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Analysis of milling of dry compacted ribbons by distinct element method

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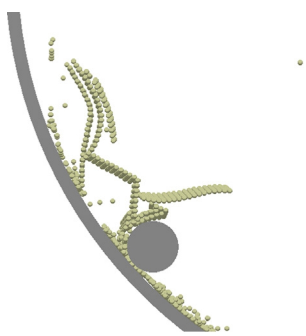
HIGHLIGHTS

- Dry compacted ribbons were represented in DEM by bonded spheres.
- The bond strength was calibrated by experimental and simulated 3 point bend tests.
- DEM estimates impact velocities and stresses the ribbons experience in the mill.
- Ribbons are exposed to such impacts and stresses to predict size distribution.
- Predicted size distribution correlates well with plant data.

GRAPHICAL ABSTRACT



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ABSTRACT

Fine cohesive powders are often dry granulated to improve their flowability. Roller compaction is commonly used to produce dense ribbons which are then milled. The material properties of the powder and the conditions in the roller compactor affect the strength of the ribbons, however there is no method in the literature to predict the size distribution of the product of ribbon milling. Here we introduce a method, by using the Distinct Element Method (DEM) to determine the prevailing impact velocities and stresses in the mill, with bonded spheres representing the ribbons. The bond strength is calibrated by matching experimental results of three point bend measurements and predictions from numerical simulations. The ribbons are then exposed to the dynamic conditions predicted by the DEM, by dropping them from a controlled height to cause fragmentation, and subsequently stressing them in a shear cell under the conditions again predicted by the DEM. The fragments are sheared under these conditions to represent repeated passage of bars over the fragments at the mill base. Sieve analysis is used here to determine the particle size distribution under given mill conditions. The predicted size distribution of the mill product compares well with the plant data. It is found that the mill speed and length of ribbons fed to the mill have no significant influence on the product size distribution for the range tested.

1. Introduction

Fine particulate materials often pose handling problems, such as poor flowability, adversely affecting dose uniformity. To overcome this, particle size can be increased by a variety of granulation methods, such as wet granulation in rotating or tumbling vessels

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(Salman et al., 2007), or dry granulation through direct compression in a roller compactor (Guigon et al., 2007), followed by milling. The products of a roller compactor are typically relatively large sheets, known as a ribbons (> 10 mm in length and width), and as such they are subjected to a subsequent size reduction process by milling. Miguelez-Moran et al. (2008) investigated the effect of lubricants on ribbon density and found that the method of loading in the compactor is crucial in determining density homogeneity, with average density increasing with peak pressure and nip angle. Khorasani et al. (2015) determined ribbon porosity using NIR, mercury porosimetry and oil intrusion, all of which indicated reduced ribbon porosity with compaction pressure. Muliadi et al. (2013) showed that the predicted density distribution of roller compacted pharmaceutical powders by Finite Element Method (FEM) can correlate well with experimentally-measured values by X-Ray Tomography (XRT). Both experiments and the FEM showed that when the rollers are free to move laterally the inlet stress does not affect the average ribbon density. Ribbon density and strength increase as the hardness of the feed material is reduced, as indicated by nano-indentation measurements (Al-Asady et al., 2015).

During milling the size reduction can be driven by impact, shear and compression to varying degrees, depending on the mill type, operational conditions and material properties. Consequently, it is difficult to determine the suitable conditions to use for milling of a given ribbon product from roller compaction. Vanarase et al. (2015) compared the performance of a hammer mill and a co-mill in milling extrudates of alumina-magnesia. It was shown that the impeller speed was influential in the size reduction in the co-mill, but not in the hammer mill. An increase in outlet mesh size leads to a coarser product, with the hammer mill being more efficient in allowing particles closer to the mesh size to exit. Samata et al. (2012) showed that the profile of the screen in a cone mill is influential in determining the milling performance, with a mesh opening inclined to the direction of motion of the impeller leading to better gripping and minimising the production of fines. Yu et al. (2013) investigated the influence of magnesium stearate on formed ribbons of micro-crystalline cellulose (MCC) and dicalcium phosphate dihydrate (DCPD), and their subsequent milling performance in an oscillating mill. Comparing the MCC and DCPD ribbons, it was found that the size reduction in the mill could be related to the fracture energy measured by 3-point bend tests, although it should be noted that the DCPD ribbons were too fragile to be tested in this manner.

Hare et al. (2011) developed a method for predicting size reduction of pharmaceutical particles in an agitated vessel by estimating the bed stresses and strains using the Distinct Element Method (DEM). They related the experimentally-determined breakage to the applied stress and strain, the latter predicted by DEM. DEM is most commonly applied to spherical particles, though it can be used to represent non-spherical particles by the clumped sphere method (Favier et al., 1999). Moreno and Ghadiri (2003) simulated impact breakage of agglomerates generated using DEM with bonds of a given strength acting between contacting particles. The breakage mechanisms of chipping and fragmentation were both replicated by the DEM, with the extent of breakage being a function of the bond surface energy expressed by Weber number. Calvert et al. (2011) simulated aerodynamic dispersion of cohesive clusters by Computational Fluid Dynamics (CFD) coupled with DEM, and showed the same functional group is also applicable to their case. Breakage of non-spherical particles has also been investigated by Grof et al. (2011), who showed that DEM could be combined with experiments to develop breakage kernels for compression of needle-shaped particles. Kozhar et al. (2015) simulated the compression of irregular titania particles and demonstrated that the bonded sphere approach could better capture the force-displacement response of the experiments, in comparison to the clumped sphere method.

To our knowledge DEM has not yet been applied to milling of dry compacted ribbons in the literature. Here we propose a combined experimental and computational approach to better understand the impact and shearing conditions that the ribbons experience under different mill operations, and to predict the resulting particle size distribution and residence time.

2. Materials and methods

The mill considered in this work is a rotary bar mill, as shown schematically in Fig. 1. It consists of a large horizontally-aligned hemi-cylinder with a length to diameter ratio of 1.89, and vertical side walls, within which four horizontal cylindrical bars of 20 mm diameter, held by a thin disc attached at each end, rotate around an axis, centrally aligned within the hemi-cylinder. The mill diameter is approximately 5–10 times the length of the ribbons. The cylinder is constructed using a sieve mesh with an outlet diameter of 3 mm, through which milled material exits. The rotating bars contact the hemi-cylinder when the mill is empty. However, as ribbons are fed into the top of the mill, the weight of feed material in the mill and the nipping of the ribbons by the rollers cause the sieve mesh to be separated from the bars by a narrow gap. As the ribbons enter the mill they may be impacted by a bar at the top of the mill, or they may bypass an upper bar and impact the base of the mill. The likelihood of impact at the top is influenced by the rotational speed of the bars, the sheet length and thickness, and entrance velocity. In the mill analysed here, the entrance velocity is determined by the vertical distance between the mill entrance and the roller compactor, and the feed rate to the roller compactor. Two ribbon formulations are considered in this work; formula A and formula B, as shown in Fig. 2. The ribbon lengths, widths and thicknesses are shown for both formulas in Table 1. After the fragments from initial impacts settle at the base of the mill they are continually sheared by the rotating bars until they are small enough to pass through the sieve mesh.

In this paper, a combined experimental and computational approach to predict milling performance of the above system is described (shown in Fig. 3), implemented and compared to plant data. To understand the impact velocities and stresses that the ribbons experience in the mill, the ribbons are represented in the DEM by using two separate approaches, namely clumped spheres, following the approach of Favier et al. (1999), and bonded spheres using the method developed by Brown et al. (2014). Since the

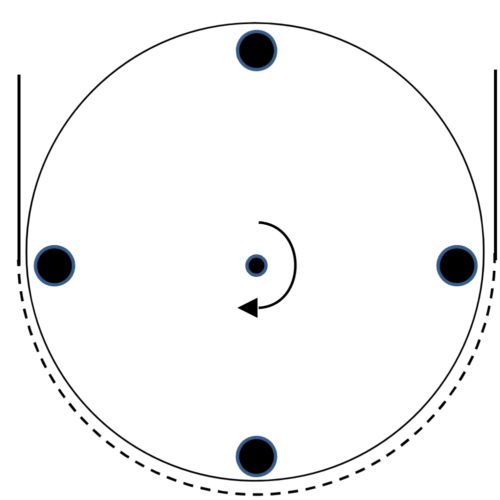

Fig. 1. Bar mill geometry.