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Understanding wet granulation in the kneading block of twin screw extruders

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HIGHLIGHTS

- In situ examination of granule development inside extruder.
- Granule based on compression, compaction and fragmentation in kneading block.
- Granule structure controlled fracture strength of the wetted mass before compaction.
- Formulation dependent effects on granule development observed.

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ABSTRACT

The adoption of twin screw granulation to pharmaceutical manufacturing requires detailed understanding of the process, with one notable area of influence being screw design. A kneading block is a common screw element that is used to compress the wet granular mass, seemingly controlling the particle size and fracture strength of an end product. The present study provides a detailed examination of how this screw element functions for the wet granulation process, looking at the influences of feed rate (10–30 kg/h) and formulation on its operation. Trials were done in a 27 mm twin screw extruder with different powder formulations consisting of lactose monohydrate, microcrystalline cellulose and hydroxypropyl methylcellulose which were wetted with a foamed binder. The process was characterized by its pressure profile and residence time distribution. A visualization technique known as 'screw pullouts' was used to directly observe the state of granulation along the screw length and samples were collected from each screw segment for particle size and porosity analysis. The kneading block was found to provide a unique compaction–fragmentation step that increases the consistency and strength of a granular product. However, its effectiveness in doing so is strongly influenced by the granular state of powders entering this non-conveying element, which is formulation dependent. The compressive stress developed in the kneading block appeared generally low and allowed for deagglomeration or fragmentation of the agglomerated mass in downstream conveying zones depending upon the fracture strength of the powder. For the formulation containing microcrystalline cellulose, the kneading block served no function whereas for other tested powders, it was highly influential to granule size and porosity. The most notable benefit of the kneading block to all tested materials appeared to be distributing the interstitial binding liquid rather than to compact the powders.

1. Introduction

With growing interest in continuous granulation technologies like twin screw granulation, the majority of studies have focused on characterizing their final products against more familiar batch process operations. In regards to familiar processing variables, namely binder concentration, binder viscosity, and binder drop size relative to the primary particle, all these influence granulation in the same manner whether done in an extruder or in a high shear batch mixer (Keleb et al., 2004). This similarity has led some to propose nucleation regime maps following methodologies developed for high shear batch mixers (Tu et al., 2013). However, this classical approach was found to have limited usefulness in describing twin screw granulation. Unlike the batch mixer which shows only a few design variants based on the equipment

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manufacturers, the screw design of a twin screw extruder is readily manipulated by the user into a multitude of configurations based on a large selection of conveying elements and non-conveying elements (most notably but not exclusively being kneading blocks and combing elements). This means that a nucleation map is not manufacturer-specific but rather will change each time a process incorporates a new screw design. The sensitivity of granule properties to the size of the extruder (Djuric et al., 2009; Shah, 2005) will further restrict how broadly a nucleation map may be used from lab scale to production scale operations. Success in predicting the operations of an extruder should start with understanding the elementary functions of its screw elements upon granule development, as well as recognizing the additive influence of screw elements immediately upstream and downstream. The purpose of this paper is to examine granule size development in one of the most popular non-conveying screw elements of a twin screw extruder, the *kneading block*, and recognize upstream and downstream influences on its performance.

Non-conveying elements like kneading blocks have a distinctive role in the process since unlike conveying zones where the extruder operates starved, these zones of the screw are fully filled with powder and are pressurized, except under states of extreme starvation which have no practical economic value. The kneading block is the most commonly used type of non-conveying element in screw designs, whether for wetting, granulation, or hot melt extrusion in the pharmaceutical field. The term 'block' refers to the fact that it is comprised of a stack of kneading discs offset from each neighbor by angles of 30°, 45°, 60° or 90°. Each disc is normally bilobal, meaning it has two tips, and is shaped such that side-by-side discs wipe one another as they rotate. Refer to the kneading block shown in Fig. 1 for an example of its configuration. Increasing the *offset angle* of kneading discs notably increases the fraction of coarse particles (> 1 mm) in the product and those larger particles exhibit a correspondingly lower friability as well as irregular shape (Djuric and Kleinebudde, 2008; Van Melkebeke et al., 2008; Thompson and Sun, 2010). Compaction of the wet powder due to increased compressive stresses on the material through the kneading block has been assumed as the principle cause for such large granule growth. The offset angle ceases to have any influence on particle size only when the process is highly starved and the channel experiences a low degree of channel fill (Thompson and Sun, 2010; Vercruysse et al., 2012) – likely a situation to rarely occur in industrial processes. The frequency of oversized particles can be notably diminished if the kneading block is not located at the very end of the screw but rather moved further back, which appears to affect the size but not the density of granules (Van Melkebeke et al., 2008). The friability or density of a granule is strongly influenced by the length of the kneading block, with a higher number of discs or thicker discs leading to a more highly compacted state for the powders (Van Melkebeke et al., 2008; Djuric and Kleinebudde, 2008; Thompson and Sun, 2010; Vercruysse et al., 2012).

While the design variables of a kneading block named above have a significant effect on the state of granule development in a twin screw granulation process, operating conditions are similarly important though in a less definitive manner. Feed rate is reportedly the strongest process variable influencing particle size and granule density/strength for screw designs including kneading blocks (Dhenge et al., 2012; Thompson and Sun, 2010; Thompson et al., 2012b) yet not in all cases (Vercruysse et al., 2012). Screw speed has a smaller influence on granule properties with larger extruders (27 mm dia.) (Thompson and Sun, 2010; Thompson et al., 2012b), though for small extruders (16–19 mm dia.) some have reported minimal influence (Dhenge et al., 2010; Vercruysse et al., 2012) while others have indicated a much stronger influence (Keleb et al., 2001, 2002). There appears to be no clear rule in the use of these process variables for the moment.

The mentioned studies above have all hypothesized upon the function of a kneading block but mostly without direct measurement or observation. The only rigorous study to date to look at the progressive development of granules along the screw and relate physical changes of those granules to the kneading block was done by Dhenge et al. (2012). They adjusted the configuration of a screw so that different zones of the original screw design were moved closer to the machine exit in order to build up a size profile for granule development along the screw length; samples were collected upon exit. In the present paper, a classic technique to the polymer industry was used, known as *screw pullouts*, to make direct observations and measurements of granules as they developed along the length of the screw. The purpose of this paper is to definitively identify the physical phenomena that are dictating granule development along the screws, particularly around a kneading block. The study is specifically focused on two critical variables affecting the state of compaction in the kneading block, namely feed rate and powder formulation. Other variables like

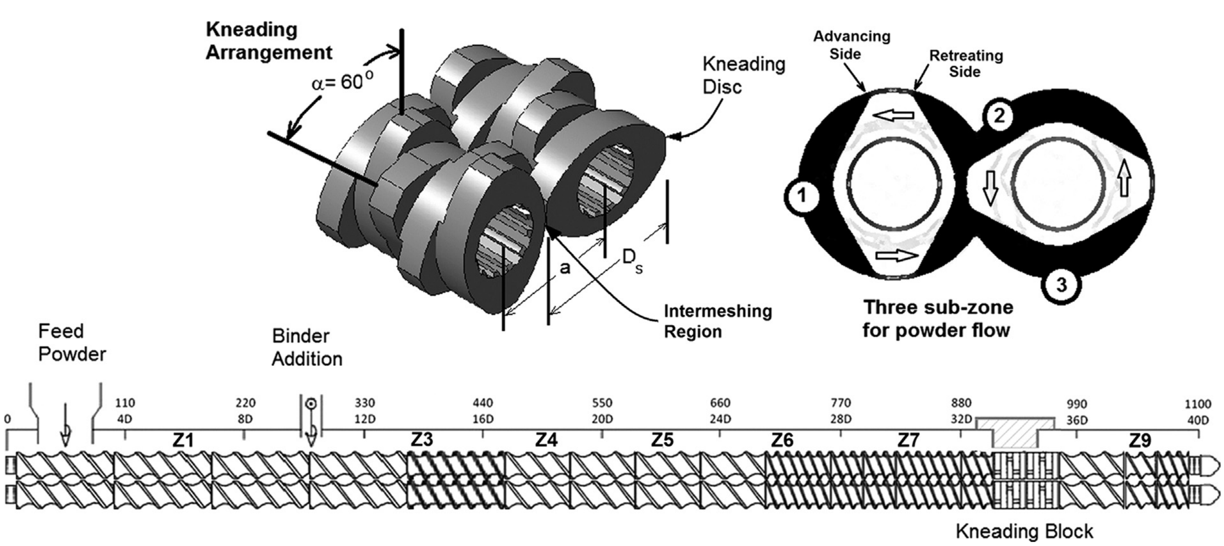

Fig. 1. Screw layout and magnified view of a kneading block with a 60° offset between kneading discs. Cross-sectional view of the kneading block highlights the three powder-carrying zones as the screws rotate.