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Prediction of Attrition in Agitated Particle Beds

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Abstract

The majority of pharmaceutical powders produced through crystallisation are dried in agitated dryers. The rotation of the impeller causes shear deformation of the bed, which enhances the drying rate, but also leads to particle breakage. A method of predicting the extent of breakage occurring due to agitation is described and applied for Paracetamol in a small-scale dryer. The distributions of stresses and strains in the bed are estimated using the Distinct Element Method (DEM). The information obtained here is then coupled with the measured attrition of Paracetamol in an annular shear cell in order to predict the attrition in the agitated bed. The experiments are carried out on dry material so as to establish purely the effect of stresses and strains on attrition, whilst keeping moisture content and temperature constant.

The shear cell provides uniform condition for stresses and strains so that the breakage taking place under relatively well-defined conditions is quantified. In contrast, the prevailing shear stresses and strains in the agitated bed have wide distributions, as little shearing takes place near the impeller shaft, whilst there are considerable shearing stresses near the impeller tip. Therefore the bed is divided into a number of segments for which the extent of attrition can be evaluated for each segment, based on the shear cell data. A good quantitative agreement is found between the predictions and experimental results obtained for the attrition of Paracetamol in the small scale dryer. The resulting prediction also suggests that, for a given number of impeller rotations, the extent of breakage is independent of impeller speed in the range tested (20 – 78 rpm). This is expected as the prevailing strain rates are too low for the inertial effects to be dominating and the shear stresses are independent of shear rates within the range investigated. The attrition prediction suggest that over half of the attrition occurs in the bottom third of the bed, with increased attrition at greater radial distances. The attrition is also predicted to occur predominantly 30^{o} in front of and behind the impeller.

1. Introduction

The majority of active pharmaceutical ingredients (APIs) are produced through crystallisation to generate a product of high purity. After crystallisation the liquor is removed from the API particles, forming a wet mass, which is washed with solvent to enhance API purity. The removal of the liquor and washing is often carried out using a filter dryer The bulk liquor is filtered through the base of the dryer, and the remaining moisture is then driven off by the application of heat to the vessel walls. An impeller agitates the bed to enhance the drying rate and to prevent the occurrence of hot-spots, which could adversely affect this portion of the bed by over-heating, whilst leaving the rest of the bed relatively moist. The agitation leads to unwanted breakage (attrition) of the particles, which results in their degradation and deviation from desired particle sizes and possible changes in surface chemistry, leading to subsequent manufacturing problems.

There is little reported work in the literature on agitated bed drying of pharmaceutical particles, whilst significant problems are often experienced with new APIs (Kukura et al., 2005). The only significant work is due to Lekhal et al. (2003 and 2004). Lekhal et al. (2003) analysed the extent of attrition of cubic KCl crystals when agitated in a small-scale dryer. They showed that for the operational range chosen, the impeller speed had negligible effect on the extent of attrition, which instead could be related to the number of impeller rotations. The breakage was shown to be minimal at the early stages of drying, however once a particular moisture content was reached, around 2% in this case, the attrition rate increased dramatically. Lekhal et al. (2004) showed that the attrition of needle-like l-threonine in the same vessel followed a similar trend, though the increase in attrition began at a moisture content of 4%. This moisture content, below which attrition is greatly increased, could relate to the time when the surfaces of the particles in the bed no longer have complete moisture coverage. For the needle-like particles a lack of complete moisture coverage would occur at a higher moisture content because the specific surface area is greater. Consequently, a higher degree of bulk cohesion would exist, leading to an increased shear stress within the bed (and hence, increased torque on the impeller), and consequently the attrition rate increases.

The above findings are useful in understanding breakage in agitated dryers; however new APIs are developed frequently, and during the early stages of development only a limited amount of material is available. Therefore it would be beneficial to be able to predict the breakage behaviour of a given material in an agitated dryer with a relatively small sample. It is proposed that this could be achieved by analysing the breakage behaviour of a small sample of the material in a well defined, bulk system that could represent the local behaviour of the dryer bed. The breakage in the dryer is predominantly caused by shear deformation of the bed as the material in front of the blade is swept upwards and then falls into the wake region behind the impeller. The bed has regions of transient flow and stagnation, resulting in distributions of shear stresses and strains. Therefore the bed behaviour subjected to the compressive and shear stresses and strains throughout all transient states must be analysed in order to predict the attrition occurring therein.

In this work the Distinct Element Method (DEM) is utilised to simulate the dynamics of particle motion in the agitated bed, from which the distributions of stresses and strains are evaluated. The information obtained here is then used in an experimental programme, whereby the attrition of the material under these conditions is measured in an annular shear cell (introduced by Paramanthan and Bridgwater, 1983a) under constant moisture content and temperature. This provides material response characteristics under well-defined conditions of shear stress and strain. By coupling this information with the distribution of stresses and strains obtained from the DEM, a prediction of attrition is obtained. In a parallel experimental work, the actual attrition in a small-scale dryer is analysed to enable a comparison to be made with the simulation predictions in order to evaluate the suitability of this technique to predict attrition under such complex conditions. The scope of the work reported here is outlined in Figure 1. We analyse the case of attrition in a dry bed, representing the end of the drying process, and no heat is applied to the vessel. This allows the effect of the stresses and the flow in the vessel on the extent of attrition to be first established, before the coupled effects of temperature and moisture content could be analysed.

2. Materials and Methods

The attrition of Paracetamol (Form I), supplied by Rhodia, Saint Maurice, L'Exil, France, is assessed as a model material. This form has a monoclinic crystal structure with an aspect ratio of approximately 1.4 and fails in semi-brittle mode (Bentham et al., 2004). To be able to analyse breakage, it is necessary to use a feed material with a narrow size distribution, as the extent of breakage is dependent on particle size. It is also preferable to use sufficiently large crystals to be able to measure breakage easily. The mass of material required for the experimental work dictates that size analysis should be carried out by sieving; this is achieved using a Haver & Boecker EML 200 digital plus T sieve shaker. The Paracetamol crystals used here are from the sieve fraction 500 – 600 μm. It is important to note that the size classification by sieving refers to the second largest dimension of a particle (Allen, 1975); as such the Paracetamol crystals are approximately 1 mm in length, as analysed by optical microscopy.

For the DEM simulations, the Paracetamol particles are represented by spherical particles, as otherwise the simulation time becomes too long. However, the contact properties are chosen such that they replicate the material bulk behaviour, such as the torque and velocity field. This approach enables the distribution of stresses and strain rates that give rise to the torque acting on the impeller, as measured experimentally, to be estimated. The Paracetamol particles can then be subjected to such conditions of stress and strain in the shear cell to quantify their extent of attrition.

The current computer memory and power limitations do not allow the bed to be simulated in a reasonable time with actual particle size. Therefore, the particles are scaled up to a median size of 3 mm. However, the particle density of the simulated particles is set so that the total mass of the simulated bed is equal to that of the real bed, following the approach of Hassanpour et al. (2009). The simulated bed height is also equal to that of the real particles, consequently the simulated particle density is identical to the real particle density in this case. The simulation particles are generated with a Gaussian distribution, and a span equal to that of the Paracetamol feed sieve size range (resulting in diameters of 2.73 – 3.27 mm).

The coefficient of restitution, e, of the Paracetamol particles has been quantified by high-speed video recording and motion analysis. A sample of 25 particles were individually dropped onto a stainless steel platen and their incident and rebound velocities estimated from the video recordings. Due to the non-spherical nature of the Paracetamol particles, the rebound direction was rarely normal to the platen, therefore, the magnitude of resultant rebound velocity was used. From this approach the average coefficient of restitution of the Paracetamol particles was found to be 0.42. Damping is used in the equation of particle motion in the DEM to account for inelastic energy losses. Tsuji et al. (1993) proposed equation 1 & 2 to relate damping to coefficient of restitution. Using this approach, the coefficient of restitution for Paracetamol results in a damping coefficient, ζ, of 0.27 in the simulations. The properties of the experimental and simulated materials are shown in table 1

$$\zeta = \frac{\alpha}{\sqrt{1+\alpha^2}} \tag{1}$$

where,

$$\alpha = -\frac{1}{\pi} \cdot \ln e \tag{2}$$

The force-displacement behaviour of the Paracetamol particles was established using an Instron 5566 series mechanical testing machine. 25 Paracetamol particles were individually subjected to quasi-static compression by a flat piston. Each particle was placed onto the piston on its most stable face, then compressed by the piston at a rate of 1 mm/min. The force-displacement behaviour of three of these particles is shown in Figure 2. A linear spring and dashpot model (Cundall and Strack, 1979) is used in the simulations here, where the stiffness represents the mechanical response of the Paracetamol particles up to a displacement of 1 %. Following the model developed by Thornton and Yin (1991) for a contact between two particles with equal shear modulus and Poisson’s ratio the shear stiffness, K_s, is related to the normal stiffness, K_n, by equation 3.

$$K_s = \frac{(1+\upsilon)\cdot(2-\upsilon)}{2(1-\upsilon^2)} \cdot K_n \tag{2}$$

This approach is used to calculate the shear stiffness of Paracetamol for the simulations, for which v is taken as 0.3 (see table 1).

A photograph of the agitated bed used in this work is shown in Figure 3. It is geometrically similar to an industrial dryer. The glass vessel is cylindrical with a diameter of 94 mm, containing a stainless steel, double-bladed, retreat-curve impeller with a diameter of 90 mm. The blades have an inclination of 60^o to the horizontal, and a height of 20 mm. A porous white plate forms the base of the vessel.

The bed height is 50 – 60 mm to maintain geometric similarity with the industrial scale. In the case of Paracetamol, this requires 250 g of material in each experiment. The impeller is rotated at a constant speed for a given time. In order to use impeller speeds that are representative of the industrial scale, the following scaling law is used:

$$\frac{\omega_1}{\omega_2} = \left(\frac{D_2}{D_1}\right)^{\alpha} \tag{3}$$

where ω is the impeller speed and D the diameter of the blades, in scales represented by subscripts 1 and 2, and a is the scaling power. For scaling using constant Froude number, $\alpha = 0.5$, whilst for keeping the tip speed constant, $\alpha = 1.0$. Tardos et al. (2004) suggested that for scaling up of high shear granulators, it is best to keep the shear stress constant, giving $\alpha = 0.8$, subject to impeller geometry. These three scaling laws result in impeller speeds of 20, 45 and 78 rpm in the small-scale vessel. The attrition at these three speeds is measured at three rotational times in separate experiments, so as to investigate the effect of strain rate and total strain within the range of operational impeller speeds. The rotational times of 5, 20 and 80 minutes are selected to ensure the extent of attrition is sufficient to be measured with good statistical reliability.

The extent of attrition under shear deformation is analysed using an annular shear cell, shown schematically in Figure 4. The shear cell design is based on the work of Paramanathan and Bridgwater (1983a), developed for analysing attrition and has been manufactured by Ajax Equipment Ltd, Bolton, UK. The cell has inner and outer diameters of 120 and 160 mm respectively, resulting in an annular width of 20 mm. The material is placed in the cell and gripped by groove rings to prevent slippage against the cell base. The lid, also containing groove rings, is lowered onto the bed surface to apply a normal stress to the bulk material. The applied load is adjusted by the use of an appropriate counterbalance, based on a technique introduced by Bridgwater et al. (2003). The cell base is then rotated at 10 rpm, whilst a stopper arm attached to the cell lid prevents its rotation – thus generating a shear profile across the height of the bed. Once a pre-specified strain has been achieved, the cell rotation is ceased. The prevailing shear stress causes attrition of the particles, the extent of which is known to be dependent on the magnitude of this stress and the applied strain (Paramanathan and Bridgwater, 1983b; Ouwerkerk, 1991; Neil and Bridgwater, 1994; Ghadiri et al., 2000).

The effect of stress and strain on attrition is complex, as the local contact forces experienced by the particles are dependent on their position and orientation within the bed. Attrition occurs as a result of loading conditions and the motion of particles. At the onset of shearing, the bed must expand to allow movement; this necessary dilation consumes energy and causes immediate breakage. After this dilation the bed is looser and easier to move. Throughout the shearing process, there exist local regions where the forces acting on the particles are greatest. The distribution of the contact force magnitude is very wide and difficult to predict exactly as it is highly dependent on the bed fabric (Antony and Kuhn, 2004). The force anisotropy is likely to be greater for non-spherical particles (Azéma et al., 2009). When breakage occurs, there is a sudden change in the distribution of contact forces in the vicinity of the broken particle, fines resulting from the breakage event may percolate through the particles below. As the bed is sheared further, the contact forces and the bed fabric continually evolve. In light of this highly complex behaviour in the bed, no mechanistic models have been developed to describe attrition occurring through shearing. Consequently, several authors have applied empirical formulae to describe the attrition. Gwyn (1969) analysed attrition in a fluidised bed and found the extent of attrition, W, is given by equation 5.

$$W = k_G \cdot t^{m_G} \tag{4}$$

where t is time, k_G and m_G are attrition constants. This equation was found to suitably describe attrition in a shear cell by Bridgwater (1989); however this approach does not take into account the effect of applied normal stress. Ouwerkerk (1991) found that the attrition of catalyst carrier beads could be best described by

$$W = k_O \cdot \left[\left(\frac{\sigma^2}{\sigma_{ref}} \right) \cdot \Gamma \right]^{m_O} \tag{5}$$

where σ is the applied stress, σ_{ref} is a reference stress that describes a characteristic value, for example the crushing strength of the particles, Γ is the strain, k_O and m_O are fitting parameters. Neil and Bridgwater (1994) and Ghadiri et al. (2000) found that the relative influence of strain and stress is better described by equation 7.

$$W = k_N \cdot \left[\left(\frac{\sigma}{\sigma_{SCS}} \right) \cdot \Gamma^{\varphi} \right]^{\beta} \tag{6}$$

where σ_{SCS} is the side-crushing strength of the material, k_N is the proportionality constant, β is the power index and φ expresses the relative influence of strain to stress on attrition. This approach is capable of providing a good correlation for a wide variety of materials (Bridgwater et al., 2003), since three fitting parameters are used, although it cannot be predictive.

3. Simulation of the Agitated Bed and its Validation

In order to use the stress and strain distributions in the dryer from the DEM simulations for attrition predictions, it is necessary to first validate the simulations to ensure the behaviour is representative of the experiment. To do this, two features (one macroscopic and one at single particle level) are monitored and compared, the torque and particle velocity. The torque acting on the impeller is measured in the experiments and calculated in the simulations; if the torque – an indication of the shear stress present in the bed – is comparable, then the magnitude of the stresses within the bed can be assumed to be well-represented by the simulation. The strain rates obtained from the simulation are validated experimentally using Positron Emission Particle Tracking (PEPT) (Hassanpour et al., 2009); this has been carried out and reported by Hare (2010). A brief summary of the comparison between the PEPT and DEM results is given in Appendix A.

The torque acting on the impeller at 20, 45 and 78 rpm is shown in Figure 5 for the experimental and simulated cases. The torque is independent of impeller speed within the range of speeds tested; this is in agreement with the conclusions of Tardos et al. (2003), who showed that the shear stress is independent of strain rate at dimensionless shear rates less than about 0.15 – 0.25; in this case the dimensionless shear rate at 78 rpm is 0.05. An increase in either sliding or rolling friction coefficients results in an increase in the angle of internal friction (Gröger and Katterfeld, 2006), and therefore an increase in the torque acting on the impeller. The friction coefficients used in the simulations in this work are adjusted to provide the closest agreement in torque with the experiments. The internal angle of friction of Paracetamol was measured using a Schulze shear cell at a preconsolidation stress of 10 kPa and major principal stresses of 2, 4, 6 and 8 kPa. The internal angle of friction was found to be 37^{o}, which corresponds to a friction coefficient of 0.7. If this frictional behaviour was entirely due to sliding friction, a sliding coefficient of friction equal to 0.7 would be suitable in the simulations; however, rolling friction also contributes to this bulk friction value. It is difficult to measure the individual contributions of sliding and rolling friction to this bulk friction value, therefore, an approximation is made. The use of a rolling friction in the DEM means that the sliding friction coefficient must be less than 0.7 in order to maintain a suitable internal angle of friction. Here the sliding friction is set to 0.5, as its influence was considered to be greater than that of rolling friction. However, the contribution of rolling friction is not trivial. The rolling friction is adjusted to obtain a similar torque to that seen in the experiments; this similarity occurs with a rolling friction coefficient of 0.1 with an impeller speed of 78 rpm. The fluctuations in the torque obtained from the simulations are much greater than from the experiments. This may be because only 13,500 particles are present in the simulated bed, whereas millions are present in the experimental case. A slight variation in a few contacts in the simulation would result in a notable change to the torque. Another contributing factor may be the greater sampling frequency in the simulations. For this reason, the moving average of the torque over a period of 25^{o} of rotation has been included in Figure 5. A good agreement is obtained for the other impeller rotational speeds, i.e. 20 and 45 rpm, as shown in Table 2. Figure 5 shows that the torque in the simulation at 78 rpm peaks after approximately 0.05 impeller rotations. This is because the impeller speed in the simulations is set to its final value at the start of the rotation. However, in the experiments the impeller reaches its final speed after a period of about 5 seconds, hence the presence of the initial peak in the simulation torque, but not in the experimental torque.

Different combinations of sliding and rolling friction coefficients may lead to the same internal angle of friction of the bed, and hence the calculated torque. Therefore, it is prudent to check other dynamic characteristics of the bed. In this work, the velocity distributions within the simulated and real particle beds are also compared, with the latter measured by Positron Emission Particle Tracking (PEPT), using the facility of the University of Birmingham (Parker et al., 1993) . The results are reported in Appendix A, and provide further evidence for validation, since the particle velocities estimated by the DEM compare well with those measured by PEPT. Therefore, the stresses and strain rates predicted by DEM can be considered to be valid, despite assuming larger particles having a spherical shape, and will be applied to the shear cell attrition measurements.

4. Stress and Strain Rate Analysis

The stresses and strain rates vary in the dryer bed both spatially and temporally. In order to assess distributions of states of stress and strain rate, a sector of the bed is considered, as shown in Figure 6a. This volume, within which the stresses and strain rates are calculated, is referred to as 'the measurement sector'. It is divided both radially and vertically into a number of cells with equal volume (Figure 6b) for which the stresses are calculated at each time interval. Within each cell, the forces acting on each particle whose centre is inside this cell are obtained to estimate the normal stresses:

$$\sigma_{\mathrm{ii}} = \frac{1}{\mathrm{V}} \sum_{1}^{\mathrm{N}} F_{\mathrm{ii}} \cdot r_{\mathrm{p}} \tag{7}$$

where σ is stress, V is the volume of the cell, N is the number of particles in the cell, and F is the force acting on a particle in direction ii with radius r_p (Bag, 1996). The deviatoric stress, τ_D, is calculated from the three normal stresses using equation 9 (Luding, 2008).

$$\tau_{\mathrm{D}} = \frac{\sqrt{\left(\sigma_{\mathrm{xx}} - \sigma_{\mathrm{yy}}\right)^2 + \left(\sigma_{\mathrm{xx}} - \sigma_{\mathrm{zz}}\right)^2 + \left(\sigma_{\mathrm{yy}} - \sigma_{\mathrm{zz}}\right)^2}}{\sqrt{6}} \tag{8}$$

The calculations are carried out throughout one complete impeller rotation. The impeller is rotated twice prior to these calculations to ensure the bed has reached a steady state, as indicated by the consistent torque record beyond this point (Figure 5). This provides an average stress for each of the cells. The velocity in the direction of impeller motion (v_θ) is analysed at the vertical and radial boundaries of each cell to obtain the relevant strain rate tensor, γ.

$$\gamma = \frac{1}{2}\sqrt{\left(\frac{\partial v_\theta}{\partial r} - \frac{v_\theta}{r}\right)^2 + \left(\frac{\partial v_\theta}{\partial z}\right)^2} \tag{9}$$

In this case, each cell contains an average of approximately 100 particles; this was deemed sufficiently large to give an average that is not so dependent on individual particles, and small enough to allow the distribution of stresses and strain rates with radial and vertical position to be considered. The dimensions of these cells are equivalent to 4 – 8 particle diameters in each direction, which is comparable to the width of the shear band in the shear cell experiments. As the number of cells is increased in such an analysis, the resolution of the variations with location is enhanced. However, this is only valid up to a limit, beyond which the averages obtained are highly dependent on individual particles, and so are less meaningful.

The stress distribution throughout cells 2, 5 and 8 in the measurement sector, at 20, 45 and 78 rpm, are shown in Figures 7, 8 and 9, respectively. An impeller rotation of zero represents the moment when the tip of one of the impeller blades touches the front of the measurement sector, indicated in Figure 6a. The variation of stresses with impeller position is largely similar at all three impeller speeds investigated. Figures 7 and 8 show that, in the lower two thirds of the bed, the shear stress is low in the wake of the impeller blade (at approximately 20 – 80^o rotation), but increases when the second blade of the impeller approaches the measurement sector. The forces developing from the impeller propagate through a force chain some way into the bed in front of the impeller blade, thus explaining this behaviour. The stress continues to rise as the impeller rotates, peaking at an impeller rotation of approximately 150 – 180^o, slightly before the impeller blade reaches the measurement sector. The stress then decreases, as the measurement sector is in the wake of the blade. This behaviour is repeated when the other blade approaches the measurement sector again. The stresses are greater in the lower third of the bed (e.g. cell 2, shown in Figure 7) than the middle of the bed (e.g. cell 5, shown in Figure 8). In the upper third of the bed (e.g. cell 8, shown in Figure 9), the stresses are substantially less, since the material here is exposed to a reduced surcharge due to the reduced mass of material above. In the upper third of the bed, the stress shows negligible variation with impeller position.

In cell 2 (Figure 7), there is a large peak in the stress for 45 rpm at an impeller rotation of approximately 340^o, that is much greater than the stress at 20 and 78 rpm at this point. The exact cause has not been investigated and since this increased stress is only present for 1^o of rotation it does not have a great effect on the average stress in the cell.

The mean deviatoric stress calculated for one full rotation in each cell is shown in Figure 10. It can be seen that despite small variations between the stresses estimated at the three impeller speeds, the average stress is independent of impeller speed. However, it clearly increases with the radial position.

The shear strain rate distributions in cells 2, 5 and 8 are calculated based on equation 10 and are shown in Figures 11, 12 and 13, respectively. In contrast to the stress distributions, the strain rate is greatest over a region that extends from just in front of the blades into the wake of the impeller blades (at an impeller position of approximately 0 – 60^o). The movement of the blades causes particles immediately in front of the blades to be swept rapidly out of the path of the blades, therefore resulting in a high strain rate. In the wake of the blades the bed is more dilated, as the particles are projected over the top of the blades. This dilute stream of particles experiences less resistance to motion than throughout the rest of the bed, and has free space to fall into, hence the increased strain rate. As the particles fall onto the stagnant bed further behind the impeller blades (75 – 150^o), their velocities are greatly reduced, hence, the strain rate in this region is smaller. The strain rate in the measurement sector then increases slightly as the impeller blades approach, reaching a peak again as the blade passes through this sector. The strain rates are slightly greater at lower vertical positions, as shown by Figures 11 – 13. The variation in strain rate with vertical position is much less substantial than the variation of stress with vertical position. The strain rates are increased at increased impeller speeds.

The total strain, Γ, in a measurement cell is calculated by equation 11 throughout one impeller rotation (360^{o}).

$$\Gamma_{\mathrm{c,n}} = \int_{\mathrm{t_A}}^{\mathrm{t_B}} \left(\gamma_{\mathrm{c,n}} \, dt \right) \tag{9}$$

The total strains in each cell, at each impeller speed, are shown in Figure 14. For a given vertical position, the strain increases with radial position. This increase in strain with radial position is due to the increased impeller tangential velocity with radial position. For any given position in the bed, the total strain is independent of the impeller speed, as shown in Figure 14. This suggests that the local strain rate is proportional to the impeller speed.

As shown above, the stresses in each cell do not vary much with impeller speed. Based on the criteria set out by Tardos et al. (2003), the bed is operating in the quasi-static regime for all impeller speeds tested.

5. Attrition under Shear Deformation

Having analysed the distribution of shear behaviour within the dryer, the attrition under such conditions is assessed. Bridgwater et al. (2005) and Ghadiri et al. (2000) have analysed the dependence of the extent of attrition on the applied load and strain for the well-defined system of the annular shear cell. However, the analysis presented in the previous section shows a wide variation of the shear stress and strain for the sector under consideration as the impeller sweeps through the bed of particles. Therefore a methodology is required to make use of the data obtained in an annular shear cell to predict the attrition in the agitated vessel. This is outlined in this section.

The annular shear cell has been designed by Paramanathan and Bridgwater (1983a) to simulate the attrition behaviour in a shear band of particulate materials, and is used here to analyse the breakage rate of Paracetamol under the stress and strain conditions that are predicted by DEM simulations. The cell comprises an annular ring that is filled with particles to a sufficient height to represent the width of a shear band, roughly six particle diameters (Neil, 1986). The cell lid is then placed on to the bed and a load is applied to provide a normal stress.

Once a load has been applied to the sample, the cell base is rotated whilst a stopper arm attached to the lid prevents its rotation. Gripping rings in the base and cell lid ensure minimal slippage of material adjacent to these surfaces. Once the desired degree of rotation has been obtained the cell rotation is ceased. The sample is removed from the cell and the extent of attrition determined by sieve analysis. The prevailing stresses in the agitated dryer are low. Therefore the highest stress applied in the shear cell (3 kPa) corresponds to the weight of the lid alone. Lower stresses are achieved by using a pulley-counterbalance system (Figure 4). The level of applied stresses is sufficiently low so that attrition of particles is mainly by formation of fine debris from surfaces by chipping and wear, rather than by particle fragmentation. To quantify the extent of attrition it suffices to separate the debris by using a sieve size that is two standard sizes below the feed sieve size (Kwan et al., 2004). The extent of attrition, W, is then quantified as follows:

$$\mathrm{W} = \frac{\mathrm{M_{de}}}{\mathrm{M_m} + \mathrm{M_{de}}} \tag{10}$$

where M_{de} is the mass of debris material that passes through the sieve designated for separating the debris (355 μm in this case) and M_m is the mass of the material that is retained on the sieve. The same approach is also used for quantifying the extent of attrition in the agitated vessel.

The attrition is analysed under a range of applied normal loads corresponding to normal stresses of 0.1, 0.8, 1.4, 2.3 and 3.0 kPa, and a range of cell rotations corresponding to extents of strain of 30, 60, 120 and 240. The strain is calculated from the cell rotation and the bed dimensions by equation 13,

$$\Gamma = \frac{\theta}{360} \cdot \frac{\pi \cdot D_c}{h} \cdot f \tag{11}$$

where θ is the angle of cell rotation, D_c the arithmetic mean cell diameter (0.14 m), h the bed height (3 mm) and f the grip factor – the ratio of the experimental strain to the strain experienced by a perfectly gripped bed. The grip factor has been measured to be 0.94 in this case (Hare, 2010).

The attrition under this range of conditions is analysed using the equation of Neil and Bridgwater (1994) (equation 7). However, in order to correlate the attrition in the agitated vessel with that of the annular shear cell, the characteristic stress which influences the torque is the shear stress rather than the normal stress. Hence the normal stress has to be replaced by the shear stress and σ_{SCS} by a characteristic shear strength. In equation 7 the side-crushing strength of a material is used when comparing the attrition of different materials. In our case only the attrition of Paracetamol is considered. Therefore the reference shear strength, τ_{ref}, is taken as constant here. The shear strength of the Paracetamol particles is not known, therefore τ_{ref} is taken as the yield stress, Y, of Paracetamol. Bentham et al. (1998) found the hardness, H, of Paracetamol to be 0.55 MPa by nanoindentation. Since $H/Y \approx 3$ (Ghadiri, 2006), the yield stress of Paracetamol is approximately 0.183 MPa, therefore τ_{ref} is equal to 0.183 MPa. Changing τ_{ref} has no effect on the fitting of equation 14 to the attrition data, but results in a change in K_N.

To allow suitable comparison with the stress analysis in the dryer simulations, the applied shear stress, τ, in the shear cell is calculated from the normal stress using the internal angle of friction (37^o for Paracetamol (Hare, 2010); the attrition has therefore been measured at shear stresses, τ, of 0.1, 0.6, 1.1, 1.8 and 2.2 kPa and shear strains of 35, 69, 138 and 277.

$$W = k_N \cdot \left[\left(\frac{\tau_D}{\tau_{ref}} \right) \cdot \Gamma^{\varphi} \right]^{\beta} \tag{12}$$

The experimental results of the extent of attrition of Paracetamol as a function of the applied strain for shear stresses of 0.1 – 2.3 kPa are shown in Figure 15. The data are fitted to equation 14, and as indicated, a good fit is obtained with $k_N = 0.71$, $\varphi = 1.28$ and $\beta = 0.49$.

6. Attrition Prediction

The stress analysis in section 4 provides an estimate of the spatial and temporal distribution of stresses and strain in the dryer (Figures 7 – 9 and 11 – 13). The experimental analysis of Paracetamol attrition in the shear cell describes the relationship of attrition to the prevailing stresses and strains (equation 14, Figure 15). These are combined here to provide a prediction of the attrition in the dryer as a result of the prevailing stresses and strains.

Considering the prevailing shear stress and strain rate for one of the cells, for example cell 2 as given in Figures 7 and 11, the incremental attrition over a differential element of impeller rotation, $d\theta$, corresponding to time dt, is given by:

$$dW = k_N \left\{ \left[\frac{\tau_D}{\tau_{ref}} \right] \times \left[\int_t^{t+dt} \gamma \, dt \right]^{\varphi} \right\}^{\beta} \tag{13}$$

Integrating the above equation over a full rotation by considering n intervals of time in a discretised form, the total attrition in cell 2 is given by

$$W_c = k_N \sum_1^n \left[\frac{\tau_D}{\tau_{ref}} \right]^\beta \times \left[\left(\sum_1^n \Gamma_{c,n}^{\varphi\beta} \right) - \left(\sum_1^{n-1} \Gamma_{c,n}^{\varphi\beta} \right) \right] \quad (13)$$

and for the whole sector the attrition is obtained by summation of the nine cells. Since the dryer bed is in a steady cyclic state at the time of stress and strain calculations, the stresses and strain rates at a given position in the bed are considered to be similar in subsequent impeller rotations. As the impeller speed is constant in each case, the strain, Γ, is proportional to the number of impeller rotations, I. Therefore, the attrition after I rotations is given by:

$$\begin{aligned} W_{I=1} &= k_N \sum_1^c \sum_1^n \left(\chi_{c,n} \left[\frac{\tau_{c,n}}{\tau_{ref}} \right]^\beta \left[\left(\sum_1^n \left(I \cdot \Gamma_{c,n} \right)^{\varphi\beta} \right) - \left(\sum_1^n \left(I \cdot \Gamma_{c,n} \right)^{\varphi\beta} \right) \right] \right) \\ &= W_{I=1} \cdot I^{\varphi\beta} \end{aligned} \quad (13)$$

The details of derivation are provided in Appendix B.

The attrition predictions for 500 – 600 μm Paracetamol at impeller speeds of 20, 45 and 78 rpm, using equation 17, are shown in Figure 16. This shows a limited variation of attrition rate with impeller speed. This is expected since Figures 10 and 14 show the stress and strain to be independent of impeller speed.

The attrition in a cell as a percentage of the total attrition, ξ_c, is given by,

$$\xi = \frac{W_c}{W_{I=1}} \times 100\% \quad (14)$$

Figure 17 shows ξ_c for all cells. The positions in line with the impeller (cells 1 – 3) contribute most towards the overall attrition (at least 50% at each impeller speed), as the stresses in these regions are substantially higher than elsewhere in the vessel (Figure 10). For a given row of cells the contribution of attrition increases with radial distance, due to a combination of increased stress (Figure 10) and strain (Figure 14). The attrition contribution for a given cell is largely independent of impeller speed. The incremental attrition in cell 3 throughout one rotation is shown in Figure 18. The percentage attrition is relatively low in this cell when the impeller is far from the measurement sector. It increases before the impeller reaches the measurement sector (at approximate impeller positions of 150^o and 330^o) and peaks when the impeller contacts the measurement sector, at impeller positions of 180^o and 360^o. It then reduces as the impeller passes through the measurement region, with negligible attrition occurring beyond approximate impeller positions of 30^o to 150^o and 210^o to 330^o.

7. Comparison with Experimental Results and Discussion

The attrition of Paracetamol has been analysed in the small-scale dryer (Figure 2) at 20, 45 and 78 rpm. The vessel is filled with 250 g of Paracetamol, then the impeller speed is gradually increased to reach the desired speed, to prevent a sudden increase in strain rate, which could otherwise result in increased attrition. This period of increasing impeller speed lasts no more than 5 s, thus, is negligible in comparison with the total rotation time. The experiments were carried out separately for 5, 20 and 80 minutes of rotation. The attrition after rotation is quantified in the same way as the experiments in the shear cell, described in section 5, and included in Figure 16 for comparison with the attrition prediction. Figure 16 shows that the attrition is almost independent of impeller speed, as predicted by the above method. The actual attrition occurring in the dryer deviates slightly from the predicted attrition, though is generally well predicted by the technique introduced in this work. The attrition measured after 3600 impeller rotations at 45 rpm is less than that predicted from equation 17. However, this is measured in just one experiment. Each data point represents one experimental run in the agitated vessel, and is subject to stochastic error.

The deviatoric stresses estimated from the dryer simulations showed an average of 1 – 110 Pa, with a maximum peak stress around 1050 Pa, whilst the attrition in the shear cell was analysed at shear stresses of 100 to 2100 Pa. The highest strain analysed in the shear cell was 240, whereas the highest strain occurring in any region of the dryer corresponding to 80 minutes of agitation at 78 rpm was approximately 17500. The correlation of attrition to stress and strain was, therefore, fitted based on data from shear stresses which were double those present in the dryer, and strains nearly two orders of magnitude lower than those estimated to occur in the dryer. The empirically derived parameters k_N, φ and β may vary if the range of stresses and strains investigated in the shear cell are substantially altered. In the initial stages of bed rotation, a large amount of attrition occurs in the shear cell as the bed dilates; beyond this stage, the rate of attrition falls to an asymptotic level. Therefore, if the maximum strain investigated in the shear cell is greatly increased, the influence of strain on attrition would be reduced (represented by a reduction in $\varphi.\beta$). This would result in a lower prediction of attrition in the dryer and could therefore affect the prediction accuracy. However, it is not feasible to investigate attrition in the shear cell at significantly large strains as broken material segregates to the bottom, and the dynamics of the sheared bed change.

The breakage of non-spherical particles in a shear cell has been modelled by Potapov and Campbell (1997) using DEM. The particles were represented by a number of Delaunay triangle elements ‘glued’ together. Criteria were defined for bond breakage between elements based on tensile and shear forces exceeding specified limits. The extent of breakage was found to be proportional to the work done for shearing the bed. This approach has the potential to be applied to simulating particle breakage in the agitated vessels. However, further work is needed to refine the breakage criteria, as much of the experimental findings of particle attrition in a shear cell (Neil and Bridgwater, 1994; Ghadiri et al., 2000; Bridgwater et al., 2003) do not follow the trend predicted by the simulations of Potapov and Campbell (1997). Consequently, a different approach was taken in our work to predict the breakage of particles in an agitated vessel. By establishing the breakage rate of the material under prevailing conditions of stress and strain using a shear cell test device, a realistic breakage criterion was established empirically. Coupling this with the stresses and strains in the agitated vessel predicted by the DEM, a reliable prediction of attrition has been made. This is most useful for prediction of attrition of industrial-scale agitated drying using a small sample quantity for attrition testing. The method shown here has been applied to a dry bed, and further work is required to understand the influence of moisture content on attrition.

8. Conclusion

Attrition during agitated drying or mixing is inevitable and difficult to predict. The attrition rate of particles in a bed subjected to shear deformation is dependent on the prevailing stresses and strains. An attempt to predict this attrition was made by estimating the distribution of stresses and strains in a small-scale dryer using DEM and quantifying the extent of attrition under such conditions by the use of a shear cell. The attrition rates under a range of stresses and strains were analysed in an annular shear cell. The data were fitted to the Neil and Bridgwater (1994) model. Coupling the model with the prevailing shear conditions obtained from the simulation results, an estimate of attrition occurring in a dryer for three impeller speeds and for a range of rotation times was made. The predictions describe attrition well; generally slightly overestimating the extent of attrition measured experimentally in the small-scale dryer. In both the experiments and the simulations, the effect of impeller speed was negligible in the range tested (20 – 78 rpm).

Based on the observations from these simulations, the strain rate is highest at the outer radial positions in the vessel and decreases slightly at increased height. The stresses are substantially greater at the base of the vessel due to contact with the impeller blades, and are also greater at outer radial positions. The greatest contribution of attrition occurs near the base of the vessel, with approximately 50% of the attrition occurring in the bottom third of the vessel. The prediction method shows that the shear cell is representative of the shear behaviour occurring in the dryer. The distribution of stresses and strains in the two systems may vary greatly, however, this is accounted for in the simulations. The attrition prediction method introduced here could be applied to other devices where attrition is dominantly caused by shear deformation, such as bins, hoppers and paddle mixers.

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Nomenclature

a	scaling law power
D	impeller diameter
D_c	median diameter of cell
e	coefficient of restitution
f	grip factor
F	force
h	bed height
I	impeller rotations
k_G	empirical constant in Gwyn relation
k_N	empirical constant in Neil and Bridgwater relation
k_O	empirical constant in Ouwerkerk relation
K	contact stiffness
m_G	rate of material degradation in Gwyn relation
m_O	rate of material degradation in Ouwerkerk relation
M	mass of material
n	number of intervals
N	number of particles in measurement cell
p	position
r	radial position
r_p	particle radius
t	time
v	velocity
$\tilde{v}$	average velocity
V	volume
W	attrition
X	multiplier in incremental attrition example
Y	power in incremental attrition example
z	vertical position
Z_a	apparent coordination number
α	damping coefficient and coefficient of restitution proportionality term

β rate of material degradation in Neil and Bridgwater formula
γ strain rate
Γ strain
ζ damping coefficient
θ angle of rotation
ξ attrition contribution
σ stress
τ shear stress
τ_D deviatoric stress
υ poisson ratio
φ relative influence of strain and stress in Neil and Bridgwater formula
χ mass fraction
ψ angular position
ω impeller rotational speed

subscripts

1 scale 1
2 scale 2
A at start of interval
B at start of interval
c cell number
de debris
ii in the i direction on face i
I=1 after one impeller rotation
m mother
n normal direction
p particle
ref reference
s shear direction
SCS side-crushing
tot total
xx in the x direction on face x
yy in the y direction on face y
zz in the z direction on face z

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Appendix A – Velocity Validation

The particle velocities in the agitated bed were assessed using Positron Emission Particle Tracking (PEPT). In this technique a tracer particle is irradiated and placed inside the vessel of interest. A detector is placed either side of this vessel. The tracer particle emits positrons, which are rapidly annihilated when contacted by an electron, thus forming a back-to-back γ-ray. These events can occur hundreds of times a second, each providing a signal to both detectors. By considering a number of consecutive γ-ray emission events, the approximate position of the tracer particle can be located by triangulation. A more detailed description of the PEPT process is given by Parker et al. (1993).

It was not possible to irradiate the Paracetamol particles directly, so an alternative tracer had to be used. In order for the flow behaviour of the tracer to represent that of the Paracetamol, it is important for the tracer to be of similar size, shape and density. Therefore, the tracer used in this case consisted of two spherical resin particles glued to each other by an epoxy, one of which was irradiated, with diameters of 500 – 600 μm. This satisfies the criteria of similar size and shape, as indicated by Figure A1. The resin had a density of approximately 1000 kg/m^3, which was comparable to, though less dense than that of the Paracetamol (1300 kg/m^3).

The position of the tracer particle in the agitated bed was tracked using the PEPT technique. The exact conditions of the experiments, described in section 6, were replicated, using impeller speeds of 20, 45 and 78 rpm. For each experiment, the tracer position was tracked for approximately 90 minutes. The velocity of the tracer particle at any point in time is calculated from its position at consecutive time periods. Since an error is associated with the measured particle position (approximately 1 mm in this case), calculating velocity using only data from two consecutive time periods can lead to erratic results with large error (Stewart et al., 2001). Therefore, the velocity is estimated using the six-point method;

$$\begin{aligned} v_n = {} & 0.1\left(\frac{p_{n+5}-p_n}{t_{n+5}-t_n}\right)+0.15\left(\frac{p_{n+4}-p_{n-1}}{t_{n+4}-t_{n-1}}\right)+0.25\left(\frac{p_{n+3}-p_{n-2}}{t_{n+3}-t_{n-2}}\right) \\ & +0.25\left(\frac{p_{n+2}-p_{n-3}}{t_{n+2}-t_{n-3}}\right)+0.15\left(\frac{p_{n+1}-p_{n-4}}{t_{n+1}-t_{n-4}}\right)+0.1\left(\frac{p_n-p_{n-5}}{t_n-t_{n-5}}\right), \end{aligned} \tag{A.1}$$

where v is the velocity of the tracer at position, p, and time, t, for data point i. From these data, v_θ is found by

$$v_\theta = v_x \sin\psi_p + v_y \cos\psi_p \tag{A.2}$$

where ψ_p is the angular location of the particle. The average velocity, $\tilde{v}_\theta$, of the tracer particle, in each of the measurement cells described in Figure 6, is calculated using the above method. The average velocity estimated in each cell by the DEM simulations, is compared to those estimated using the PEPT technique in Figure A2. In the upper third of the bed (Figure A2 a – c), the velocities measured using PEPT are extremely similar to DEM predictions at all impeller speeds. In the middle third of the bed (Figure A2 d – f) the velocities obtained from PEPT are slightly greater than those predicted by DEM. In contrast to this, the velocities of the particles in the lower third of the bed (Figure A2 g – i) estimated by PEPT are less than those predicted by DEM. To investigate this discrepancy, the velocities at varying angular positions, in cell 2 as shown in Figure A2 (h), are analysed. Since the bed behaviour has a rotational symmetry of order 2, the fundamental domain is 180^o. To describe the angular position, ψ, the notation used here is that a negative angular position indicates the particles are behind the blade, and a positive angular position indicates particles are in front of the blade. Therefore, the angular position ranges from -90° – +90°. Figure A3 shows the velocity as a function of angular position at 78 rpm, measured by PEPT and predicted by DEM, in cell 2. It is clear that the velocities measured by PEPT and predicted by DEM are similar in front of the blade, while the velocities measured by PEPT in the wake region are much less than those predicted by DEM. Since the stresses are much greater in the front of the blade, whilst they are negligible in the wake of the blade, this overestimation of the particle velocities in the wake of the blade has a minimal impact on the attrition prediction technique described in section 6. Therefore, the velocities predicted by DEM in this case are adequate in describing the behaviour of the particle bed for utilisation in the attrition prediction technique introduced in this work.

Appendix B – Attrition Prediction Derivation

Equation 14 is used to calculate the fraction of attrition expected from the applied shear stress and the total strain. However, in the dryer bed the stress is not constant in a given cell, so equation 14 cannot be applied directly as it does not account for this variation. Instead, the incremental attrition caused by the prevailing stress and strain in a given time interval must be considered. Figure B.1 shows this method for calculating the incremental attrition caused by the prevailing stress and strain in successive time intervals, in the case where the stress is constant. In this example (shown in Figure B1), the total attrition is equal to the sum of the attrition caused in each interval, thus,

$$\begin{aligned} W_{tot} &= \sum_1^n W_n = W_1 + W_2 + W_3 + W_4 \\ &= X\Gamma_{tot}^Y = X\left(\sum_1^n \Gamma_n\right)^Y \end{aligned} \quad (B.3)$$

The attrition caused in time intervals 2, 3 and n are, therefore, given by:

$$\begin{aligned} W_2 &= X\left(\Gamma_1+\Gamma_2\right)^Y - X\Gamma_1^Y \\ W_3 &= X\left(\Gamma_1+\Gamma_2+\Gamma_3\right)^Y - X\left(\Gamma_1+\Gamma_2\right)^Y \\ W_n &= X\left(\sum_1^n \Gamma_n\right)^Y - X\left(\sum_1^{n-1}\Gamma_n\right)^Y = X\left[\left(\sum_1^n \Gamma_n\right) - \left(\sum_1^{n-1}\Gamma_n\right)\right]^Y \end{aligned} \quad (B.4)$$

Therefore, the total attrition,

$$W_{tot} = \sum_1^n W_n = \sum_1^n X\left[\left(\sum_1^n \Gamma_n\right)^Y - \left(\sum_1^{n-1}\Gamma_n\right)^Y\right] \quad (B.5)$$

If the stress is not constant, the multiplier, in this case X, varies with time, and the attrition cannot be calculated directly from the total strain. Therefore, the incremental approach shown by equation B.3 must be used. Since the stress is not constant in the dryer, the attrition occurring in a given cell, c, throughout n time intervals, is given by

$$W_c = k_N \sum_1^n \left(\left[\frac{\tau_{c,n}}{\tau_{ref}}\right]^{\beta}\left[\left(\sum_1^n \Gamma_{c,n}^{\varphi\beta}\right) - \left(\sum_1^n \Gamma_{c,n}^{\varphi\beta}\right)\right]\right) \quad (B.6)$$

where $\Gamma_{c,n}$ is given by equation 11 and $\tau_{c,n}$ is given by equation B.5.

$$\tau_{c,n} = \frac{\left(\tau_{c,t_A} + \tau_{c,t_B}\right)}{2} \tag{B.7}$$

where $\gamma_{c,t}$ is the strain rate in cell c at time t, and subscripts A and B indicate the start and end point of the interval respectively. The attrition occurring in the dryer bed after one impeller rotation is, therefore, given by:

$$W_{I=1} = \sum_{1}^{c} W_c = k_N \sum_{1}^{c}\sum_{1}^{n}\left(\chi_{c,n}\left[\frac{\tau_{c,n}}{\tau_{ref}}\right]^{\beta}\left[\left(\sum_{1}^{n}\Gamma_{c,n}^{\varphi\beta}\right)-\left(\sum_{1}^{n}\Gamma_{c,n}^{\varphi\beta}\right)\right]\right) \tag{B.8}$$

where χ_c is the mass fraction of particles in the measurement cell c, compared to the particles in the entire measurement sector. Since the dryer bed is in a cyclic, steady state at the time of stress and strain measurement, the measured stresses and strain rates at a given position in the bed are considered to be similar in subsequent impeller rotations. As the impeller speed is constant in each case, the strain, Γ, is proportional to the number of impeller rotations, I. Therefore, the attrition after I rotations is given by:

$$\begin{aligned} W_I &= k_N \sum_{1}^{c}\sum_{1}^{n}\left(\chi_{c,n}\left[\frac{\tau_{c,n}}{\tau_{ref}}\right]^{\beta}\left[\left(\sum_{1}^{n}\left(I\cdot\Gamma_{c,n}\right)^{\varphi\beta}\right)-\left(\sum_{1}^{n}\left(I\cdot\Gamma_{c,n}\right)^{\varphi\beta}\right)\right]\right) \\ &= W_{I=1}\cdot I^{\varphi\beta} \end{aligned} \tag{B.9}$$

9. List of Figures

10. List Tables