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Particle size segregation promoted by powder flow in confined space: The die filling process case

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ABSTRACT

Tablet compression has great significance in the pharmaceutical industry since most of the drugs are in the tablet dosage form. The tablet press feed frame is used to fill powder into the empty dies. Die filling is one of the key steps to control final properties of tablets. Using the Discrete Element Method (DEM), a standard feed frame taken from a Manesty Betapress was simulated which represents the tableting process without the compression stage. DEM was used to understand the micro-macro dynamics of the particles inside the feed frame. Segregation behavior of a single material with a particle size distribution was investigated using this method. The DEM simulation components included 2 paddle wheel speeds (24 and 72 rpm) and 2 die disk speeds (29 and 57 rpm). Results obtained have highlighted the effect of feed frames on the powder properties. The DEM results show size segregation inside the feed frame and during the die filling stage. Velocity profiles and particle vectors show that the percolation phenomenon is the most significant segregation mechanism. Paddle wheel speed was demonstrated to be the most important factor to control particle size segregation inside the feed frame.

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1. Introduction

1.1. Die filling process

Pharmaceutical tablets are the most common and preferred solid dosage form due to simplicity of manufacture, cost of manufacturing, convenience of administration and stability [1]. Most tablets are manufactured using rotary tablet presses, where powder is filled into the dies with a feed frame and then compressed with punches, decompressed, and finally ejected. The whole die filling process is complex and dynamic and entails different stages that present distinctive features. The first stage is the powder flow from the hopper to the feed frame. The second stage is the powder flow through a confined space, which is the feed frame, forced by the paddle wheels to the die opening. And the final stage is the tablet press die-filling step. This 3-stage die filling process is one of the key steps to control the final properties of tablets. Tablet properties are the results of the powder material properties and powder behavior during all the previous stages. Therefore, the proper understanding of the feed frame system and powder behavior through the previous stages is important to reduce or avoid the possible problems during tablet manufacturing and to accomplish a successful tablet processing [2].

Some tablet problems are due to poor die filling such as, inhomogeneous density distributions [3] and tablet die weight variability. Weight variation between tablets with respect to dose and weight must be reduced to a minimum to offer people the best quality product. Uniformity of weight can be controlled from the die filling stage, which therefore ensures consistency of dosage units during the later stages of compression, decompression and ejection. Particulate materials may segregate upon processing and handling based on differences in particle properties such as size, density, or shape. This is important for the pharmaceutical industry to optimize the operation of feed frames to avoid segregation problems.

1.2. *Experimental die filling studies*

According to our literature review, there have been few publications dedicated to powder behavior inside the feed frames. Most of the knowledge resides with the equipment manufacturers and it is not readily available. Nevertheless, the die filling process has been studied extensively. However, the system used in previous publications to study this process is simple and does not take into account significant and complex features of a real tablet press feeding device (feed frame). These studies used a simple shoe delivery system that consists of a rectangular cross-sectioned box with a constant velocity over the die and a high-speed video system to determine powder flow behavior during the die filling process [2,4–8]. High shoe speed can result in a lower filling rate and in some cases, incomplete filling [9]. Other experiments have been done with a circular cross-

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sectioned feed shoe in order to investigate uniformity and pressure distribution inside dies [10]. Increasing shoe speed has a positive impact on pressure uniformity and symmetry index [11].

1.3. Computational die filling studies

Experimental measurements of particle flow are often problematic or expensive to make. Some locations and particle properties are difficult and challenging to investigate experimentally. DEM models can be used to overcome these difficulties, reduce the number of experiments, and optimize design and operating conditions. DEM models the behavior of each particle and the overall system performance is the result of all the individual interaction. The number of particles DEM can simulate is limited to the computational power because of the extensive calculations that this method entails. DEM has been widely used to simulate pharmaceutical processes and powder phenomena such as particle mixing, tablet coating [13], milling [14], sieving [15], particle flow behavior [16], size segregation [17] and other pharmaceutical operations.

DEM is a common technique to study particle flow and specially die filling [3,18–22]. It has been demonstrated that DEM can capture major features of the die filling process [18,23]. Powder trend and behavior are consistent experimentally and numerically during this process [23]. Different particle shapes and pressure build-up have been studied in terms of powder flowability and critical velocity [23]. This process could provide a non-homogeneous density distribution during powder transfer but it has a small effect on the final density distribution of the compacted component. It was demonstrated that the non-homogeneous density distribution is mainly influenced by the combination of cavity geometry and feeding shoe velocity [2,3,18]. Cohesive materials slow down the die filling process and also affect negatively the die weight variability and the force required for compression [20]. All the previous mentioned systems do not appear to model the feed frame system to a significant degree.

1.4. Particle size segregation

Particle size segregation was evaluated during die filling in the presence of air using a coupled discrete element method (DEM) and computational fluid dynamics (CFD) [21,22]. The filling process was studied with a stationary and a moving shoe [21,22]. Reducing particle density ratios and promoting bulk flow by increasing shoe speed can provide less segregation during the die filling process [21]. Vertical size segregation occurs during die filling since fine particles can percolate through the coarse particles [22]. Air can reduce this segregation since air drag effect can suppress the percolation of small particles [22]. The powder flow rate in presence of air is less than in vacuum because of the resistance of air. The air effect is negligible when nose flow is predominant because air can escape from the die cavity [22]. The percolation velocity is affected mostly by particle size and particle density, restitution and friction coefficients between particles, and void fraction of packing [24]. It also increases with reducing restitution coefficient and particle size and is related to the number of collisions and the intensity of those collisions between particles [24].

The main focus of this paper is to study the segregation phenomena using DEM simulations inside a tablet press feed frame, compare the results experimentally and describe the phenomena due to differences in particle size. This work is also focused on the feed frame processing and characterization, die filling stage, understanding of powder behavior during die filling and possible effects on tablets. The results are divided in five sections, feed frame mass hold-up, exits contributions to die filling and die weight variability, velocity profile, DEM particle size segregation and experimental results.

2. Methodology

2.1. Computational Study (DEM simulations)

During the tablet press operation, powder flows through a feed frame, enters a die, and then is compressed into tablets. This makes it impossible to determine powder properties as it is entering the die. To determine the properties and behavior of the powder inside the feed frame and dies, two main methods were used. The first method was to use a discrete element method (DEM) to answer the uncertainties of flow behavior and segregation within the feed frame and dies. The second method was experimental and relied on decoupling the feed frame from the tablet press with a custom made die disc without the compression step. This method was also used to validate the DEM simulations. The apparatus is a standard feed frame taken from a Manesty Betapress and a simulated die disc, which represents die filling process without the compression step. The feed frame has two paddle wheels. One of them rotates counter-clockwise and the other one rotates clockwise, while the die disk rotates clockwise (Fig. 1). A previous publication [23] explains in more detail the description of the Manesty Betapress feed frame. EDEM software was used to perform all the simulations. The system simulated was first build in AutoCAD® and then the drawing was imported to EDEM (DEM Solutions). The simulated feed frame system is shown in Fig. 1.

The parameters used in the simulations are presented in Table 1, where the particle properties are similar as those used in the literature [12,26–32]. Our computational power was also taken into consideration for the selection of the particle size. These simulation parameters values describe the particle properties affecting significantly the results. The cohesion model used for the simulations modifies the Hertz-Mindlin model by adding a normal cohesion force. This force takes the form: $F = kA$, where A is the contact area and k is a cohesion energy density with units Jm^3. The value of k for the simulations was 500.

In order to analyze the data from the simulations, selections were created at different locations of the feed frame. Selections allow data to be extracted from a particular area in the domain. Also, it helps to monitor any particle through a particular area. Data can be exported based on these selections. All the simulations were evaluated using the same Manesty Beta Press feed frame design. The design of experiment components includes 2 paddle wheel speeds (24 and 72 rpm) and 2 die disk speeds (29 and 57 rpm). These operating values were selected based on typical tablet press operation speeds used in the pharmaceutical industry.

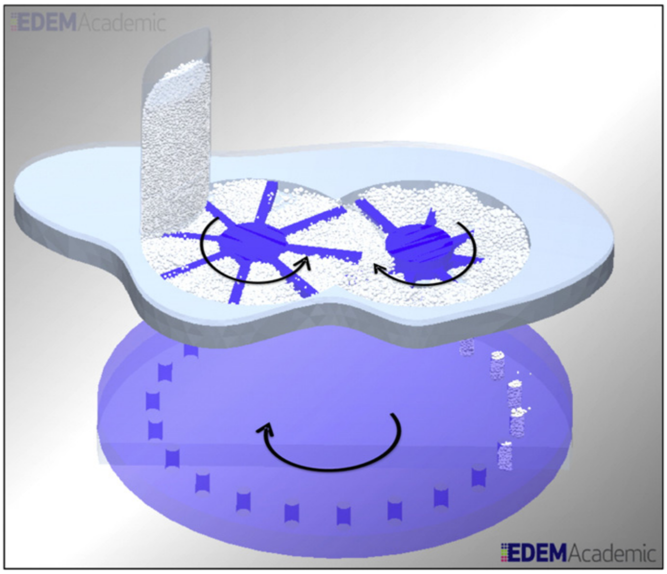

Fig. 1. Tablet press feed frame simulated in EDEM.

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