Attrition of paracetamol and aspirin under bulk shear deformation

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HIGHLIGHTS

- Breakage of paracetamol and aspirin established for given stress and strain.
- Empirically determined at low stresses (0.1–3.0 kPa).
- Aspirin breaks more easily at onset of shearing.
- Paracetamol more prone to breakage at increased stress or strain.
- Surprisingly no preferential breakage along cleavage planes.

GRAPHICAL ABSTRACT
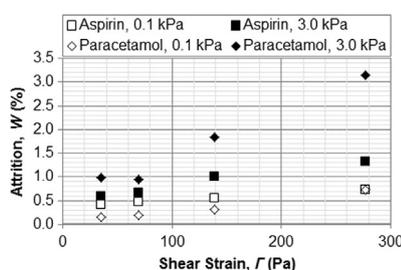
ABSTRACT

Particles are frequently exposed to shear stresses during manufacturing, which leads to breakage. This is particularly relevant to weak active pharmaceutical ingredients and is prevalent in pharmaceutical and food industries. The attrition of paracetamol and aspirin caused by shear deformation at very low stresses is investigated here. The extent of breakage of these particles is related to the prevailing shear stresses and strains. In contrast to the expected trend, smaller particles exhibited increased breakage rates. At the onset of shearing at low stresses aspirin particles experienced slightly more breakage than the paracetamol, however prolonged shearing resulted in greater breakage of paracetamol. Breakage occurred initially through chipping with some fragmentation, particularly more noticeable for aspirin, with an increase in abrasion after extensive shear strain for paracetamol. Empirical breakage relationships are proposed and when combined with process stresses and strain analyses the extent of breakage occurring in process equipment can be estimated.

1. Introduction

Particle processes invariably inflict unwanted stresses upon particles. These stresses could be caused by compression, impact, fatigue or shearing and can result in damage and breakage of the particles. One of the common causes in the unintentional breakage of particles (attrition) during processing is shear deformation. Shear stresses exist in the transition from stagnant to flowing material within a powder bed, and are prevalent in manufacturing, such as through agitation in a mixer, and in transport and release from storage.

It is highly desirable to produce particles with a specified range of sizes, shapes and surface properties to limit downstream processing problems and ensure the final product satisfies its end-use application. However, attrition causes a deviation from these characteristics, leading to material loss or reduced process efficiency. Dust may be produced by attrition, which can result in substantial triboelectric charge generation. In extreme cases, the dust generated from attrition can lead to an explosive atmosphere. Attrition is a particular problem in the pharmaceutical industry, as many pharmaceutical compounds are relatively weak and therefore prone to breakage. It is therefore of interest to understand the susceptibility of common pharmaceutical materials to attrition.

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The stresses and strains present in shear bands formed in powder processes are highly dependent on the orientation of the particle–particle contacts, known as the bed fabric, and the material properties (Antony and Kuhn, 2004). A distribution of material properties is ubiquitous in powders (Salman et al., 1995), and particle rearrangements lead to an ever-changing bed fabric. Therefore, stresses fluctuate significantly throughout a particle bed, and even within a shear band (Liu et al., 1995). Stresses also fluctuate with time (Thornton and Antony, 2000). Due to these complexities in quantifying stress and strain within particle systems, it is difficult to establish a relationship of breakage with stress, strain and strain rate in processing equipment. For this reason, Paramanathan and Bridgwater (1983a, 1983b) developed an attrition shear cell to characterise particle breakage resulting from shear stresses. We use their approach in evaluating the attrition propensity of paracetamol and aspirin.

The attrition shear cell is shown in Fig. 1 and has been made by Ajax Equipment (Bolton, UK). Further details are given by Ghadiri et al. (2000). The strain, Γ, is estimated by the following relationship:

$$\Gamma = \frac{\theta}{360} \frac{\pi D_c}{h} f \tag{1}$$

where, θ, D_c, h and f are the degree of cell rotation, the mean cell diameter, the bed height and the grip factor, respectively. Early work suggested the breakage may be proportional to the work applied to the bed (Bridgwater, 1989) as Neil (1986) found that the internal angle of friction remained unchanged during shearing, thus the shear stress, τ, is proportional to the normal stress. Therefore the work applied to the bed is proportional to $\tau\Gamma$. The two-dimensional Discrete Element simulations of Potapov and Campbell (1997) supported this conclusion of Bridgwater (1989), as did the experimental work of Kenter (1992), in contrast to the experimental work of Neil and Bridgwater (1994). Potapov and Campbell (1997) suggested four possible reasons for their simulations showing anomalous behaviour in comparison to Neil and Bridgwater (1994): (i) the two dimensional nature of the simulation, (ii) the lack of gravity, (iii) the omission of the work required for the initiation of shearing and (iv) due to the properties of the simulated material. In most cases the breakage has been shown not to be proportional to the work applied to the bed. Neil and Bridgwater (1994) measured the attrition of 11 materials over a wide range of stresses and strains and found that the breakage could not be suitably expressed as a function of the input work. Instead, they proposed a new relationship:

$$W = K_N \left[\left(\frac{\sigma}{\sigma_{SCS}} \right) \Gamma^{\varphi} \right]^{\beta} \tag{2}$$

where, K_N is the proportionality constant, σ is the applied normal stress, σ_{SCS} is the material side-crushing strength, φ is the relative influence of strain to stress on attrition and β is the rate exponent. Ghadiri et al. (2000) also showed the above relationship fits better to the breakage of catalyst beads in a shear cell as compared to input work and the earlier model of Ouwerkerk (1991). Moreover they showed that the nature of a power law model implies that a range of values of α and β may all provide a suitably close minimum fitting error, hence not leading to a unique set of values. Due to the complexities of sheared powder beds this is the best model which is currently available, as particle breakage under shear deformation cannot yet be determined by first principles based on knowledge of the mechanical properties of the particles. Consequently, the empirical approach defined above is required to describe the breakage of a given material. Moreover this analysis is extremely useful in determining the extent of breakage arising in process equipment that causes breakage by shear deformation. Neil and Bridgwater (1999) showed that the relationship of attrition to strain (the product $\varphi\beta$ in Eq. (2)) was consistent for given materials in a shear cell, a fluidised bed and a screw pug-mill. Further to this, Hare et al. (2011) showed that the breakage rate determined in a shear cell can be combined with stress and strain rate analysis using the Distinct Element Method (DEM) to successfully predict the attrition in an agitated bed.

In this work the attrition of paracetamol and aspirin are investigated using an annular attrition shear cell. The interest is to understand breakage of these particles at very low stresses of 0.1–3.0 kPa.

2. Materials and methods

The test methodology has previously been described by Ghadiri et al. (2000). Here we report our measurement and analysis for paracetamol (Form I) and aspirin (supplied by Kraemer & Martin Pharma Handels (Krefeld, Germany), and Weylchem (Mannheim, Germany), respectively). The following sieve cuts: 500–600 μm, 600–710 μm and 710–850 μm for paracetamol, and 355–425 μm, 425–500 μm and 500–600 μm for aspirin were used. Scanning electron micrographs (SEM) of these particles are shown in Fig. 2.

In each experiment the particles were sheared in the shear cell and subsequently sieved to assess breakage. Since the initial size was determined by sieving this method ensured the same shape descriptor was being measured after shearing. Sieving time and amplitude were selected to ensure that negligible attrition occurred due to sieving. The inner and outer diameters of the cell were 120 and 160 mm, respectively. The base and lid of the cell contained radially aligned saw-toothed gripping rings, with a depth of 1 mm, following the gripping criteria set out by Ghadiri et al. (2000). This provided sufficient grip for all size classifications tested. The material was weighed, prior to loading in the cell by pouring through a funnel. The bed was levelled using a T-piece to provide a bed with a relatively smooth surface and a known bed height. The bed height was controlled to be equal to approximately 5.5 times the median particle size (see Table 1), which corresponded to a naturally occurring shear band (Stephens and Bridgwater, 1978). Excess material was removed during the levelling process to achieve the conditions shown.
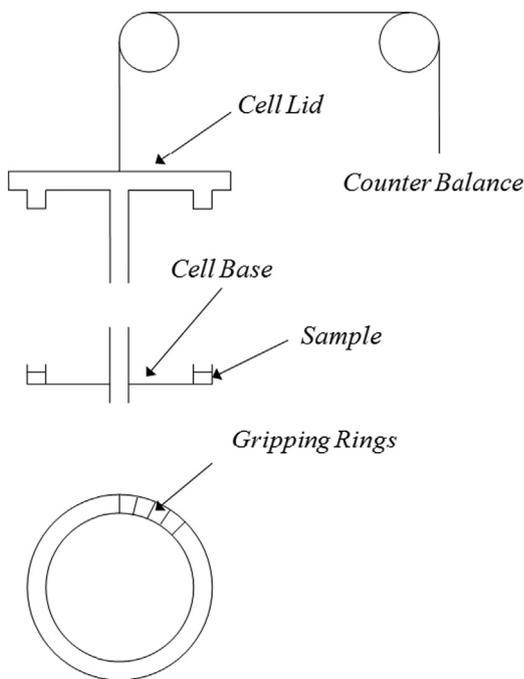

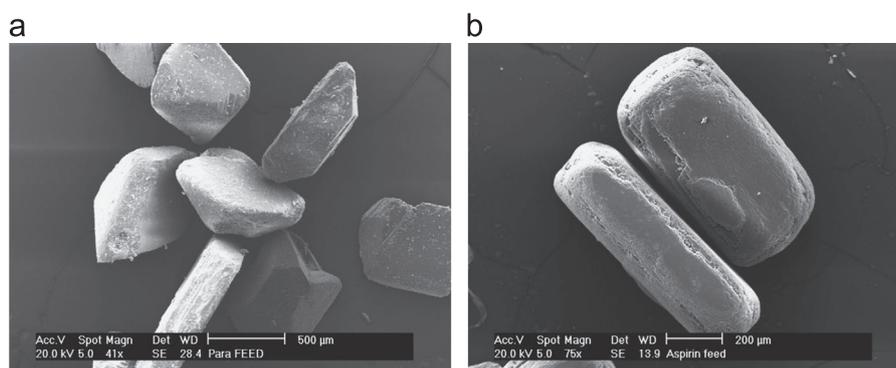**Fig. 1.** Attrition shear cell (Hare et al., 2011).

Fig. 2. SEM of feed particles (a) paracetamol (b) aspirin.

Table 1
Bed heights used in the shear cell.

Material	Size (µm)	Bed height (mm)	Mass (g)
Paracetamol	500–600	3.0	22.2–24.1
	600–710	3.6	23.9–25.6
	710–850	4.3	30.5–31.0
Aspirin	355–425	2.1	16.1–16.6
	425–500	2.5	19.3–19.7
	500–600	3.0	22.3–24.2

Table 2
Shear strain experienced by particles of various sizes in the shear cell.

Particle size (µm)	Cell rotation (°)			
	90	180	360	720
355–425	49.5	98.9	197.9	395.6
425–500	41.5	83.1	166.2	332.3
500–600	34.6	69.2	138.5	276.9
600–710	28.8	57.7	115.4	230.8
710–850	24.2	48.3	96.6	193.2

The desired normal stress was determined prior to each experiment. The weight of the lid provided a normal stress of 3.0 kPa—the maximum stress applied in the range of experiments. To apply lower stresses, a counter-weight was attached to the lid via a pulley system, in a similar manner to that of Bridgwater et al. (2003). The tension in the cable was measured using a Tensitron (TX-5000) tension meter in order to calculate the weight acting on the bed. After bed preparation the counter weight was attached to the lid before it was placed gently onto the surface, whilst ensuring the stopper arm remained in contact with the stopper. The cell was then rotated at 10 rpm until the desired cell rotation was achieved, at which point rotation was stopped. The lid was then removed and the material collected into a collection pan using a fine brush to gently transfer particles to the pan and minimise material losses. The entire contents were then transferred to a sieve stack containing the lower feed size and 5 standard sieve sizes below this. The material was sieved for 15 min with 0.7 mm amplitude and a one second pause after every five seconds of sieving, using a Haver & Boecker EML 200 sieve shaker. These sieving conditions provided consistent and reliable sizing classification. The classified material was then weighed to determine the extent of breakage. Due to inevitable material losses the mass collected was not equal to that of the feed. Therefore breakage could be assessed by a number of methods, i.e. Eqs. (3)–(5). The minimum breakage is defined by Eq. (3), the maximum by Eq. (5), and that calculated based on the material collected is given by Eq. (4). In this work the breakage was defined as that measured from the collected material (Eq. (4)).

$$R^- = \frac{M_{de}}{M_f} \tag{3}$$

$$R^* = \frac{M_{de}}{M_m} \tag{4}$$

$$R^+ = \frac{M_f - M_m}{M_f} \tag{5}$$

where M_{de}, M_m and M_f are the mass of debris (passing through the sieve), mother particles (retained on the sieve) and feed,

respectively. For each size classification of each material, experiments were carried out at normal stresses of 0.1, 0.8, 1.4, 2.3 and 3.0 kPa and cell rotations of 90°, 180°, 360° and 720°. The grip factor, f, was measured to be 0.94 for the 500–600 µm paracetamol and assumed to be the same for all sizes of aspirin and paracetamol tested. The corresponding strains for particles of various sizes are shown in Table 2.

3. Results

3.1. Paracetamol

Initially the material passing through a sieve with a mesh that is two standard sieve sizes below that of the lower sieve size (e.g. 355 µm for a feed size of 500–600 µm) is considered and referred to as the extent of attrition, following the method of Kwan et al. (2004). Eq. (6) shows a modified version of Neil and Bridgwater's formula, which accounts for applied shear stress, normalised by the bulk crushing strength of the material, σ_{str}. The value of σ_{str} is obtained by placing 2 g samples in a 20 mm diameter die, which provides a bed height of 5–10 mm, and compressing with a piston at a rate of 1 mm/min. After compression and unloading the material was sieved to determine the extent of attrition in the same manner described above for the shear cell experiments. The crushing stress required to generate 0.5 % breakage was taken to be σ_{str}, these values of crushing strength for the tested sizes of paracetamol and aspirin are given in Table 3. This extent of breakage was selected as it was in the range of extents of breakage occurring in the shear cell experiments. Shear stresses were calculated using internal angles of friction of 37° and 39° for paracetamol and aspirin, respectively, as determined through Schulze shear cell measurements. Eq. (6) was applied to each data set to determine the empirical description of the breakage as a function of applied stress and strain. The extents of attrition for 500–600 µm, 600–710 µm and 710–850 µm paracetamol are shown in Figs. 3–5, respectively, along with the best empirical fits provided by Eq. (6) for each case. In all cases the coefficient of

Table 3
Bulk crushing strength of paracetamol and aspirin.

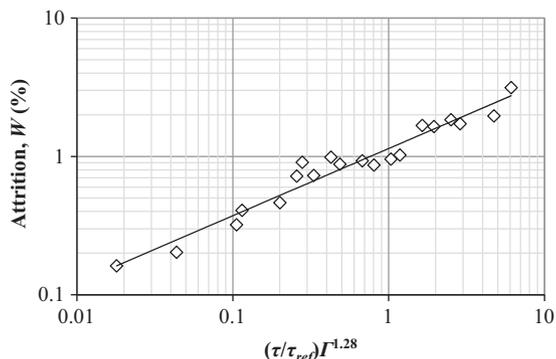| Material	Size (µm)	Crushing strength (MPa)
Paracetamol	500–600	0.49
	600–710	0.40
	710–850	0.39
Aspirin	355–425	1.08
	425–500	1.24
	500–600	1.25

Fig. 3. The extent of attrition of 500–600 µm paracetamol.
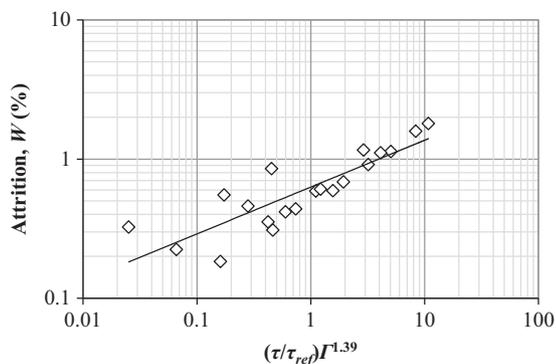
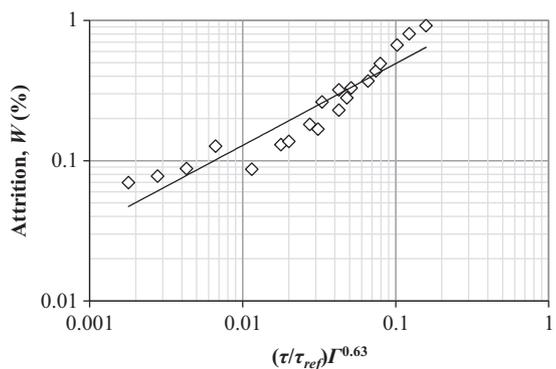**Fig. 4.** The extent of attrition of 600–710 µm paracetamol.

Fig. 5. The extent of attrition of 710–850 µm paracetamol.

variation, ξ, is close to 0.9, as shown in Table 4; indicating that the empirical equation describes the breakage data well. However, the values of φ and β vary widely between the different sizes of material, with φ ranging from 0.63 to 1.39 and β varying from 0.34 to 0.58 for paracetamol. This does not necessarily imply that the dependency of the breakage on stress or strain is significantly

different for the different sizes of material, as a variation in these fitting constants may still result in an adequate fit.

$$W = K_N \left[\left(\frac{\tau}{\sigma_{str}} \right) \Gamma^{\varphi} \right]^{\beta} \tag{6}$$

Fig. 6 shows the values of β and corresponding ξ for a range of φ. For a given value of φ the optimum value of β is selected to provide the greatest correlation coefficient. It can be seen that β does not vary much with φ, and this is also the case for the other sizes of paracetamol tested. Although the fit is poor when φ is close to zero, the fit is good in the range $0.5 \leq \varphi \leq 2.0$. The influence of φ on ξ is shown in Fig. 7 for all sizes of paracetamol tested. All three sizes exhibit similar trends, with a good fit existing for a wide range of φ. It should be noted that when the breakage data of all sizes is combined the fit is only moderately acceptable, with a maximum ξ of 0.71 when φ is 1.4 (in this instance $\beta = 0.45$). This suggests that the fitting parameters of Eq. (6) are not only material dependent, but also feed size dependent.
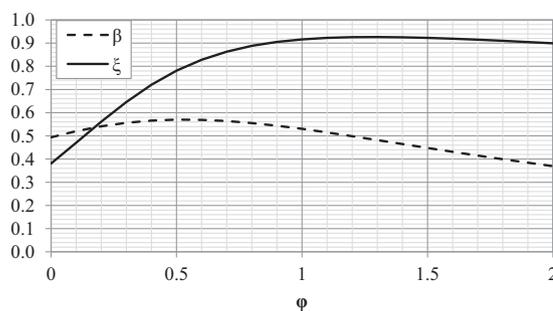

Fig. 6. Variation in β and ξ with φ for 500–600 µm paracetamol.
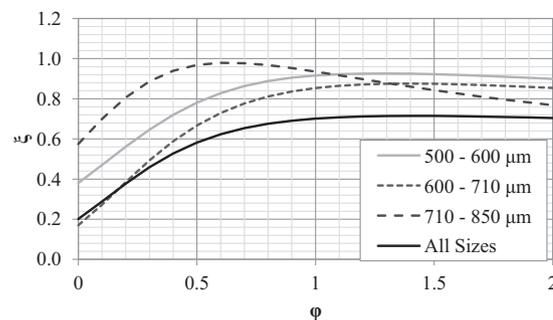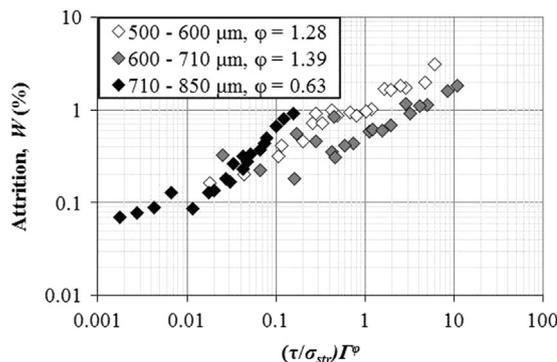

Fig. 7. Variation in ξ with φ for all sizes of paracetamol.

Fig. 8. The extents of attrition of paracetamol of various sizes.

Caution should be taken with this empirical approach, and it should be understood that the fit provides an approximation of the relationship of breakage to stress and strain for the range tested. Extrapolation of the relationship to include conditions out of this range will lead to inaccuracies.

The extents of attrition of the three sizes of paracetamol are shown together in Fig. 8. The data overlap quite well, though the extent of attrition is greatest for the 500–600 μm particles at increased stress and strain, followed by the 710–850 μm particles, with the 600–710 μm particles experiencing least breakage. The contact force is responsible for the particle breakage, and it is not known how the relationship between applied stress and contact force varies with particle size. The Distinct Element Method (DEM) may be used to investigate this.

3.2. Aspirin

The attrition of aspirin is assessed in the same manner as the paracetamol. Figs. 9–11 show the breakage data for 355–425 μm,
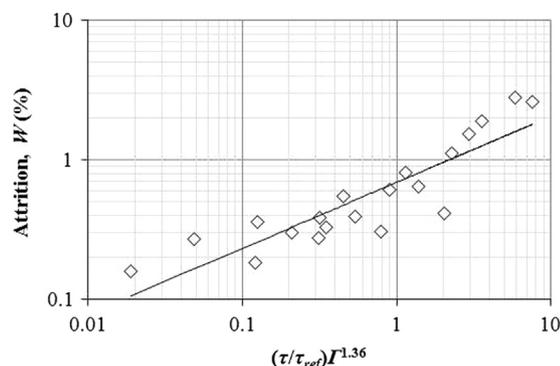
Table 4
Summary of best fits to breakage data using Eq. 6.

Material	Size (μm)	K_N	φ	β	ξ
Paracetamol	500–600	1.141	1.28	0.49	0.93
	600–710	0.630	1.39	0.34	0.88
	710–850	1.889	0.63	0.58	0.98
Aspirin	355–425	0.689	1.36	0.47	0.86
	425–500	0.725	1.17	0.35	0.77
	500–600	0.400	2.17	0.17	0.80

Fig. 12. The extents of attrition of aspirin of various sizes.

425–500 μm and 500–600 μm aspirin, respectively, along with the best fit of the data using Eq. (6). As with the paracetamol, a range of φ and β exists that provide a decent fit to the data. A summary of the best fits to Eq. (6) for all sizes of paracetamol and aspirin is given in Table 4 in the next section. The coefficient of variation, ξ, is worse than that of paracetamol.

The extents of breakage of the various sizes of aspirin are compared in Fig. 12. The attrition propensity of the 355–425 μm and 425–500 μm aspirin particles are very similar, whereas the larger 500–600 μm particles are more resistant to breakage.

3.3. Comparison of paracetamol and aspirin breakage

In an effort to compare the breakage of paracetamol and aspirin, whilst removing the influence of feed particle size, the extents of attrition of 500–600 μm paracetamol and aspirin are shown together in Fig. 13. It should be noted that both the feed and the attrition products have been separated by sieving and although these particles have the same sieve size, their different shapes mean that their particle size distributions cannot be considered to be identical. Under this range of conditions the paracetamol particles are generally more prone to breakage at

Fig. 9. The extent of attrition of 355–425 μm aspirin.

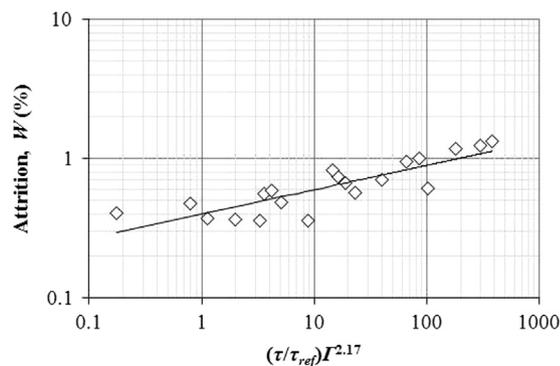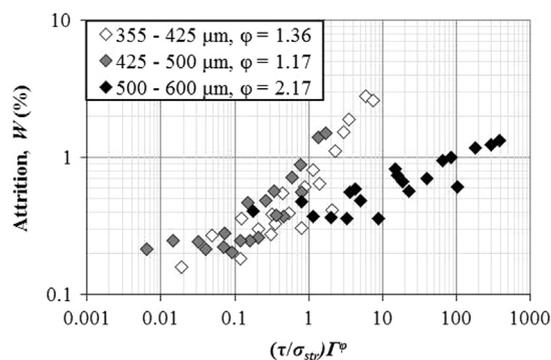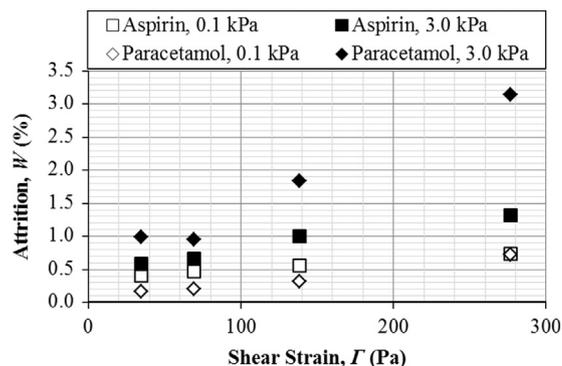**Fig. 10.** The extent of attrition of 425–500 μm aspirin.

Fig. 11. The extent of attrition of 500–600 μm aspirin.

Fig. 13. The extents of attrition of 500–600 μm paracetamol and aspirin.

higher stresses, with a maximum breakage of 3.1% compared to 1.3% for aspirin (both occur at a normal stress of 3.0 kPa and a strain of 277). However at very low stress and strain the aspirin particles experience greater breakage. This suggests the aspirin particles are not so resistant to breakage at the onset of shearing, however are more resistant to breakage after prolonged shearing than the paracetamol.

The above analysis considers the total attrition products (all material smaller than two standard sieve cuts below the lower feed size). However, it is informative to establish if breakage occurs primarily by chipping or if particle fragmentation also plays a role, considering the very low applied normal loads. For this purpose the full size distribution of these attrition products is considered. The size distribution is expressed in terms of cumulative mass percentage undersize (obtained by sieving) as a function of particle size, normalised with respect to feed size, on logarithmic scales. The data are then fitted with the correlation of Schuhmann (1940), given by Eq. (7). This equation is applied to the individual sieve cuts of the attrition products, where d_T is the sieve size that is two standard sizes below the lower feed size (355 μm) and W_T is the mass of material passing through this sieve. Fig. 14 shows the size distribution, down to 150 μm, of the paracetamol with an initial sieve size of 500–600 μm sheared at 0.1 kPa, where d_0 refers to the upper sieve size of the feed, along with the fit of Eq. (7). This relationship describes the size distribution of the sheared material well, despite the extremely small quantity of material in the lower sieve sizes.

$$W = W_T \left(\frac{d}{d_T} \right)^G \qquad (7)$$

Eq. (7) provides an extremely good fit to the breakage data for all stresses and strains applied to the 500–600 μm paracetamol, with $\xi > 0.98$ in all cases. However, a different value of G is obtained for each test condition. Fig. 15 shows the value of G for all experiments carried out for this material. A high value of G indicates that the majority of attrition products are towards the larger sizes (dominated by fragmentation), and a low value of G indicates an increased fraction of very fine material for a given total extent of attrition (indicating chipping). As the extent of attrition increases, G decreases rapidly, this indicates that as stress or strain is increased an increased fraction of generated debris is of very small sizes. A change in G suggests a shift in the dominant attrition mechanism. This reduction implies that size reduction is initially by fragmentation at the onset of shearing and it progresses towards a greater propensity for chipping and surface abrasion, since this would lead to generation of a substantial quantity of fines. Such behaviour has been observed at higher stresses for catalyst beads (Ghadiri et al., 2000) and detergent enzyme placebo granules (Ahmadian, 2008). However in the current investigation no fragmentation was observed from SEM
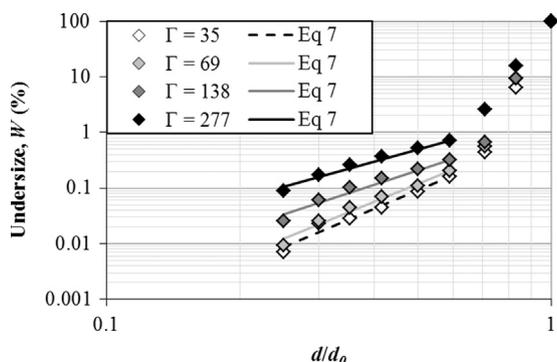

Fig. 14. Sieve Size distribution of paracetamol with an initial size of 500–600 μm after shearing with an applied load of 0.1 kPa at four strains.
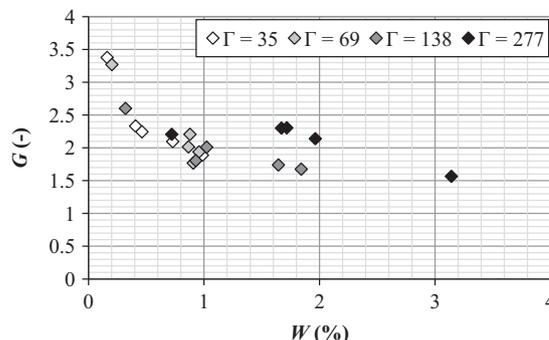

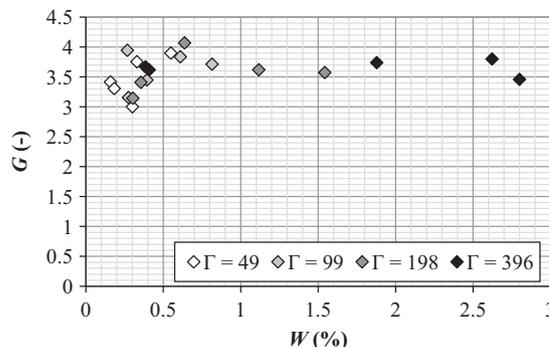Fig. 15. Variation of G with extent of attrition of 500–600 μm paracetamol.

Fig. 16. Variation of G with extent of attrition of 355–425 μm aspirin.

images of the paracetamol, hence initial breakage is expected to be through chipping. A similar trend of distribution modulus with extent of attrition is observed for the two larger sizes of paracetamol, however the reduction is not so dramatic. Once the packed bed is sheared it has to dilate first and this implies that the interlocked particles have to pass each other during which they could crush each other, hence an initial large value for G.

Fig. 16 shows the variation of distribution modulus with extent of attrition for the 355–425 μm aspirin particles. In contrast to paracetamol, the value of G is almost constant, with a value of 3–4. This indicates that fewer fines are present compared to paracetamol for a given extent of attrition. Since the value of G remains relatively unchanged, the breakage mechanism of the aspirin particles appears to be the same regardless of stress or strain. The high value of G suggests the aspirin particles have undergone attrition predominantly by fragmentation throughout all stresses and strains tested. These results show that paracetamol and aspirin are both prone to noticeable breakage even at very low stresses. Some industrial processes would expose these particles to significantly greater stresses, and consequently significant breakage is expected as a result.

4. Conclusions

The extent of attrition of paracetamol and aspirin caused by shear deformation was assessed in an annular shear cell at stresses from 0.1 to 3.0 kPa and strains of 24–396. The breakage of both materials is well described by the empirical fitting approach of Neil and Bridgwater (1994) and the size distribution of attrition products follows the equation of Schuhmann (1940). At the onset of shearing, aspirin displayed a slightly greater propensity for breakage than paracetamol at very low stresses, however as stress or strain was increased paracetamol exhibited a greater extent of breakage. aspirin produced a smaller fraction of fine material than paracetamol for a given extent of breakage. Breakage appeared to

be predominantly due to chipping under these conditions. As shearing progressed a greater fraction of the paracetamol attrition products were fines, indicating increased abrasion, whereas no change in the distribution of breakage products was observed for aspirin.

Particle breakage due to shear deformation is very complex and is not amenable to rigorous analysis providing predictive capability, hence empiricism is still the order of the day, and this requires the material to be exposed to controlled conditions, such as in the shear cell, in order to elucidate the breakage behaviour and quantify the size reduction. Once this has been achieved the breakage occurring in a process can be predicted by combining this information with the DEM analysis, such as the approach taken by Hare et al. (2011) for agitated beds. At present the authors are unaware of any predictive approach for breakage under known stress and strain, based on material properties, hence the method followed here is recommended to understand breakage of particles in a given process.

Acknowledgements

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