Aspects of Screw Design for Wet Granulation Extrusion

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ABSTRACT

This paper discusses our preliminary trials examining the particle size and particle shape of granules produced by different screw elements in a co-rotating twin-screw extruder. In general, conveying elements did not provide the necessary consolidation to allow large particles to grow in significant quantity, whereas the different mixing elements led to the large particles desired for tabletting. Granules from mixing elements varied from near-spherical to chains to grains to plate-like (disk) shapes depending on the type selected.

INTRODUCTION

In the development of twin-screw extrusion for granulation, current research has primarily focused on understanding the influence of process parameters and formulation variables on particle size and final tablet properties. Kleinebudde [1] studied the influence of water content and screw speed on the performance of the extruder. Remon's research group at Ghent University [2-4] studied the granulation of -lactose monohydrate in a twin screw extruder using an aqueous polyvinylpyrrolidone (PVP) solution as a binding agent. The authors found that granulation of lactose could be done with considerably less water in an extrusion process compared to a high shear mixer [2]. A group at Pfizer [5] studied wet granulation in a co-rotating twin-screw extruder and provided the first insight into the importance of screw design. They tested conveying elements and two mixing elements, kneading blocks (dispersive mixer) and discharging elements (distributive mixers). Kneading blocks tended to block the flow yet the discharging elements (otherwise known as comb elements) provided granulate discharge with evenly sized particles.

With twin screw extrusion as a new potential technology for particulate processing, it is likely that new screw element designs will have to be developed in order to satisfy the unique issues of processing granular matter; however, it is first necessary to establish the performance of available elements. To that ends, the aim of this paper is to discuss the granulation features of commercially available screw elements.

EXPERIMENTAL

MATERIALS - Lactose monohydrate was used as excipient (EMD Chemicals Inc., CA). Polyvinyl-pyrrolidone 100 GM (Alfa Aesar, M.W. 40000) was selected as the binder. No model active ingredient was included in the results discussed in this paper.

PROCEDURE - The experiments were performed in a 24 L/D Leistritz ZSE 27-HP co-rotating twin screw extruder using a simple screw profile, with an example shown in Figure 1. Different screw designs were examined which differed in the elements located 180 mm from the screw tips. The element types tested were: 1) conveying element with a 30mm or 40mm pitch, 2) kneading block with elements that were 30°, 60° or 90° offset from one another, and 3) discharging (comb) element, forwarding or reversing flow capacity. Figure 2 shows examples of each element type. The temperature of all zones was kept at 30°C by water cooling. The throughput rate was held constant at 2 kg/h while the screw speed was set to either 30 RPM or 80 RPM; the degree of channel fill was fixed as either 30% or 70% by these conditions.

The prepared binder solution consisted of 33 wt% PVP in distilled water. The binder solution was tumble

Fig 1. Example screw design with the highlighted section indicating the variable zone.

Fig 2. Examples of different screw elements

blended at 5-12% (w/w) with the excipient at 5-12% (w/w) for the trials. The pre-blend mixture was added to the extruder from a Brabender T20 twin-screw gravimetric feeder.

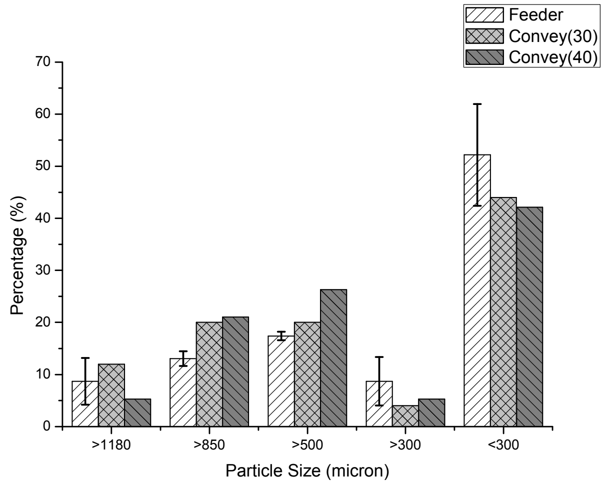
CHARACTERIZATION - After processing the extruded samples were air dried overnight at room temperature. For brevity the analysis discussed is limited to particle size distribution (PSD) and granule shape. The particle size distribution was determined by a mechanical sieve with screens of differing mesh size. The granule shape was inspected by reflective light optical microscope.

RESULTS AND DISCUSSION

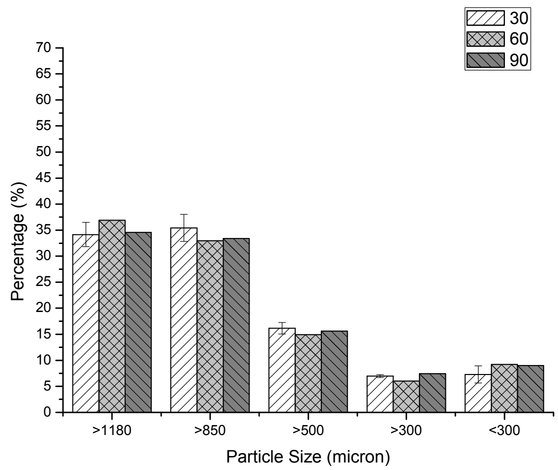
For Sections 3.1-3.2 the binder solution was mixed at 7.5 wt% with the lactose, based on optimal operating conditions determined by Keleb [2, 3].

INFLUENCE OF SCREW DESIGN ON PSD

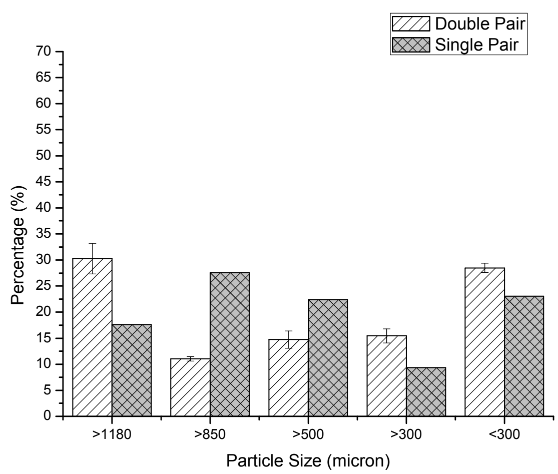
The effect that the different shear field of each element had on granulation, i.e. balancing particle growth versus rupture, was primarily assessed by particle size determination. The data disclosed pertains to the 30% degree of channel fill state and samples were taken at the element in question. Figure 3(a) compares the PSD for material entering the extruder from the feeder with samples from a 30mm or 40mm pitch conveying element. We see minor particle growth for the lactose/binder mixture in the 500-800μm region, preferentially for the element with the 40mm pitch. The greater free volume of the larger pitch will have comparatively lower shear on the particles and give less opportunity for rupture. Conversely, the kneading elements produced much larger particles as seen in Figure 3(b) which shows the PSD for the kneading block with elements at 30°, 60°, and 90° offset. Surprisingly there was little difference between the three blocks despite their different capacity for drag conveying versus pressure driven flow. Unlike the near-spherical granules found using the conveying elements, these particles formed plate-like shaped granules in the kneading elements. The lactose powder was compressed into thin disks within the nip region between two intermeshing elements. Finally, Figure 3(c) shows the PSD for the discharging element (two pairs of forwarding elements one after another or a single reversing pair). Both configurations resulted in particle growth, though the single pair exhibited more evidence of growth in balance with particle erosion. For the forwarding set (double pair), granules were nearly spherical whereas for the reversing pair, the exiting granules looked like reptating chains. With the reverse discharging element, too much backwards pressure occurred and the powder compacted too densely for the rupture mechanism



(a)



(b)



(c)

Fig 3. Effect of screw elements on PSD to control particle size.

All of the subsequent discussion, the particle size distribution corresponded to the die exit of the extruder.

INFLUENCE OF SCREW SPEED ON PSD

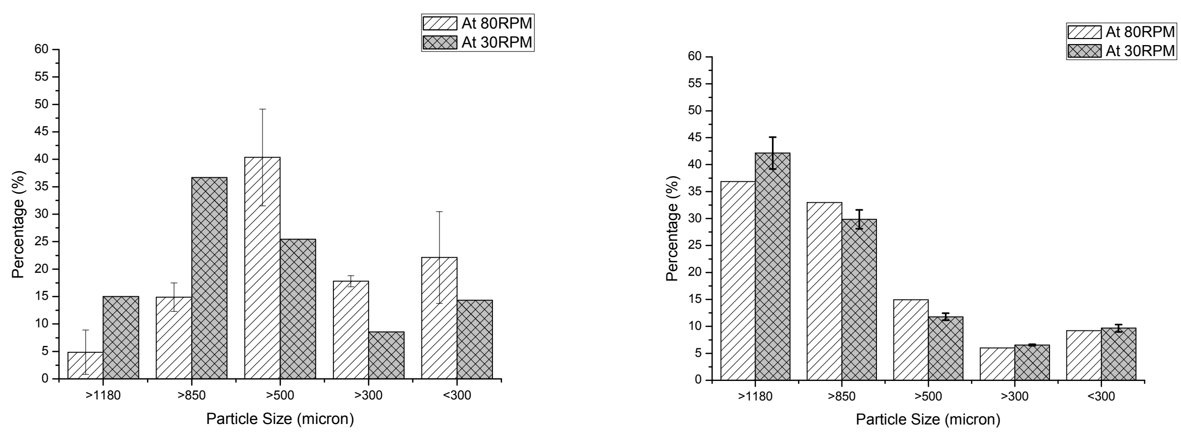
For any twin screw extruder, both mass flow rate and screw speed are important controllable parameters since they affect the degree of channel fill which in turn affects the residence time and mixedness of a sample. In our case, the flow rate was fixed and screw speed was used to control the degree of fill. The PSD of granules exiting the machine at 30 RPM and 80 RPM for the 30mm conveying element or 60° offset kneading block are shown in Figure 4. For the conveying element, slower screw speed means more fill and more material exposed to the shear plane (i.e. the barrel); however, we see that higher shear (i.e. 80 RPM) was more important to induce particle rupture in this case. For the kneading block, the degree of fill bears little importance by itself since the element will almost always completely fill, and so we see higher shear by increased screw speed led to only a minor increase in particle rupture.

INFLUENCE OF BINDER CONCENTRATION ON PSD

The aqueous binder concentration is a critical factor on the morphology of granule. Granulation is only feasible within a certain water concentration range. Using the work of Keleb [2] to provide an operating window, four concentrations were examined in these experiments: 5%, 7.5%, 10%, and 12% (w/w). With 5% (w/w) concentration, granulation failed for all screw designs because of high frictional stresses between the lactose and the extruder surfaces. On the other hand, with 12% (w/w) concentration, the high proportion of water in the extrudate only resulted in a paste due to the efficient dispersion of the binder within the powder during extrusion. Figure 5 shows PSD for 7.5 and 10% (w/w) binder concentrations at 80 RPM. Across all screw elements examined, the higher binder concentration led to larger granules, in particular there was a notable loss of fine particles below 500 μm.

BINDER INJECTION VS PREBLENDING

As an alternative to the preblending method, the lactose was added by the feeder while the aqueous PVP solution was metered into the feed opening as liquid droplet by a peristaltic pump (Masterflex C/L). By droplet addition, granulation is controlled by immersion nucleation rather than distribution nucleation mechanism which results in poor binder spreading and large granules (>500 μm) in the both screw designs due to limited shear. Future work will compare the use of a dual manifold spray nozzle to the preblending method and droplet method.



(a) (b)

Fig 4. Effect of degree of channel fill on PSD, a) 30mm pitch conveying element and b) 60° offset kneading block.

CONCLUSION

The screw element selected determines the particle shape and particle size distribution of exiting granules in granulation extrusion. Granules varied from near-spherical to chains to grains to plate-like (disk) shapes depending on the screw element selected. Conveying elements did not provide the necessary consolidation to allow large particles to grow in significant quantity, whereas the different mixing elements led to the large particles desired for tabletting. Further experimentation will help to understand the rupture mechanism between the different mixing elements.

ACKNOWLEDGMENTS

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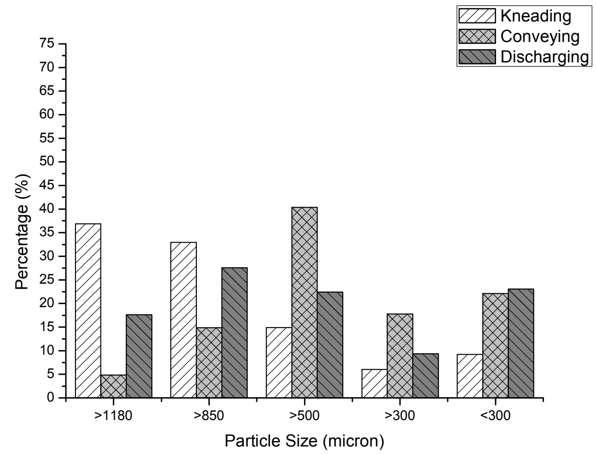
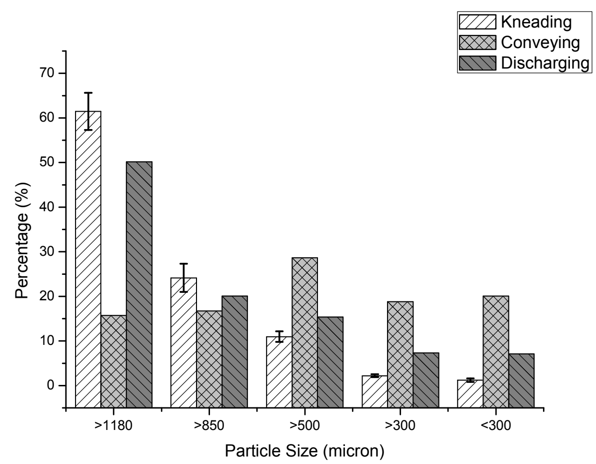


(a)



(b)

Fig. 5. Effect of aqueous binder concentration on PSD, a) 7.5 wt% binder and b) 10 wt% binder.