Prediction of bulk particle breakage due to naturally formed shear bands

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Abstract. In many particle processes there exist both stagnant and flowing regions. Shear bands naturally form between these regions as a transition from zero to full velocity. Particles within shear bands are prone to breakage through surface erosion, chipping and fragmentation when exposed to substantial shear stresses. The precise nature and extent of breakage is difficult to describe mechanistically, due to the distribution of particle strengths and forces acting upon them. Consequently the breakage of particles within shear bands is typically defined empirically using an attrition shear cell. The use of such empirical relationships to predict breakage in larger processes is severely limited in the literature.

This paper describes a method of predicting bulk breakage in an agitated vessel by establishing particle breakage caused by applied stress and strain in a shear cell. The Distinct Element Method (DEM) is utilised to estimate the distribution of stresses and strains within the agitated bed, this distribution is then applied to the breakage relationship to predict total attrition. The DEM analysis requires a number of measurement cells to be considered within the bed. It is imperative that the dimensions of these cells are comparable to that of a naturally occurring shear band. With this measure in place the method outlined here describes the experimental breakage well. This method can be applied to any system where particles break due to shear deformation.

Keywords: Attrition, Breakage, Shear, DEM.
PACS: 83.80.Fg, 89.20.Bb

INTRODUCTION

In many particle processes the movement of particles relative to each other is inevitable. In the transition from relatively stagnant regions to more rapidly flowing regions of a bed, shear bands naturally develop. These shear bands are typically of the order of 5 – 10 particle diameters in length [1]. The contact forces that exist within these regions can lead to significant damage to the particles, ultimately resulting in an undesirable product and reduced efficiency of the process. Several empirical relationships have been developed to describe particle breakage as a function of applied stress and strain [2-4], using an attrition shear cell similar in design to that introduced by Paramanathan and Bridgwater [5].

An example process in which damage occurs due to shear deformation is in agitated drying of active pharmaceutical ingredients (APIs). Following the crystallisation of an API the slurry is commonly filtered and then particles are dried in an agitated contact dryer. Moisture is evolved from the bed by heat supplied to the vessel walls, whilst the bed is gently agitated by a vertically aligned impeller to ensure the remaining moisture is evenly distributed throughout the bed. However this agitation leads to breakage of the API [6], which in some cases may be so pronounced that agitated contact drying is not a viable process route for production. It is therefore of interest to develop a method for predicting the extent of breakage that would occur during the drying process for a given material, prior to production. This paper introduces a method for predicting breakage in agitated drying, which is applicable to all processes where breakage occurs due to shear deformation.

The extent of breakage during the early stages of drying, when the moisture content is relatively high, is negligible. When the moisture content is low the shear resistance of the bed increases, thus resulting in a significant increase in the extent of breakage [7, 8]. For this reason a completely dry bed is utilized in this work, which represents the end of the drying process.

PREDICTION METHOD

In order to develop and test the predictive method for determining breakage due to agitation, a small-scale vessel is used (Figure 1). The vessel has an internal diameter of 94 mm, whilst the impeller diameter is 90 mm, with a blade height of 18 mm. A 250 g bed of 500 – 600 µm Paracetamol particles is placed into the vessel, thus providing a bed height of approximately 50 – 55 mm. The vessel and bed dimensions are geometrically similar to an industrial process, whilst the clearance between the impeller and vessel wall is sufficiently large to ensure particles are not crushed in this region [9], therefore the resulting breakage is caused by the shear deformation of the

bed. The bed is agitated at a constant impeller speed for a range of rotational times. After each individual experiment the powder is sieved to assess the extent of breakage, which is deemed to be the percentage of material that passes through a 355 μm sieve, given the above feed size.

In order to predict the extent of breakage from the shearing of Paracetamol in the agitated vessel, the stresses and strain rates in the bed are estimated by DEM. The extent of attrition of the material under varying conditions of stress and strain is established empirically in an annular shear cell, following the method of Neil and Bridgwater [3].

FIGURE 1. Agitated Vessel

DEM STRESS AND STRAIN ANALYSIS

The agitated bed of Paracetamol is simulated using PFC3D (developed by Itasca). The particles are represented by poly-disperse spheres, with median diameters, d_{med}, of 2, 3 and 4 mm in separate simulations, have sliding and rolling friction coefficients of 0.5 and 0.1, respectively. A linear spring contact model is used, with normal and shear stiffnesses of 10 and 8.3 kN, respectively. These particle properties have been shown to provide an adequate representation of the actual agitated bed [10].

The stresses and strain rates are assessed within a sector of the simulated bed for one complete rotation, once the bed has reached a steady-state. This measurement sector is divided radially and vertically into an array of measurement cells, as shown in Figure 2. The stresses and strain rates are assessed using sector sizes (θ_{sec}) of 10, 25 and 40°, and 2, 3 and 4 cell divisions (n_d). At intervals of approximately 1° of impeller rotation, the deviatoric stress, τ, and the strain rate, γ, are assessed using equations 1 and 2, respectively.

$$\tau = \frac{\sqrt{\left(\sigma_{xx} - \sigma_{yy}\right)^2 + \left(\sigma_{xx} - \sigma_{zz}\right)^2 + \left(\sigma_{yy} - \sigma_{zz}\right)^2}}{\sqrt{6}} \quad (1)$$

$$\gamma = \frac{1}{2}\sqrt{\left(\frac{\partial v_\theta}{\partial r} - \frac{v_\theta}{r}\right)^2 + \left(\frac{\partial v_\theta}{\partial z}\right)^2} \quad (2)$$

where σ is the normal stress acting in direction i on face i, v_θ is the particle velocity in the rotational direction of the impeller, and r and z are radial and vertical positions, respectively.

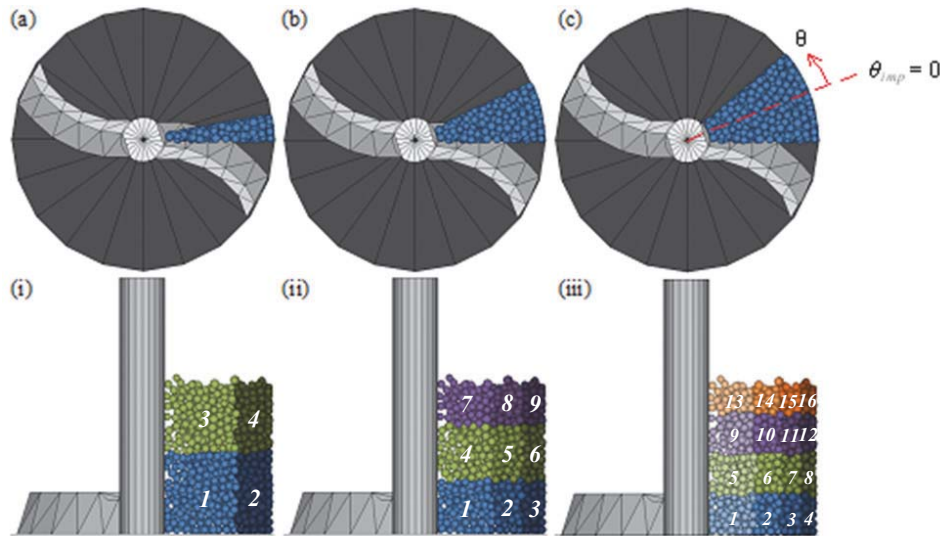**FIGURE 2.** Measurement sectors and cells (a) $\theta_{sec} = 10°$ (b) $\theta_{sec} = 25°$ (c) $\theta_{sec} = 40°$, (i) $n_d = 2$ (ii) $n_d = 3$ (iii) $n_d = 4$.

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Stress Distribution

The deviatoric stress variations with impeller rotational position, θ_{imp}, are shown for cell 3, the outer-most bottom cell, when $\theta_{sec} = 25^{\circ}$ and $n_d = 3$ in Figure 3. When θ_{imp} equals 0, 180 or 360° the impeller is centrally aligned within the measurement sector, as the impeller rotates anti-clockwise θ_{imp} increases. Figure 3 shows that the stress is at a maximum in the vicinity of the blade and decreases in the wake of the blade. The stress is negligible far from the blade and increases from approximately 60° in front of the impeller.
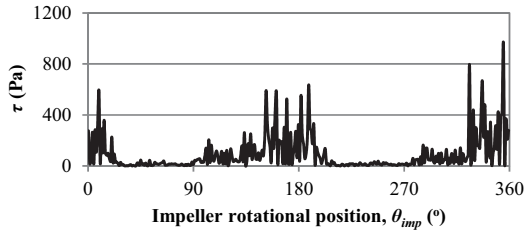

FIGURE 3. Deviatoric stress in cell 3 for $\theta_{sec} = 25^{\circ}$ and $n_d = 3$.

The average deviatoric stresses throughout one full impeller rotation in each measurement cell are compared in Figures 4, 5 and 6 for $n_d = 2$, 3 and 4, respectively. For all values of n_d the stresses are greatest at lower positions in the bed, and increase radially. The stresses obtained in this analysis generally increase when n_d is increased, therefore it is important to specify the appropriate cell size and number..
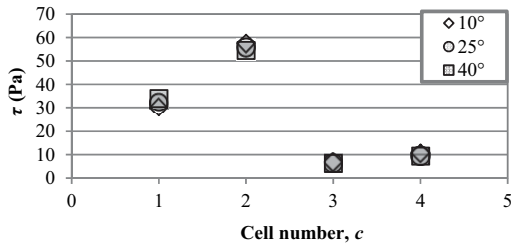

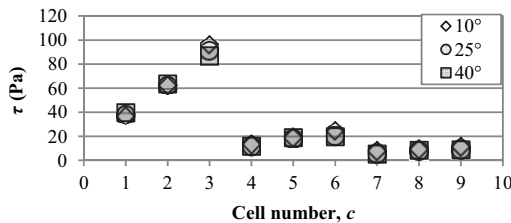**FIGURE 4.** Deviatoric stress distribution for $n_d = 2$.

FIGURE 5. Deviatoric stress distribution for $n_d = 3$.
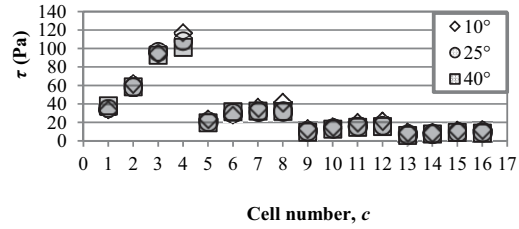

FIGURE 6. Deviatoric stress distribution for $n_d = 4$.

ATTRITION PREDICTION

The attrition of 500 – 600 µm Paracetamol particles was measured under controlled conditions of stress and strain in an annular shear cell. The extent of attrition, W, defined as the mass fraction forming the debris, was determined by classifying product material with a sieve size < 355 µm to be debris. The relationship of W to applied stress and strain was established following the approach of Neil & Bridgwater [5], for stresses of 0.1 – 3.0 kPa and strains of 30 – 240.

$$W = K_N \left[\left(\frac{\tau}{\tau_{ref}} \right) \Gamma^{\varphi} \right]^{\beta} \quad (3)$$

where K_N, φ and β are empirically determined constants, τ_{ref} is a reference failure stress and Γ is applied strain. For 500 – 600 µm Paracetamol K_N, φ and β are 0.71, 1.28 and 0.49, respectively, τ_{ref} is taken as the failure stress, which is 0.183 MPa for the Paracetamol [10].

The attrition within the agitated bed is predicted by applying equation 3 to the stresses and strains obtained from each measurement cell at each time interval, consequently the predicted value of attrition in cell c, $W_{c,n}$, and throughout the entire bed, W_I, after I rotations, are given by equations 4 and 5, respectively.

$$W_{c,n} = K_N \left[\frac{\tau_{c,n}}{\tau_{ref}} \right]^{\beta} \left[\sum_{1}^{n} \left(I \cdot \Gamma_{c,n} \right)^{\varphi\beta} - \sum_{1}^{n-1} \left(I \cdot \Gamma_{c,n} \right)^{\varphi\beta} \right] \quad (4)$$

$$W_I = \sum_{1}^{c} \sum_{1}^{n} \chi_{c,n} \cdot W_{c,n} \quad (5)$$

where $\chi_{c,n}$ is the mass fraction in cell c at time interval n. The predicted extents of attrition after one impeller rotation, $W_{I=1}$, are given for the cases of $d_{med} = 3$ mm and $\theta_{sec} = 25^{\circ}$ in Tables 1 and 2, respectively.

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TABLE 1. Predicted attrition after one impeller rotation, $W_{I=1} \times 10^2$ (%) for d_{med} = 3mm

θ_{sec} (°)	n_d		
	2	**3**	**4**
10	0.99	1.21	1.28
25	0.97	1.14	1.24
40	0.96	1.11	1.13

TABLE 2. Predicted attrition after one impeller rotation, $W_{I=1} \times 10^2$ (%) for θ_{sec} = 25°

d_{med} (mm)	n_d		
	2	**3**	**4**
2	0.80	0.95	0.97
3	0.97	1.14	1.24
4	1.08	1.35	1.43

The predicted attrition increases when particle size or the number of cell devisions is increased or when the sector size is decreased. All of these changes result in a decrease in the average number of particles within a measurement cell, $N_{p,ave}$, therefore the deviatoric stress approaches that of a single sphere. Figure 7 shows the predicted attrition after one impeller rotation as a function of $N_{p,ave}$.
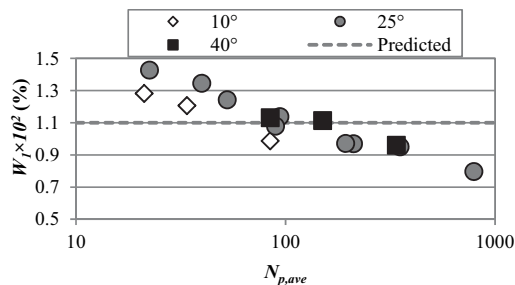

FIGURE 7. Predicted attrition after one impeller rotation for various θ_{sec}, n_d and d_{med}.

Figure 7 shows that the predicted attrition is dependent on the average number of particles per measurement cell; decreasing as $N_{p,ave}$ increases. The initial cell arrangement adopted by Hare *et al.* [5] of d_{med} = 3mm, θ_{sec} = 25° and n_d = 3 provides a good prediction ($W_{I=1}$ = 0.0114). Under this arrangement the dimensions of the measurement cell are around 4 to 8 particle diameters in each direction. This is comparable to the width of a naturally occurring shear band, and the bed adopted in the shear cell experiments utilized in this prediction. Therefore this prediction approach provides a good agreement with the experiments, providing the measurement cells approximate the length-scale of the naturally forming shear band phenomena. If the measurement cells are too small or too large then the attrition will be over or underestimated, respectively.

CONCLUSIONS

The attrition caused by shear deformation within an agitated bed has been predicted by analyzing the stress distribution using DEM and coupling this with an attrition function derived from shear cell experiments. The method provides a suitable prediction if the dimensions of the measurement cells in the DEM analysis are representative of the natural shearing phenomenon. If the measurement cells are too small then the attrition is overestimated, whereas it is underestimated if the measurement cells are too large. DEM is frequently used to estimate internal bed stresses, however the dimensions of the measurement regions are often selected arbitrarily. The findings of the attrition prediction method developed here have far reaching implications for the assessment of internal bed stresses, as the length-scale of the averaging procedure is of paramount importance.

ACKNOWLEDGMENTS

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