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Original Research Paper

Consolidated states of dilation in granular material: Experimental investigation and comparison of rheological properties

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ABSTRACT

The intention of this study was to investigate the consolidation states of dilation of granular material with respect to mechanical shear, size and component fraction, geometry and scale of the processing equipment. Dilation of pharmaceutical powders and mixtures was measured in a rotating drum at various speeds of rotation (10, 15, 20, 25 rpm). It was interesting to note that the dilation did not change with an increase in the rotational rate of the cylinder. Furthermore, studies carried out in various sizes of cylinders showed that the dilation was poor in the lower and higher volume of cylinder. An optimum volume of the cylinder with highest dilation was found to exist in the middle scale. Decrease or increase in the cylinder volume hindered the dilation of powder. Lubricated blends exhibited higher bed density than the non-lubricated blends, and therefore showed lower dilation. More interestingly, higher dilation was achieved at 35–45% of fill level of cylinder. Any further increase or decrease of powder fill level beyond this range drastically decreased the ability of powders to dilate. Eventually, dilation of powders decreased with increased in powder bed density. Cohesive materials when mixed with free flowing powders could improve the flowability (reduced dilation) only when they constituted 40–60% of blend composition. Percentage of free flowing material below this composition was found to be ineffective in reducing the dilation.

1. Introduction

Poor flow properties of powders are known to cause problems in manufacturing process and powder handling in pharmaceutical industries. Shear forces and particle attrition during the movement of granular material were known to be some of the important reasons impacting powder flow properties [1–3]. Flowability of powders can be limited depending on the ability to dilate which in turn was known to be dependent on the particle coagulation time [4]. Despite recent technological advancements, cohesion and improper dilation of granular materials can often lead to batch failures. Moreover, cohesion was believed to be dependent on the operation parameters [5]. Likewise, particle size, length of the chamber, velocity and amplitude of wall displacement can affect powder dilation [6,7]. As a result, the powder mixture can lose the ability to consolidate resulting in improper distribution of constituents in the formulation. Therefore, it would be useful to understand the factors contributing to the dilation of powders and develop an alternate method of mixing which can enhance the consolidation of the mixture.

An interesting way to improve the flowability of powders is to identify and optimize the region of dilation in which granular flow exists without interstitial porous gaps due to cohesion. Without improving dilation, this task is often more challenging when the size of the vessel is changed, scaled up or down or processed through various geometries of constriction. For example, it was believed that a powder in order to be effectively discharged through a constriction in a hopper should have significant dilation [8]. By evaluating the friction between grains, it was showed that a randomly packed irregularly shaped particles exhibited wider distribution of stress [9,10]. Similarly, a critical steady flow regime was identified at higher dilation level [11]. Normally, large friction leads to shear resistance and low bulk density thereby delaying the horizontal momentum transfer [12]. Given that dependency on density, it is perhaps more important to consolidate the granular flow in variable density mixtures.

The challenge of content uniformity and constituent consolidation in the mixture lies in the ability of the powder to mix with enhanced flow. As widely observed, morphological properties and dilation density are potential evaluation parameters for

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consolidation index [13,14]. Homogeneity in the mixing process was known to be affected by axial or radial mixing, fill levels and rotational speeds of the blender [15]. Recently, it has been suggested that the mixing mechanism was a combination of azimuthal, axial and radial mixing [16]. Hence the dilation aspect in the powder mixture will vary depending on the type of mixing as well. Most of the dilation problems in mixing processes mentioned in the literature [17–19] can be attributed to particle geometry and size factors rather than the operation factors. Even though, these processes were tried and tested with respect to intrinsic material properties, they invariably require methodologies which identify the limitations of vessel geometry. It should be noted that qualitative flow characteristics should be carefully selected for good mixing performance [20,21].

Thus, there is clearly a need to identify the boundaries of dilation in order to consolidate the powder flow with respect to scalable geometries. Hence, in this study, we report our initial investigation focused on identifying consolidated regions in powder flow dependent on intrinsic powder properties as well as geometric factors of the mixing equipment. An investigation on dilation with respect to rotational rate of the vessel, effect of geometrical size of the vessel and fill fraction of granular media on extent on dilation and the role of granular media density on dilation is presented. Finally, the influence of constituents of the blend and composition on identifying the regions of consolidated composition and dilation regions is identified.

2. Materials and methods

2.1. Materials and sample preparation

A total number of eleven pharmaceutical blends were used in this study. The blends consist of lactose (Foremost Farms – 90 μ), microcrystalline cellulose (MCC – Avicel 101, particle size – 50 μ), magnesium stearate (MgSt) (Mallenkrodtt) and colloidal silica [Cab-O-Sil (CS), grade M5-P, glidant, particle size – 0.2–0.3 μ]. The blends were divided into five sets: (1) 100% Pure blends of fast flo lactose, avicel 101, avicel 102 and avicel 200. (2) The blends in set-1 were mixed with 1% MgSt and varying composition of Cab-O-Sil (0.2%, 0.4% and 0.6%). (3) Avicel 101 mixed with a varying composition of fast flo lactose between 10% and 90% (4) Avicel 102 mixed with a varying composition of fast flo lactose between 10 and 90% (5) Avicel 200 mixed with a varying composition of fast flo lactose between 10% and 90%. The composition of the blends is given in Table 1. Blends were mixed in a v-blender running with a shell speed of 15 rpm and intensifier bar at 500 rpm. A total quantity of 4 lb of each blend was collected from v-blender after mixing. All the blends were prepared when the measured humidity in the air was 40–50%.

Table 1
Blends and their composition used in the dilation study.

Blend	Composition
Set-1	
B1	Fast flo lactose
B2	Microcrystalline cellulose (PH 101)
B3	Microcrystalline cellulose (PH 102)
B4	Microcrystalline cellulose (PH 200)
Set-2	
B5	99% Microcrystalline cellulose (PH 101) + 1% magnesium stearate
B6	98.8% Microcrystalline cellulose (PH 101) + 1% magnesium stearate + 0.2% Cab-O-Sil
B7	98.6% Microcrystalline cellulose (PH 101) + 1% magnesium stearate + 0.4% Cab-O-Sil
B8	98.4% Microcrystalline cellulose (PH 101) + 1% magnesium stearate + 0.6% Cab-O-Sil
Set-3	
B9	90% Microcrystalline cellulose (PH 101) + 10% fast flo lactose
B10	80% Microcrystalline cellulose (PH 101) + 20% fast flo lactose
B11	70% Microcrystalline cellulose (PH 101) + 30% fast flo lactose
B12	60% Microcrystalline cellulose (PH 101) + 40% fast flo lactose
B13	50% Microcrystalline cellulose (PH 101) + 50% fast flo lactose
B14	40% Microcrystalline cellulose (PH 101) + 60% fast flo lactose
B15	30% Microcrystalline cellulose (PH 101) + 70% fast flo lactose
B16	20% Microcrystalline cellulose (PH 101) + 80% fast flo lactose
B17	10% Microcrystalline cellulose (PH 101) + 90% fast flo lactose
Set-4	
B18	90% Microcrystalline cellulose (PH 102) + 10% fast flo lactose
B19	80% Microcrystalline cellulose (PH 102) + 20% fast flo lactose
B20	70% Microcrystalline cellulose (PH 102) + 30% fast flo lactose
B21	60% Microcrystalline cellulose (PH 102) + 40% fast flo lactose
B22	50% Microcrystalline cellulose (PH 102) + 50% fast flo lactose
B23	40% Microcrystalline cellulose (PH 102) + 60% fast flo lactose
B24	30% Microcrystalline cellulose (PH 102) + 70% fast flo lactose
B25	20% Microcrystalline cellulose (PH 102) + 80% fast flo lactose
B26	10% Microcrystalline cellulose (PH 102) + 90% fast flo lactose
Set-5	
B27	90% Microcrystalline cellulose (PH 200) + 10% fast flo lactose
B28	80% Microcrystalline cellulose (PH 200) + 20% fast flo lactose
B29	70% Microcrystalline cellulose (PH 200) + 30% fast flo lactose
B30	60% Microcrystalline cellulose (PH 200) + 40% fast flo lactose
B31	50% Microcrystalline cellulose (PH 200) + 50% fast flo lactose
B32	40% Microcrystalline cellulose (PH 200) + 60% fast flo lactose
B33	30% Microcrystalline cellulose (PH 200) + 70% fast flo lactose
B34	20% Microcrystalline cellulose (PH 200) + 80% fast flo lactose
B35	10% Microcrystalline cellulose (PH 200) + 90% fast flo lactose

2.2. Methods

The dilation measuring unit consists of a cylinder rotating on a platform at various rotational speeds as can be seen in Fig. 1. The percentage expansion in powder bed in a rotating cylinder is measured in the dilation method. The unit consists of a drum backlighted with LED and a digital camera. Image processing of the shadow on the end of the drum is done by the dilation program. The powder in the bed was compacted by putting the cylinder filled with powder on an auto tap meter. Care was taken such that the powder was filled to 45% of bed volume and later compacted to be not less than 40%. For all experimental runs, dilation of the powder bed was assumed to be uniform along the axis of the cylinder. The powder was compacted by running the machine for 1000 taps. The cylinder was rotated at set rotational speeds as desired at the experimental run. Although the standard speed was set at 15 rpm for all experimental studies, some specific experiments involving the effect of rotational speed were tested with a drum speed

Fig. 1. Instrument used for measuring dilation. Powders were tumbled in a rotating cylinder at various speeds of rotation and the percentage change in bed expansion was captured using a LED lights on one end and a digital camera and a computer interface on the other end.

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