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Function of upstream and downstream conveying elements in wet granulation processes within a twin screw extruder

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ABSTRACT

Understanding the function of individual screw elements in twin screw wet granulation has become an area of intense study but has yet to contemplate the interactions of adjacent elements. The present work examines the influence of conveying elements around the critical compression zone created by a kneading block. Granulated samples were prepared using nine different screw configurations with flight pitches of 20, 30 and 40 mm and tested at two different degrees of channel fill utilizing a placebo formulation of 20 wt% microcrystalline cellulose in α-lactose monohydrate. Samples were taken directly from the upstream and downstream conveying elements as well as the machine exit and analyzed for particle size, shape, apparent porosity and fracture strength. The results showed that the particle size, shape, and fracture strength of granules produced were significantly influenced by the pitch of downstream conveying elements while upstream conveying elements had no effect on exiting particles. Both zones were found to be insensitive to the degree of fill, though the variance in measurements decreased at higher fill. Similar results were observed when the formulation was adjusted to included 15% ibuprofen (though the ratio of lactose to microcrystalline cellulose remained constant).

1. Introduction

In the literature for twin screw granulation, kneading blocks and comb mixing elements are consistently recognized by researchers as providing significant functions in granulation, directly impacting the properties of granules as well as finished tablets [1–5]. For the kneading block, the disc offset angle design can notably increase the breakage of oversized particles while producing granules with lower friability and highly regular shape [1,4,6]. Additionally, the friability or density of produced granules is strongly influenced by the length of a kneading block, i.e. the number of discs in its set. Higher numbers of discs or thicker discs lead to higher compaction of powders [4,6,7]. While these design features impact granulation and have been well researched a fundamental understanding of how screw elements both upstream and downstream of the kneading block impact granule properties does not currently exist. Furthermore, the degree of filling and extent of powder wetting can also contribute to the final granule properties. It is the purpose of this paper to better understand the influence of conveying elements that are normally adjacent to a kneading block.

A conveying element is principally defined by its number of flights and the pitch of those flights. The most common conveying element for co-rotating twin screw extruders is bilobal, meaning that two flights turn around the shaft at an offset of 180 degrees. The flight pitch (p), which refers to the axial distance between two adjacent flights along the length of the screw, is called 'square pitched' when it is equal to the inner diameter of the barrel (D), i.e. $p/D = 1$; the less commonly used term *flight lead* which refers to the axial distance corresponding to one turn of a flight will not be used in this paper. Being square pitched is considered a reference condition by screw designers, even in twin screw machines. A larger pitched conveying element (i.e. $p/D > 1$) has a larger volume that can accommodate more powder and is least likely to become pressurized under normal starve fed operation; it is commonly used in zones where materials are being fed into the extruder or liquid binder is added. A smaller pitched conveying element (i.e. $p/D < 1$) has a smaller volume and is more readily pressurized under normal conditions; it is often considered to be better suited to push material into fully filled zones like a kneading block. The relevance of flight pitch for a conveying element on wet granulation has been studied for screws without a compression zone (i.e. kneading block or comb mixing element) [6,8]. In those cases it was found that granule size and flowability were significantly influenced by pitch and flow rate. However, these finding may not extend to zones around a compression screw element like a kneading block. Findings by van Melkebeke and coworkers [4] have noted that conveying elements downstream of a kneading block were required to reduce the fraction of coarse granules (>1 mm) exiting their process, thereby improving the granule yield. Their results imply a synergistic relationship with respect to the arrangement of screw elements beneficial to the particle size control generally attributed to a kneading block [3] however

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guidance to screw designers on how to use that understanding for twin screw granulation has not yet been provided.

This article examines the influence of flight pitch on the function of upstream and downstream conveying elements neighboring the compression zone of a screw design for wet granulation. Three values of flight pitch were employed (p/D = 0.67, 1, or 1.33) for the zones before and after the kneading block as well as two different degrees of screw channel fill based on changed flow rate for a fixed screw speed.

2. Materials and methods

2.1. Materials

Two formulations were used in this work. The majority of trials utilized a placebo formulation containing 20 wt% microcrystalline cellulose (Avicel PH 101, FMC Biopolymers Corp; Philadelphia, PA) with α-lactose monohydrate (Meggle Pharma Flowlac 100, Mutchler Inc.; Harrington Park, NJ). In a small set of trials, a second formulation containing 15 wt% ibuprofen USP (Spectrum Chemical; Gardena, CA), 68 wt% lactose monohydrate and 17% microcrystalline cellulose was used. The binder solution consisted of 4% hydroxypropyl methylcellulose (METHOCEL™ E3PLV Cellulose Ethers, The Dow Chemical Company; Midland, MI) in distilled water.

2.2. Twin screw granulator

Granular samples using the placebo formulation were prepared by a ZSE-HP 27 mm 40 L/D co-rotating intermeshing twin screw extruder (American Leistritz Extruder Corp.; Sommerville, NJ, USA) with nine different screw configurations. The general screw configuration, which closely resembled the design used in an earlier study of the kneading block [3], is shown as a schematic drawing in Fig. 1. The design consisted of conveying elements spanning the length of barrel zones Z0-Z8 which served the purposes of wetting, nucleation and early granular growth. A 10-disc 60° offset kneading block where its discs were bilobal and 5.6 mm thick. The last half of barrel zone Z8 and barrel zone Z9 show additional conveying elements at the end of the machine. The conveying zones under study and highlighted in the figure were 90 mm in length, placed immediately upstream and downstream of the kneading block and consisted of flight pitches of 20, 30 or 40 mm; note that the 30 mm pitch is considered square pitched for this machine. The length of each conveying zone (i.e. 3 L/D) was chosen such that it was long enough to exert its own influence on granule development yet not so long that the effect could not be reasonably ascribed to the compression taking place within the kneading block. The nine designs corresponded to a full factorial experimental design (3^2) exploring the three flight pitches for the two zones. All collected samples were referenced in this study based on their 'flight pitch combination', for example '20–40' which denoted granules collected from screws with an upstream pitch of 20 mm and downstream pitch of 40 mm.

Each screw configuration was tested at two flow rates, 5 kg/h and 15 kg/h (referring to powder addition), which produced an estimated degree of channel fill of 10% and 30%. The premixed powder formulation was fed into the feed zone (Z0) of the extruder by a T-20 gravimetric feeder (Brabender Technologie Ltd.; Mississauga, ON). The binder solution was fed as a foam (85% foam quality, [9]) into the second barrel zone (Z2) by means of a side stuffer. The site of liquid addition was set a considerable distance back from the kneading block in zone Z8 to ensure only uniformly wetted solids were under study; the uniform coloring of the powder by a dye included with the binding liquid was taken as evidence of uniform wetting in the zones of study. The mechanical foam generator was supplied by The Dow Chemical Company (Midland, MI), which consisted of separate liquid metering and gas flow rate controllers. The liquid-to-solids (L/S) ratio of 30% was selected to maximize the yield of particles in a desired size range of 0.5–2 mm. All experiments were performed at a constant barrel temperature of 35 °C and at a constant screw speed of 300 RPM.

A smaller (2^2) experimental design was conducted with the formulation containing ibuprofen looking at the 20 mm and 40 mm pitch only, and only at 15 kg/h. A L/S = 38% was required to adequately granulate this formulation. This smaller study was completed to gain understanding of how the results would differ for a non-placebo formulation.

2.3. Granules collection

Granular samples were directly collected from both the upstream and downstream conveying elements of interest in this study as well as from the extruder exit, after 5 min of stable operations. The *Screw Pullout* method, described in an earlier paper [3], was employed for direct sampling of granular material from these two conveying zones as well as allowed direct observation of the granulation process. To prevent the screws from slowing down gradually, the granulation process was abruptly halted to preserve the state of granules inside. Screw pullouts were repeated 3–4 times for each trial condition to collect a reasonable sample size (~5 g) for particle size determination; this sample size showed comparable uncertainty to measurements at the exit of the machine. Samples were air dried at 35% relative humidity for two days and then sealed in bags prior to testing. Final moisture content was less than 1.8% for all samples, based on analysis using a Mettler-Toledo HG63 moisture analyzer.

2.4. Particle size analysis

The particle size distribution (PSD) was determined using a Ro-Tap RX-29 sieve shaker (W.S. Tyler Inc., Mentor, OH, USA) with nominal openings of 3350 μm, 2360 μm 1180 μm, 850 μm, 500 μm, 250 μm, 125 μm, as well as a bottom pan. The amount of sample used for PSD characterization was approximately 5 g for the upstream and downstream conveying elements and 100 g for the collected granules at the extruder exit. Each sample was sieved by mechanical agitation for 5 min which was found sufficient to separate granules weakly bound together by drying without influencing the state of granulation produced by the process.

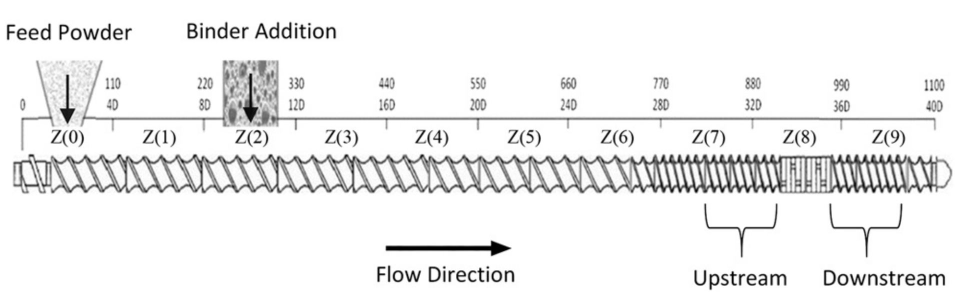

Fig. 1. Extruder configuration.