

where β is the ratio of cohesive contact force over the normal stress and particle size, i.e. $f_c/(\sigma_n d_p)$ for a two-dimensional geometry, α is a coefficient accounting for the structure of the packing or the details of dissipation mechanisms during flow. Its equivalence for bulk powder materials is the modified Bond number, describing the ratio of the adhesive force over the particle weight, i.e. f_c/mg . For a wide range of conditions, their simulation predictions for the dependence of bulk cohesion, following Coulomb's law, are unified against the cohesive inertial number. Their analysis prescribes the dependence of bulk cohesion as well as bulk friction coefficient on the shear strain rate. A method and device are therefore required for validation of such simulation trends. As mentioned above, there is no shear cell which can characterise the bulk parameters in the dynamic regime. There are only three instruments available for powder rheometry: the Couette device (Tardos et al., 2003), the FT4 Powder Rheometer of Freeman Technology (Freeman, 2007), as analysed recently by a large number of workers, see e.g. Bharadwaj et al. (2010), Hare et al. (2015), Nan et al. (2017a-c), Wilkinson et al. (2017) and Vivacqua et al. (2019) and the powder cell of Anton Paar Modular Compact Rheometer, as recently analysed by Salehi et al. (2018). There are of course other instruments, such as GranuDrum of GranuTools (Lumay et al., 2012), Hall Flow Tester (ASTM B213-17) and Hosokawa Micron Powder Tester (ASTM D6393-08), with the latter integrating

several test methods into one instrument, giving indirect measures of bulk behaviour under flowing conditions. Recently, Ogata (2019) has reviewed the latest works on the evaluation of flowability and floodability of powders with a focus on the experimental methods. In the work reviewed here, we provide a complementary critique of current trends and challenges in the characterisation methods and instruments, and analysis of dynamics of cohesive powder flow with a focus on the rheological properties and methods for their characterisation. The effects of particle size and shape, fluid drag, cohesion and strain rate are covered.

2. Instruments for powder rheometry

Powders exhibit extreme dynamic flow behaviour from highly frictional to almost inviscid, as influenced by particle properties, the drag of the fluid medium and the strain rate. We first review the state of the art in measurement of the bulk rheological response of cohesive powders, subjected to shear straining, followed by a critique of current understanding of the effects of particle properties and process conditions. The dynamic flow testers may conveniently be classified into two categories: (i) those which address powder rheometry by measuring the powder resistance to shearing as a function of shearing speed by a rotating impeller or surface, and (ii) other dynamic flow testers in which the flow behaviour and characteristics are indirectly inferred. In the first category the Couette device, powder cell of the Anton Paar Modular Compact Rheometer and FT4 Powder Rheometer of Freeman Technology are placed. The rotating drum and Ball Indentation Method (BIM) at high strain rates, are placed in the second category. We also include here a review of powder spreadability testing by a blade in view of its increasing importance in additive manufacturing and its relation with powder flowability.

2.1 Powder rheometers

2.1.1 Couette device

This is a simple device based on the design of a classic liquid rheometer, consisting of two co-axial vertical cylinders in which powder is sheared in the annular gap between the two cylinders by rotating the inner cylinder whilst keeping the outer cylinder stationary (Lun et al., 1984; Tardos et al., 1998). Such a device is common for rheological measurements of liquids, though was first reported for powders by Savage and Sayed (1984). It is necessary to ensure the powder is gripped at the wall to provide a no-slip boundary condition and therefore ensure that the entire bed is sheared, as such sandpaper is typically glued to the cylinder walls (Tardos et al., 2003). The

normal stress is adjusted simply by varying the mass of powder above the sheared bed, referred to as the ‘overburden’. Sensors are placed at the outer wall that enable measurement of normal and shear stresses across the height of the sheared region.

A Couette device can be operated in ‘batch’ or ‘continuous’ mode; with a slow, downward axial flow of powder being introduced in the latter, with the removed powder being recirculated to the top, whilst no such axial flow is induced in the former. Batch mode (no axial flow) results in a near constant packing fraction regardless of the shear rate induced by the Couette (Vidyapati, 2012), and consequently leads to shear stress remaining constant across the range of shear rates (Langroudi et al., 2010). It should be noted that the reported work is for large, granular media, and for fine, cohesive powder it could be expected that packing fraction would still vary with strain rate in batch mode. In continuous mode, at low shear rates the packing fraction is lower than that in the batch mode operation, and consequently so is the shear stress, however an increase in shear rate results in dilation and a subsequent increase in shear stress as the flow approaches the collisional regime. Experimental work reported by Savage and Sayed (1984), Qin (2000), Klausner et al. (2000) and Tardos et al. (2003) show the dependency of the shear stress on the strain rate for a number of materials in the quasi-static, intermediate and dynamic regimes, where there is a general agreement that the shear stress increases with strain rate in the intermediate and dynamic regimes. Tardos et al. (2003) defined approximate boundaries of the dimensionless shear strain rate for the quasi-static to intermediate ($\gamma^0 = 0.15$ to 0.25) and intermediate to rapid ($\gamma^0 = 3$) granular flow regimes, based on the dimensionless strain rate given by Eq. (2). Vidyapati et al. (2012) carried out DEM simulations of this device and showed agreement with these boundaries, and indicated that the number of enduring contacts reduces with increasing strain rate, and increases with solid fraction and particle friction.

Langroudi et al. (2010) expressed the bed stress ratio using Eq. (5).

$$\frac{\tau}{\sigma} = a + b\gamma^n \quad (5)$$

where τ and σ are shear and normal stresses, respectively, and a , b and n are constants. This can be further generalised by substituting $a = \tan(\phi)$, where ϕ is the internal angle of friction, thus at zero shear rate Eq. (5) reduces to the Coulomb yield condition with no cohesion.

The vertical force due to gravity compressing the powder is not constant throughout the bed; varying significantly with depth since the cylinders are tall (Tardos et al., 1998). Contradictory trends of vertical and radial stress variations throughout the bed height have been

shown, with Tardos et al. (1998) finding the stress varied with ρgz , Tardos et al. (2003) finding stress scaling with $3\tan(\phi)\rho gz$, and Gutam et al. (2013) showing an exponential growth of stress with z . Furthermore, Gutam et al. (2013) show that the direction of σ_{rz} switches at the onset of shearing, and that the stress variation across the powder bed is dependent on the gap width. It is suggested that this could be driven by a downward flow of particles near the outer wall, due to the anisotropic bed fabric; an observation that shows that the strain field is not as simple as that intended by the Couette design. Anisotropy of the bed fabric has been shown for much simpler systems of static sand piles (Vanel et al., 1999) and during shear (Howell et al., 1999), and hence it is expected that this would indeed also be the case in the more complex Couette flow. Furthermore, Gutam et al. (2013) state that Couette is one of the few devices where gravity is perpendicular to the shear direction, which may have significant effects on the rheology, however this is also true of blade-driven powder rheometers that are discussed further below. Another drawback of the Couette device is the potential for wall slip at the rotating inner cylinder, and steps should be taken to mitigate it. This device also requires a large quantity of powder, so it is not suitable for instances where the powder quantity is scarce.

2.1.2 Powder cell of the anton paar modular compact rheometer

The Anton Paar powder rheometer measures the torque required by an impeller blade to rotate at a fixed height inside a powder bed. Different impeller shapes are available (cylindrical, including one having grooved surfaces, blade, and a ring with radial fins) to investigate the resistance to shearing and influence of wall friction. The powder cell is a 50 mm diameter glass cylinder, having a porous base plate for aeration. The torque sensor can measure a wide range (10^{-8} to 0.3 Nm), making it possible to accurately measure the torque whilst the powder is fluidised and sheared. In a standard test procedure the blade is placed at 10 mm above the bottom of the cell and can be rotated from a very slow speed of 0.001 to 500 rpm. The powder bed can also be conditioned prior to torque measurement to remove any residual vertical friction due to insertion of the impeller in the bed. Recently, Salehi et al. (2017), evaluated the Anton Paar powder cell for characterising flowability at low stresses using glass beads, sand and alumina powder. They reported that the minimum torque necessary to rotate an impeller in a powder bed depends on material properties, air flow rate, impeller depth and shape of the impeller. Furthermore, Salehi et al. (2018) compared torque measurements between different sizes of glass beads and different impeller geometries and used DEM to analyse the dynamic behaviour of the powder. Differences between the temporal profiles of torque

of different impeller shapes are reported which might be attributed to the differences in shearing surfaces and the formation of instantaneous high magnitude force chains. Moreover, as the flow response is measured at a fixed position of the rotating component in the powder bed, the results may not be representative for the whole powder bed (e.g. due to a number of factors such as segregation, local aeration, slipping, etc.). On the other hand, the vertical stress is constant throughout the measurement, which is advantageous for analysis.

2.1.3 FT4 powder rheometer of freeman technology

This instrument (referred to as FT4 herein) measures the mechanical work done by a twisted-shape impeller while penetrating into a powder bed at given rotational and downward translational speeds (Freeman, 2007). The expended work is taken as a measure of flowability under dynamic condition. Two types of test can be carried out: a downward test and an upward test. In the downward test the impeller rotates anticlockwise with its blade surface facing downward, thus compressing and shearing the powder bed. The work expended in this test has been termed ‘Basic Flow Energy (BFE)’. In the upward test, once the downward traverse is complete and the impeller is close to the bottom of the bed, the rotation direction is reversed (clockwise) and the impeller cuts and lifts the powder bed i.e. a dominant tensile and shearing action. The work associated with this action per unit mass of the material is known as ‘Specific Energy (SE)’.

Nowadays, FT4 is used extensively in industry to provide a relative measure of the ease with which powder flows. For example, Fu et al. (2012) reported that FT4 could be used to identify the importance of the effect of particle size and shape of three grades of lactose particles. Li et al. (2018) attributed the torque fluctuation of highly cohesive powders to breakage of cohesive clusters. Wilkinson et al. (2017) carried out a parametric evaluation of flowability as indicated by FT4 through statistical and sensitivity analyses using DEM. A question which naturally emerges is how the work expended on both penetrating and withdrawing the impeller is influenced by particle properties, and whether it can actually reflect bulk rheological properties, such as bulk friction and shear viscosity. In recent years, a good number of attempts have been made to elucidate the correlation between the expended work and the bulk mechanical and rheological features of cohesive powders. Using silanisation to provide controlled bulk cohesion for otherwise free flowing glass ballotini, Hare et al. (2015) showed that the expended work can be simulated by Discrete Element Method (DEM). Several attempts have been made to link the measured flow energy from FT4 to processes that the powder experiences at high strain rates (Goh et al., 2018; Mellin et al., 2017). However, no direct correlation has

been consistently found. In another study, Li et al. (2018) showed that cohesive powders exhibit cyclic torque in FT4, and as cohesion increases, the cycle time increases, whilst free-flowing powders act like liquids with no periodic response. It is noteworthy that powder flow in industrial operations is not necessarily actuated by a moving impeller, and therefore the influence of the presence of the impeller on the powder rheology cannot be ignored, due to the potential of slip on the impeller tip and containing walls, and the local aeration or jamming of particles in the impeller-wall clearance. Nevertheless, to use FT4 and Anton Paar instruments as powder rheometers, it is clearly necessary to analyse the mechanics of powder flow induced by the impeller and relate the expended work to the bulk powder rheological characteristics. For this purpose, Ghadiri and co-workers have carried out a systematic study of the effects of cohesion, air drag and strain rate, and particle shape on bulk powder rheology by analysing the stress and strain fields within FT4 using numerical simulations by a combined DEM-CFD approach (Nan et al., 2017a, b, c; Vivacqua et al., 2019). With current computer power and memory, it is too challenging to use fine cohesive powders with a complex shape in such simulations. For this reason, each effect of cohesion, air drag, and particle shape has been analysed separately as a function of strain rate using large particles for which experimental validation can be made. Salient features of the outcomes of their analysis are summarised in section 3 on the analysis of cohesive powder flow.

2.2 Other dynamic flow testers

2.2.1 Rotating drum

One of the most practical geometries used to study the flow properties of powders, or more generally granular materials, is the rotating drum (e.g. GranuDrum of GranuTools and Revolution Powder Analyzer of Process-technik). It is a free surface flow test under dynamic avalanching condition, contrary to shear testers, where a powder bed is subjected to a compressive load and the resistance to shearing is measured (Nalluri and Kuentz, 2010; Lumay et al., 2012; Yang et al., 2016). The free dynamic flow inside the rotating drum is similar to many industrial processes such as mixers, granulators, and heap formation. The non-cohesive (or mildly cohesive) powder flow inside the rotating drum is commonly defined as a function of the Froude number $Fr = R\Omega^2/g$ (where Ω is the rotational speed and R is the drum radius). It can take on one of a number of regimes such as slumping, rolling, cascading, cataracting and centrifuging. By measuring the flowing angle or speed, all of these regimes can be characterised (MiDi, 2004; Fischer et al., 2008; Morrison et al., 2016). For cohesive powders, determining these parameters is difficult due to the irregular surface of the

flow and the lack of a continuous regime. In this context, some authors propose to measure the standard deviation of the fluctuations of the flow interface in order to characterise the cohesive powders (e.g. Alexander et al., 2006; Lumay et al., 2012, 2016). However, such dynamic tests are generally very challenging to interpret, as adhesive contacts cause clump formation, and adhesion and particle friction have a coupled effect on flow. Although more relevant to free-flowing powders, rotating drums are very effective in causing segregation, adversely affecting the homogeneity of the powder and hence rheological response.

2.2.2 Ball indentation method (BIM) at high strain rates

This test method was initially developed for quasi-static testing of the flow resistance of a powder bed against the penetration of a spherical indenter, which promotes shearing of the bed without further compression (Hassanpour and Ghadiri, 2007). The flow resistance, expressed as hardness, can be related to unconfined yield strength by the constraint factor—a material dependent property—which can be established by carrying out measurements of hardness and unconfined yield strength under identical consolidation conditions. Zafar et al. (2017) have shown the constraint factor to be independent of consolidation stress for a given material. This technique has the advantages of being applicable at low consolidation stresses and requiring only a very small quantity of powder, whilst the disadvantage is measuring hardness rather than unconfined yield strength directly.

The BIM has recently been extended to the dynamic range by dropping a ball onto a powder bed and measuring its penetration depth (Zafar et al., 2019). The dynamic flow resistance expressed by the dynamic hardness of the powder bed surface, H , is determined by

$$H = \frac{1}{2} \frac{MV^2}{U} \quad (6)$$

where M is the mass of the indenter, V is the indenter velocity at impact and U is the volume of the crater formed by the indentation (Tirupataiah and Sundararajan, 1990). On the face of it, dynamic indentation appears to be a relatively quick and simple approach for assessing powder flowability under a wide range of strain rates. However, as well as the disadvantages listed above, this technique is also challenging to apply in practice and requires further development. Moreover, dependence of the constraint factor on strain rate needs to be established if the powder yield stress, rather than the flow resistance, is desired.

Since the indenter is fired at the sample at a given velocity (or released from a given height), the depth to which it penetrates the powder bed is not directly controlled. It has been shown for quasi-static indentation that the

hardness should be measured in a given acceptable range of the penetration depths (Pasha et al., 2013; Zafar et al., 2017). So iterative refinement of test conditions may be required in order to generate reliable flow measurement results at higher strain rates. If the quasi-static hardness is known *a priori*, then the penetration depth can be estimated for an indenter of given size, density and velocity, and hence a maximum velocity which enables reliable measurement can be determined. However, since the expectation is that flow resistance will vary with strain rate, this approach can only provide an estimate of the upper operational range. Other practical considerations of the dynamic indentation test are the requirement to measure penetration depth (to determine the crater volume) and the indenter velocity. A high-speed camera can serve both functions. In order to explore a wider range of strain rates, a variety of indenters of differing material and size can be applied from a range of drop heights, as shown by Tirapelle et al. (2019)

2.2.3 Powder spreadability testers for additive manufacturing

Characterisation of bulk cohesive powder flow has recently attracted increased attention for powder spreading in additive manufacturing (AM). However, there is no standard method for testing whether a given powder can produce the required spread uniformity for further processing. Across the community, every possible characteristic of bulk mechanical properties is being analysed in an attempt to characterise features of powder properties which give uniform spreading (e.g. Nguyen et al., 2017; Carrozza, 2017; Han et al., 2019). However, the recent work of Nan et al. (2018) shows that uniform spreading is affected by transient jamming and arching. Therefore, spreadability and flowability are two different measures of powder bulk flow characteristics, albeit inter-related. The former refers to flow of powders in narrow gaps, whilst flowability is more a measure of bulk behaviour, i.e. not having boundary constraint. Nan et al. (2018) characterised the physical and mechanical properties of gas-atomised stainless steel powders for DEM simulation of powder spreading. They show that for spreading gaps typically used in metal AM applications, empty patches/spaces could be found within the spread particle layer, as shown in **Fig. 1**, which adversely affects the uniformity of the spread powder layer. They find that the characteristic particle size, D , for which 90 % particles by number have a diameter less than D , best describes the transient jamming condition. By using Eq. (7) for diagnosing the empty patches, the transient jamming is found to have a frequency in the range 10–100 Hz, as shown in **Fig. 2**. In Eq. (7) ΣV_p is the total volume of spread particles and the denominator is the volume swept by the spreader blade. A gap size, δ , that is less than about $3D$ causes transient

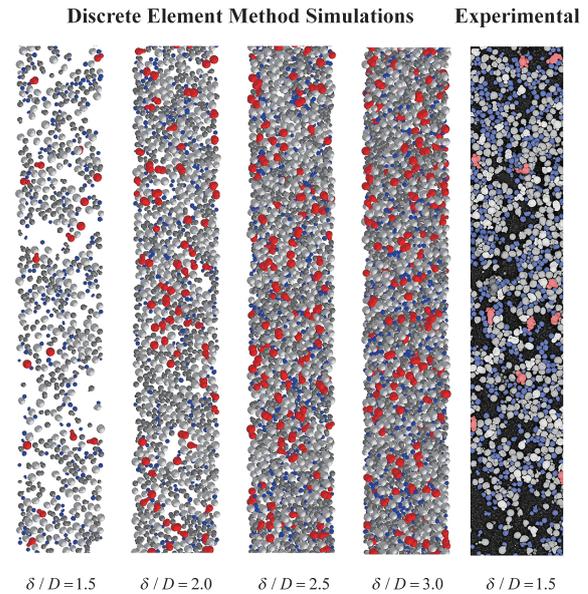


Fig. 1 Discrete Element Method simulation and experimental validation of spreading of gas-atomised metal powders by a blade, showing empty patches and size segregation in the spread layer as a function of spreader gap height. Narrow gaps cause transient jamming, as evident by empty patches. The frequency of formation and size of empty patches can also be inferred, as shown in **Fig. 2**. The particles are coloured based on their projected area equivalent circle diameter, blue (15–25 μm), dark grey (25–35 μm), light grey (35–45 μm), and red (45–55 μm). The number density of the largest particles (shown in red) is the lowest for the smallest gap, as predicted by Nan et al. (2018). The simulation results are for four gap heights and the experimental one is a scanning electron micrograph for the smallest gap, courtesy of Mr M.T. Hussein. Gap heights are normalised with respect to the characteristic particle size below which 90 % of particles by number lie (D_{90}).

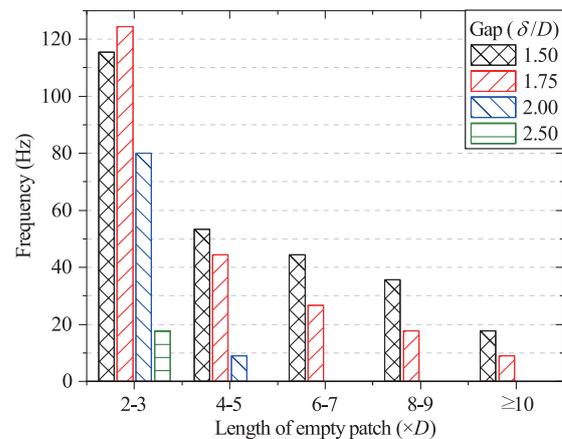


Fig. 2 Frequency of empty patches for each patch length as a function of gap height (Nan et al. (2018), <https://doi.org/10.1016/j.powtec.2018.07.030>. Copyright: (2018) Elsevier B.V. under <https://creativecommons.org/licenses/by/4.0/>).

jamming, leading to empty patches.

$$\frac{\sum V_p}{\Delta x \times \Delta y \times (\delta - \delta_c)} < 0.1 \quad (7)$$

Nan and Ghadiri (2019) identify a critical spreading speed above which the mass flow rate of powder through the gap is independent on the blade spreading speed. Haeri (2017) identified the optimum blade tip shape to produce a spread particle layer with volume fraction and surface roughness comparable to a roller at the actual operation conditions. Geer et al. (2018) and Han et al. (2019) measured the repose angle and pile bulk density of metal powders, and then used DEM simulations to calibrate and characterise the sliding and rolling friction coefficient and surface energy of the powder to be used in their simulations of the spreading process. Desai et al. (2019) also developed a DEM calibration method based on angle of repose testing and powder rheometry for AM, which was designed around multiple characterization experiments applicable to the spreading step. However, particle adhesion and sliding friction influence the repose angle in a complex way, which is not yet fully understood and hence calibration methods should be treated with caution. More work is needed to identify and characterise factors which influence spreadability of powders in AM and develop appropriate instrument for its characterisation.

3. Analysis of cohesive powder flow

3.1 Effect of cohesion

The relative cohesivity of a powder is most commonly described by the granular bond number, Bo , the ratio of the attractive force (adhesive or cohesive) over particle weight (Castellanos, 2005). Cohesion is commonly due to van der Waals, liquid bridge and/or electrostatic forces. There are extensive analyses of bulk cohesion reported in the literature, but its influence on the dynamic flow behaviour has not been as widely investigated. Recently, Hare et al. (2015) made large spherical glass beads (1.7–2.1 mm) cohesive by silanising their surfaces and measured their surface energy by the Drop Test Method (Zafar et al., 2014). They measured the work (basic flow energy) required to penetrate the rotating impeller into the cohesive powder bed, and showed that this could also be quantitatively predicted by DEM using the elasto-plastic adhesive model of Pasha et al. (2014). Bulk cohesion simply lifts up the baseline of powder resistance to bulk motion and the functional dependence on the shear strain rate has the same trend, as recently analysed by Vivacqua et al. (2019). They simulated the FT4 test using DEM for both cohesive (two levels) and cohesionless contacts, using Luding's contact model (Luding, 2008) and calculated

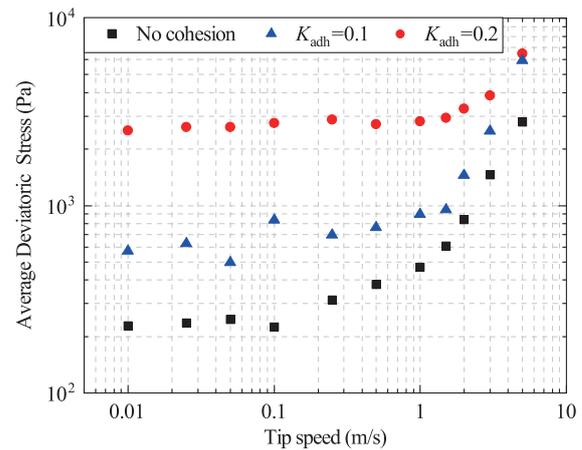


Fig. 3 DEM prediction of shear stress on the impeller blade of the FT4 rheometer as a function of impeller blade tip speed for glass beads for three adhesive indices according to the contact model of Luding (2008). Simulation data are from Vivacqua et al. (2019); <https://doi.org/10.1016/j.powtec.2018.10.034>. Copyright: (2019) Elsevier B.V. under <https://creativecommons.org/licenses/by/4.0/>.

the shear stress on the impeller blade as a function of the impeller tip speed, as shown in **Fig. 3**. Clearly, at low speeds the lines are parallel with the intercept with ordinate being a function of cohesion, and interestingly at high speeds interparticle cohesion has little influence on the shear stress acting on the impeller blades.

The above experimental observations are consistent with the recent numerical analysis of Berger et al. (2015), who used contact dynamics simulations to investigate cohesive shear flows at a wide range of strain rates. Their simulation predictions for the dependence of bulk cohesion, following Coulomb's law, on the shear strain rate shows a logarithmic decrease for a wide range of conditions. This effect was attributed to the reduction in the coordination number with increasing shear strain rate, expressed in terms of inertial number.

3.2 Effect of shear strain rate and medium fluid drag

As the particles become finer, the effect of air drag becomes more pronounced at large shear strain rates. So, this has to be taken into account in the analysis of fine cohesive powder dynamics. Guo et al. (2011a, 2011b) propose a dimensionless number made of the product of the Archimedes number and the ratio of particle envelop density over the fluid density (i.e. an air sensitivity index) to account for the influence of air drag in die filling. They define a critical value of this group below which the fluid drag effect is notable. However, this approach is more relevant to lean particle-fluid systems. Nan et al. (2017a, 2017b) used large particles (for faster simulation) with air

permeation from the base of the FT4 rheometer to provide notable drag. In this way the effect of air drag and strain rate could be analysed in reasonable time for glass beads of 0.3–0.35 mm and polyethylene spheres of 0.5–0.6 mm for permeating gas velocities ranging from no gas flow to near fluidisation. The expended work for penetrating the rotating impeller into the bed and gas pressure drop could be both measured and predicted, thus providing further validation of the simulations. In the absence of air, the expended work increases with the speed of the impeller, as intuitively expected. An interesting outcome of their analysis is that the expended work scales with the potential energy of the bed above the impeller height, except for the top 10 mm, where the entrance effect of the blade is very strong.

Nan et al. (2017a, 2017b) also analysed the stress and strain fields in cells in front of the blade of FT4 rheometer. Their work reveals the sensitivity of powder rheology to both the strain rate and gas velocity. Their simulation results show that the normalised expended work (with respect to the potential energy of the bed mass above the mid-position of the impeller blade) for all conditions including permeating air is actually a linear function of the prevailing normalised shear stress, with the latter in turn being a function of the strain rate and the prevailing pressure, as the latter changes with height. The most notable outcome of their analysis is the prediction of the bulk friction coefficient as a function of the inertial Number. The effect of permeating air simply modifies the prevailing pressure on the particles, whereby increasing the permeating air speed reduces the pressure and therefore increases the inertial number. By introducing the permeating air, the powder flow, which is initially in the quasi-static state, could transform into the intermediate or even dynamic state, where the bulk friction coefficient is shown to increase almost linearly with the inertial number.

Bruni et al. (2007, 2005) and Tomasetta et al. (2012) used a mechanically stirred fluid-bed rheometer to study the rheology of aerated and fluidised powders, which was followed by Salehi et al. (2017, 2018). To keep the bed in quasi-static state, the rotational speed of the impeller and gas superficial velocity were chosen to have low values. Based on the method of differential slices (Janssen's approach) and the Mohr-Coulomb description of the powder failure, they developed a model to describe the applied torque. In their model, the effect of air drag on the powder stress was considered by subtracting the pressure gradient force from the gravity force. The most interesting outcome of their work is that the applied torque of the impeller could be linked to the stress state of the powder. However, as the powder bed was in quasi-static state, their analyses did not reveal the response of powder rheology to the variation of the strain rate or flow regimes. In

conclusion, the dynamic behaviour of fine cohesive powder affected by the fluid medium drag is still a very challenging task to analyse, but based on the above observations, the trend shown in **Fig. 3** for large cohesive particle should also prevail for fine powders, subject to taking account of fluid drag effects.

3.3 Effect of particle shape

Granular flow is highly affected by particle shape due to interlocking of the particles. High fidelity simulation of particle shape provides a complexity which is ill-afforded by the DEM approach, from the viewpoint of contact mechanics. Therefore, approximating to simple shapes is the order of the day. There are a good number of approaches for consideration of shape of particles in DEM simulations: clumped spheres, (Favier et al., 1999), elliptical (Rothenburg and Bathurst, 1991), polygonal (Cundall, 1988), bonded assemblies of polygons (Potapov and Campbell, 1996), spherosimplices (Pournin et al., 2005), super-quadratics (Williams and Pentland, 1992), and digitalisation of particles of arbitrary shapes by voxel packing (Jia and Williams, 2001). For particles with a rounded shape, the clumped sphere approach of Favier et al. (1999) is commonly used. For example, Hare et al. (2013) used this approach to show that the stress ratio in a shear box is greater for rods than for spheres, though decreases as aspect ratio is increased from 1.25–2, remaining constant beyond this due to alignment of the particles. Pasha et al. (2016) simulated mixing of corn kernels by this method, showing that an adequate description of the effect of particle shape on particle velocity profile in a mixer could be achieved. Alizadeh et al. (2017) used X-Ray tomograms of spray dried particles to construct clumped spheres and obtained reliable prediction of the repose angle and segregation of binary mixtures. Nan et al. (2017c) simulated the rheological behaviour of rod-like particles in the FT4 rheometer, using the clumped-sphere approach. They reported the flow energy required for the FT4 impeller penetrating the powder bed is much larger for rod-like particles than for spheres. This was attributed to the combined effect of the coordination number and excluded volume. They also proposed an empirical correlation for the binary mixture of rodlike particles with aspect ratio $AR = l_{max}/l_{min} - 1$ (i.e. $AR = 1.5$ and 3.0) and spherical particles (i.e. $AR = 0.0$). The flow energy was larger for non-spherical particles than that of spherical particles, due to interlocking between particles.

For crystalline solid structures with faceted shapes, sharp edges and corners, it is inappropriate to use clumped spheres. Recently, Vivacqua et al. (2019) used bonded Polyhedra to construct faceted shapes in the DEM simulation software package ROCKY-DEM, ESSS, Brazil, and followed the same approach as Hare et al.

Table 1 Polyhedra used by Vivacqua et al. (2019) for simulating faceted shapes. <https://doi.org/10.1016/j.powtec.2018.10.034>. Copyright: (2019) Elsevier B.V. under <https://creativecommons.org/licenses/by/4.0/>.

Name	Number of faces	Number of corners	3D shape
Deltahedron	16	10	
Faced cylinder	12	20	
Actual paracetamol shape	25	44	
Dodecahedron	12	20	
Truncated polyhedron	14	16	

(2015) and Nan et al. (2017c) to analyse the effect of faceted particle shape on the powder flow rheology for both adhesive and non-adhesive particles using the FT4 rheometer. They investigated the effect of the shapes as illustrated in **Table 1**. The results obtained by Vivacqua et al. (2019) are shown in **Figs. 4 and 5**. They agree qualitatively with the trend shown previously by Nan et al. (2017a); the average shear stress is initially independent of the strain rate, corresponding to the quasi-static value, but starts increasing at large strain rates. The notable point is that faceted shapes present much larger shear stresses, with vertices and edges influencing the resistance, but follow the same trend as that of the spheres, with the base line shifted to larger values of shear stress (as with the effect of cohesion discussed above). A similar trend also prevails when the interparticle adhesion is increased for these faceted shapes. In both cases the normalised shear stress ($\tau/\rho_p d_p^2 \gamma^2$) obeys the following relationship with the inertial number for spheres (Eq. 8) and all faceted shapes, shown in **Table 1**, unified and given by Eq. (9):

$$\frac{\tau}{\rho_p d_p^2 \gamma^2} = 0.481 I^{-1.743} \quad (8)$$

$$\frac{\tau}{\rho_p d_p^2 \gamma^2} = 0.918 I^{-1.754} \quad (9)$$

Thus, two rheological models with roughly the same power index, but with different pre-exponential constants, are obtained for cohesive spheres (Eq. 8) and faceted particles (Eq. 9). These equations point toward the possibility of obtaining a unified rheological model incorporating the effect of cohesion, strain rate and shape.

In a subsequent study by Lopez et al. (2019), powder flow of faceted particles in screw feeders was analysed. Analysis of the stresses in the screw based on the same

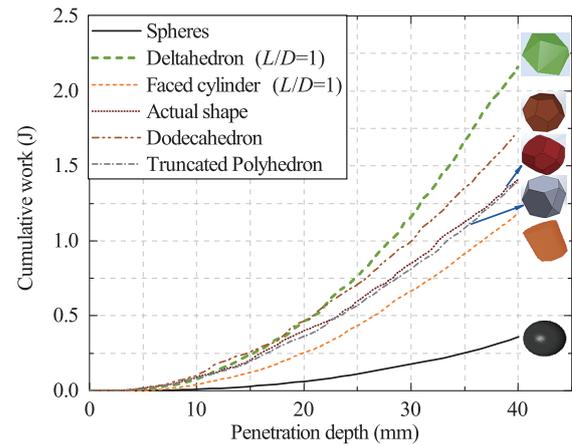


Fig. 4 DEM prediction of expended work to penetrate the impeller blade of FT4 rheometer into a bed of faceted particles in the standard test method. Simulation data are from Vivacqua et al. (2019): <https://doi.org/10.1016/j.powtec.2018.10.034>. Copyright: (2019) Elsevier B.V. under <https://creativecommons.org/licenses/by/4.0/>.

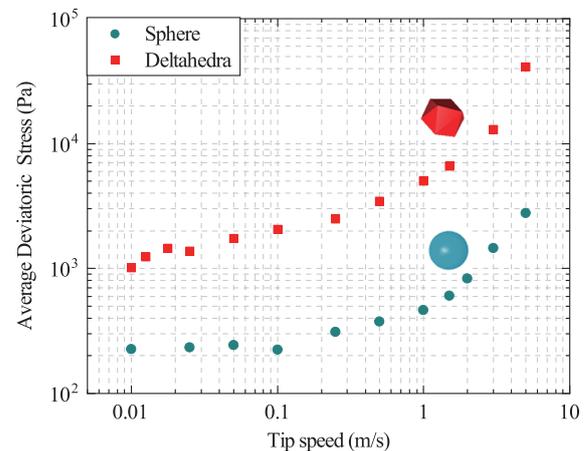


Fig. 5 DEM prediction of shear stress on the impeller blade of FT4 rheometer as a function of impeller blade tip speed for spherical and deltahedra shape particles. Simulation data are from Vivacqua et al. (2019): <https://doi.org/10.1016/j.powtec.2018.10.034>. Copyright: (2019) Elsevier B.V. under <https://creativecommons.org/licenses/by/4.0/>.

approach as Vivacqua et al (2019) showed a linear relationship in a logarithmic plot between the non-dimensional shear stress and the inertial number for different cohesion and strain rate levels. When both analyses are plotted in the same graph, an overlap between the lowest shear rates of the FT4 and the highest shear rates of the screw feeder exists. This shows the close relationship between the flow regimes in both devices for the same shear rates.

The apparent viscosity ($\eta = \tau/\dot{\gamma}$) follows a linear trend with the inertial number on a log-log plot, with a negative slope for both spheres and faceted particles indicating a shear thinning behaviour. The slope obtained for cohesive particles, however, deviates from the behaviour of

non-cohesive particles for some strain rates. Lopez et al. (2019) attributed the trend to transient jamming and clustering of the particles. Recently Macaulay and Rognon (2019) analyse the dynamics of cluster formation under shear deformation and concluded that cluster diffusivity increases up to two orders of magnitude with increasing cohesion. In their work, they proposed a new relationship between cluster size, its lifetime and cohesion for two dimensional flows under shear induced deformation.

Cohesion level and shape of the particles have a prominent effect on the regime transition. In the case of the latter, transition between the quasi-static regime and the intermediate regime occurs for smaller values of the shear rate and higher stress values. On the other hand, if cohesion is first introduced and then increased for spherical particles, the average level of stress increases for the same system, while the regime transition between the quasi-static and the intermediate flow regimes gradually disappears for increasing cohesion levels. Transition to the rapid flow regime occurs for the same shear rate.

In conclusion, particle shape influences the angle of friction in bulk failure of particles and the presence of vertices and edges in faceted shapes strongly influences the resistance to shear deformation. Approximating real crystal shapes by truncated polyhedron shapes provides a close match in the shear deformation behaviour between the two shapes, as shown for Paracetamol crystals by Vivacqua et al. (2019). It appears that cohesion affects the incipient yield but does not influence bulk shear viscosity.

3.4 Powder mixtures

Formulated powder mixtures have long been used in industry to provide product innovations as well as overcoming poor product performance due to segregation, powder flow problems, tableting issues and instabilities. For example, multi-component detergent powders containing bleach activator and enzymes are prone to segregation. Issues related to tableting and tablet strength prevail in the pharmaceutical industry and are commonly resolved by formulating the powder. Individual powder components have their own flow characteristics and their mixing ratio not only affects the product performance but also the flow behaviour of the powder mixture. When the overall mixture is too cohesive, the powder mixture flows poorly and could be very difficult to handle in the required processes (e.g. discharge from silo). On the other hand, if it is very free flowing, powder segregation could result in non-uniformity of the product from batch to batch. To address extreme cohesivity, the surfaces of cohesive active ingredients may be coated with very fine powder, acting as spacers to improve the overall flow behaviour of the powder (Fulchini et al., 2017).

There have been several studies looking at improved

flowability of pharmaceutical active ingredients (APIs) using dynamic powder flow testers. Qu et al. (2015) reported that coating the surface of cohesive ibuprofen with flow aids (i.e. silica or magnesium stearate) improves the flowability, as measured by the FT4 rheometer aeration test. In their investigation, the total energy measured by the rheometer for raw cohesive ibuprofen was considerably higher than for those coated with flow aids, for a wide range of air velocities (0 to 30 mm/s).

In recent years, the interest in predicting the flow behaviour of multi-component mixtures has been increased. In a recent work for example, Capece et al. (2015) developed a methodology for averaging the granular Bond number for a powder mixture and linking that to the flow function coefficient as measured by shear cell testing. Their approach is applicable to the onset of flow. However, most of the reported works in the literature for dynamic powder flow are based on empirical approaches. In an ongoing research programme entitled Virtual Formulation Laboratory, supported by the Engineering and Physical Sciences Research Council, UK, (<https://gow.epsrc.ukri.org/NGBOViewGrant.aspx?GrantRef=EP/N025261/1>), the dynamic flow behaviour of cohesive powder mixtures is under investigation based on the single particle properties of the formulation components. Following a similar approach to that of Capece et al. (2015) it is shown that by averaging the Bond numbers of individual components with different cohesion/ adhesion, size and density, the rheological characteristics of the mixture, i.e. bulk friction coefficient and apparent bulk viscosity can be predicted as a function of the shear strain rate, expressed in the non-dimensional form by the inertial number.

4. Conclusions

For characterisation of cohesive powder flow under dynamic conditions only a handful of devices are available, namely the FT4 Powder Rheometer, Anton-Paar Powder Cell and Couette device. They act as a rheometer for powders, measuring the rheological response of powder under varying shear strain rates. Experimental classification of the flow regime has so far been established with the Couette device, but wall slip and the presence of secondary flows pose a challenge to its use, in addition to lack of commercial availability. For rheometers the underlying relationship between the torque and bulk rheological properties, such as bulk friction and apparent shear viscosity, is imperative and has yet to be established.

Other dynamic testers include the rotating drum and ball indentation method. The rotating drum is applicable to free surface flows and does not have the versatility to control applied stresses; furthermore, it is difficult to

interpret a relevant cohesion parameter from such tests. Ball indentation is a relatively new test method which allows stress to be controlled and a range of strain rates to be applied by manipulating indenter properties. However, its operating range is narrow, due to the limited acceptable indentation depth.

An increasingly important sector dealing with powders operated at high strain rates is additive manufacturing (3D printing). There is currently great effort being put into relating the spreading performance in additive manufacturing to various flow testers. However, in contrast to flow resistance in shear flows, additive manufacturing processes utilise a very thin powder layer in close proximity to wall boundaries. As such the flow behaviour is strongly influenced by transient jamming/arching. A different approach is needed to describe the rheology of spreading of thin powder layers for the increased demand of powder spreading in additive manufacturing.

The prediction of bulk rheological characteristics of fine cohesive powders is still a grand challenge. Such powders are in dynamic cluster forms, the size and packing density of which depends on the stress history that the powder has experienced. The current research effort is aimed at addressing the influence of particle properties on bulk rheological behaviour in a systematic way, but there is still a long way to go to develop fully predictive tools.

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Colin Hare is a Lecturer of Chemical Engineering at the University of Surrey, UK. His expertise are in powder flow, mixing/segregation, coating, particle breakage and the Discrete Element Method. His research is primarily funded by industry and addresses secondary processing of powder products, considering the role of particle properties and process conditions on product performance.



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I graduated in Chemical Engineering from Università della Calabria (Italy), where I also completed my PhD in 2011. I have carried out postdoctoral research work at several international institutions, namely the University of Leeds (UoL), Nottingham (UoN), British Columbia (UBC), Università della Calabria (UNICAL) and Qatar University (QU). I am currently Particle Engineering Senior Scientist at Johnson Matthey, Billingham, UK. My research interests are related to several areas of particle technology and multiphase flow, including powder flow, powder compaction, DEM modelling, fluidization and electrocoalescence.



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