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Relationship between residence time distribution and forces applied by paddles on powder attrition during the die filling process

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ABSTRACT

The die filling process, which happens prior to the compaction step, affects directly the quality of the final product (tablets). As paddles pass through the powder bed inside the feed frame, shear is applied to particles. Therefore, it is important to establish a relationship between residence time distribution (RTD) and forces applied by the feed frame paddles on powder attrition and overlubrication during the die filling process. The pulse injection method was used to study the RTD. The discrete element method (DEM) was also used to measure the RTD of particles by size ranges and the mean residence time (MRT) inside the feed frame. The experimental and DEM simulation design of experiments included 2 paddle wheel speeds (24 and 72 rpm) and 2 die disk speeds (29 and 57 rpm). Experimental results show that higher paddle wheel speeds lead to lower mean residence time, narrower RTD profiles, and larger number of paddle passes. The simulations RTD profiles are similar to an ideal continuous stirred tank reactor (CSTR) profile with a sharp peak and a tail. Operating conditions, feed frame exit contributions, and outlet mass flow rate affect the MRT. The RTD analysis by particle size shows that particle size segregation occurs where the smaller particles are leaving the feed frame faster. The level of powder confinement inside the feed frames can have a significant effect on powder attrition. As particle size increases, the compressive and tangential force applied to it increases. The tangential force data show that the level of attrition is also affected by the paddle wheel speed. This study explains in detail different factors that can affect the residence time distribution and the force applied to particles that can ultimately cause particle breakage and powder overlubrication.

1. Introduction

1.1. Die filling process

The die filling process, which happens prior to the compaction step, affects directly the quality of the final product (tablets). This process consists mainly of three steps: powder flow through the tablet press hopper, powder flow promoted by paddle wheel movement through a confined space (feed frame) and powder transfer from the feed frame to the dies [1]. Different groups have studied this third step extensively [2–7], but the focus of this work is on the powder phenomena during the second step.

The die filling process used to be treated as a black box. Recent studies [1,8–10] have contributed to a better understanding of this process during tablet manufacturing. A proper understanding can be used to avoid some problems such as high tablet weight variability and non-uniform tablet pressure distribution. As the paddles pass through the powder bed inside the feed frame, shear is applied to the particles. It has been demonstrated that this shear can cause powder attrition and therefore modify the particle size distribution (PSD) of the material [10]. The shear applied to the lubricated material was also demonstrated to affect powder overlubrication and, therefore, dissolution rate of tablets, tablet hardness, and powder flowability [9].

1.2. Residence time distribution in solid unit operations

The residence time distribution (RTD) is defined as the probability distribution of time that solid or fluid materials spend inside one or more units in a continuous flow system [11]. It is important to have an understanding of the material flow profile in every unit operation in order to characterize mixing and flow within the unit operation. It is also useful to develop models, design equipment, and troubleshooting. RTD studies are commonly used in many industrial processes such as pharmaceutical products and continuous manufacturing of chemicals, plastics, and food. There are many RTD applications in solid processes such as continuous mixers [12–14], extruders [15,16], rotary drums [17], fluidized beds [18] and centrifugal mills [19].

It has been demonstrated that the second step in the die filling process could generate powder attrition and over lubricate the material [9, 10] due to the amount of shear force applied to the particles. In consequence, it is important to establish a relationship between RTD, powder

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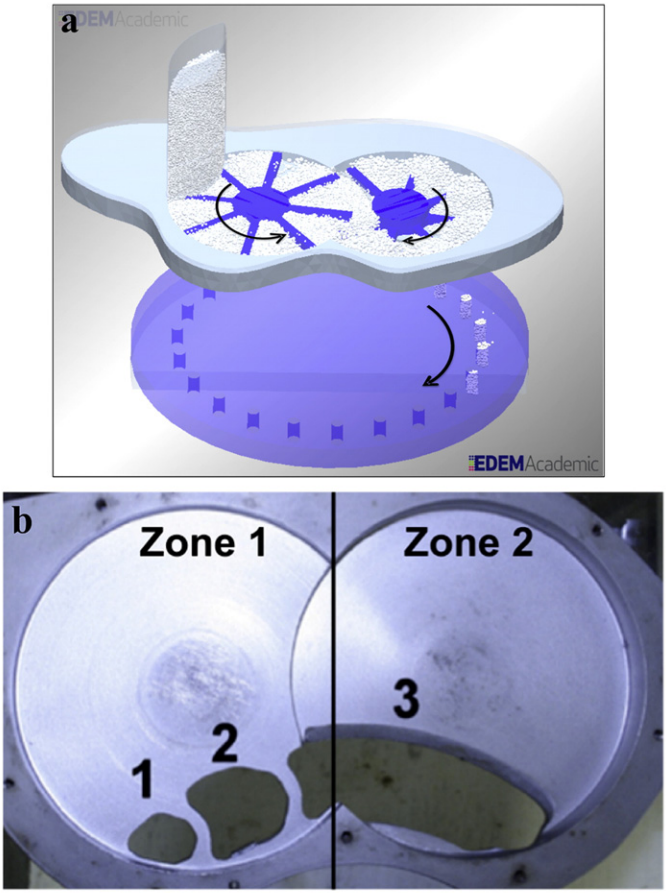

Fig. 1. Manesty Beta Press feed frame exits and zones.

breakage, and powder overlubrication. The RTD analysis is also important to understand some particle phenomena inside the feed frame such as particle size segregation. There are two common ways to perform RTD measurement: pulse injection and step change of a tracer at the inlet of a continuous system. Previous work [8] on RTD analysis during the die filling process has used the step change measurement. To our knowledge, none have used the pulse injection measurement for feed frames which is the method used in this work.

DEM has been used to study pharmaceutical processes such as feeding [20], mixing [13,21], granulation [22], and coating [23]. One advantage of measuring RTD using DEM is that this method does not

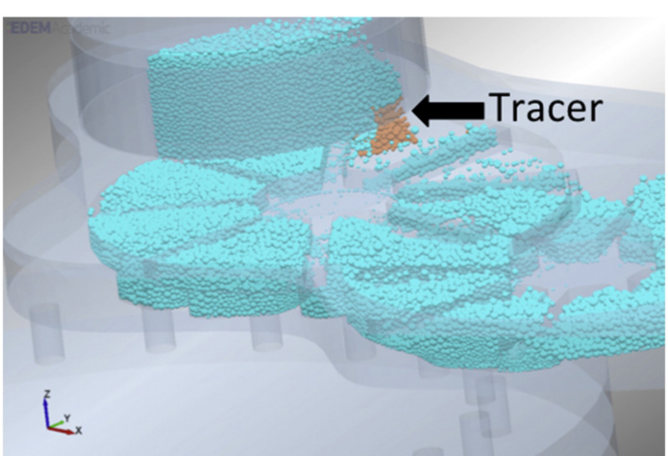

Fig. 2. Manual selection of tracer.

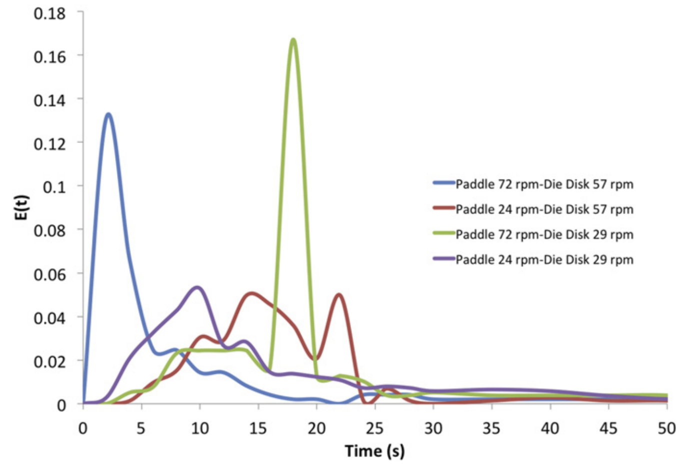

Fig. 3. $E(t)$ distribution as function of operating conditions.

introduce the problem of tracer disturbance since a group of all particles in the system is tracked. It also provides the capability of measuring the RTD of particles by size ranges. To achieve reliable conclusions, a correlation with computational data (DEM) and experimental data should be established. The main goal of this work is to develop a relationship between residence time distribution (RTD) and the forces applied by the feed frame paddles on powder attrition and overlubrication during the die filling process with experimental and numerical RTD data.

2. Materials and methods

2.1. Experimental section

2.1.1. Pulse injection tracer experiments

The materials used in the experimental study were Tablettose 70 Agglomerated Monohydrate Lactose Ph. EUR/USP-NF/JP (Malkerei Meggle Wasserburg GmbH & Co.) with 125 μm median particle size and Naproxen sodium (Zhejiang Tianxin Pharmaceutical Co.) with a median particle size of 28 μm as the tracer. The equipment used in this study consists of a loss in weight feeder (Gericke, model 153) to feed the lactose at a controlled rate, and the Manesty Beta Press feed frame. The feed frame has two paddle wheels. One of them rotates counterclockwise and the other one rotates clockwise, while the die disk rotates clockwise (Fig. 1a). It also has three exit holes with different sizes and shapes (Fig. 1b).

The design of experiment (DOE) components includes 2 paddle wheel speeds (24 and 72 rpm) and 2 die disk speeds (29 and 57 rpm). These operating conditions were selected based on typical tablet press operation speeds used in the pharmaceutical industry. The lactose went through the feeder and entered the feed frame at a feed rate of around 410 g/min. The feed frame was operated for 2 minutes before taking samples. This period minutes represents approximately eight times the time it takes for the system to reach mass steady state in the DEM simulations. After this preliminary period, 5 g of Naproxen was added to serve as the tracer. Samples were taken at the exit of the dies every 2 s for the first 30 s, every 5 s until the 2 min mark and every 10 s until the 3 min mark.

2.1.2. UV–Vis spectroscopy

UV–Vis spectroscopy was used to measure the concentration of Naproxen in each sample and then determine the residence time distribution. The calibration curve for concentration of Naproxen was obtained using a Genesys 10S UV–Vis Spectrophotometer at 318 nm to determine the residence time distribution (RTD). Subsequent dilutions that ranged from 0% to 50% were prepared from a stock solution, and their absorbance was measured. Validation samples were obtained to