Powder Technology 315 (2017) 290–299

Contents lists available at ScienceDirect

Powder Technology

journal homepage: www.elsevier.com/locate/powtec

Granule breakage in twin screw granulation: Effect of material properties and screw element geometry

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ARTICLE INFO

Article history:
Received 15 December 2016
Received in revised form 23 March 2017
Accepted 1 April 2017
Available online 6 April 2017

Keywords:
Twin screw granulation
Granule breakage
Conveying elements
Distributive mixing elements
Dynamic yield strength
Granulation rate processes

ABSTRACT

This study is the first to explicitly measure the influence of material dynamic yield strength (DYS) and screw element geometry on the breakage process in twin screw granulation. Granule breakage is the key mechanism for controlling granule size within the Twin Screw Granulator. Novel experiments which isolated breakage from other granulation rate processes were performed using conveying and distributive mixing element configurations and 2 and 3 mm cylindrical pellets of model materials (DYS from 0.5 to 137 kPa). Daughter size distributions and survivor pellet shape visualization was used to infer that the breakage mechanism in conveying elements (CE) is primarily edge chipping whereas in distributive mixing elements (DME), breakage is a combination of chipping and crushing. The maximum size of granule that could remain unbroken (3.49 mm for CE and 3.18 mm for DME) was determined by the largest available gap size in the element as measured by an analysis of the screw elements' open volume geometry. Below the maximum size, breakage probability varied inversely with granule strength up to 9 kPa. For granules stronger than 9 kPa DYS, breakage characteristics are independent of formulation properties and depend only on screw element geometry. This helps explain why twin screw granulation is more robust with respect to formulation changes compared to high shear wet granulation. Implications for using the results for both optimizing screw element design and calculating kinetic parameters for population balance modeling are discussed.

1. Introduction

Granulation of powders is a widely used industrial unit operation designed to agglomerate small particles into larger granules in order to improve flow properties, minimize dust hazards, and reduce segregation risks. The process is complex and, thus, has been the topic of many research investigations [1–4]. Of particular interest in this work is continuous wet granulation. Continuous processing of material has been demonstrated to decrease product variability, offer improved process control, and improve usage efficiency. Continuous wet granulation has been commonly performed using high shear granulation and the area has been studied in detail by several researchers [2,5–9].

The present work focuses on the operation of a twin-screw granulator (TSG), a novel method for continuous granulation that has several advantages over other continuous granulators. TSGs are suitable for large material throughput, can be easily customized, have short material residence times, and reduced footprint and capital costs when compared to other continuous granulators. Hence, there has been considerable interest in TSG operation in recent years. Little is known about the physics of wet granulation in a TSG and, thus, there is potential for optimizing the twin screw granulation process.

2. Background and objective

Twin screw granulation has been shown to provide better control over granule size and shape as compared to other granulation methods [10–12]. The differences between twin screw granulation and high shear wet granulation (HSWG), for example, are primarily due to (1) nucleation being separated, by design, from the other rate processes and (2) the short time scale of granulation in the TSG resulting in a different rate controlling mechanism in the granulator [13–16]. In order to understand what factors affect the attributes of granules produced using TSG, experiments designed to isolate the effects of particular rate processes are needed.

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http://dx.doi.org/10.1016/j.powtec.2017.04.011
0032-5910/

Several prior studies have focused on understanding the effect of screw element geometry on the performance of a TSG. It has been shown that a TSG operates in the mechanical dispersion regime with the breakage of wet mass being an important rate process for effective liquid distribution [17]. Breakage and liquid distribution in TSG is a strong function of the screw element geometry. For example, conveying elements (CEs), which are low shear transport elements, result in a bimodal granule size distribution due to poor liquid distribution [16,18,19]. Furthermore, the granule size distribution is observed to be more bimodal when the pitch of the screw elements is decreased [15]. In contrast, kneading elements exert significant shear on the wet mass and result in dense, elongated granules [13,20,21]. The offset angle between the kneading element discs and the number of kneading elements control the degree of shear within the kneading elements, which in turn affects granule size and porosity [22]. Distributive mixing elements (DMEs), also known as comb mixer elements, produce monomodal granule size distributions [14–16]. Breakage and layering have been shown to be the most important rate processes in this type of screw element. Since the screw element design is known to produce different granule properties, it is worthwhile to examine how these elements affect fundamental rate processes such as nucleation and wetting, coalescence and consolidation, and breakage [23]. In this work, we focus specifically on breakage.

Granule deformation and breakage primarily depends upon the dynamic yield strength (DYS) of the wet material, which in turn is a strong function of formulation properties such as primary particle size, binder viscosity, and binder surface tension [3,4,24]. The effects of formulation properties on breakage have been studied to some extent in the twin screw granulation literature. These studies have shown that increasing the binder concentration in the liquid phase results in a smaller mass fraction of fines and more uniform binder distribution throughout the different granule sieve cuts, especially in kneading elements [17,25]. However in conveying elements, increasing binder viscosity results in a more bimodal granule size distribution with larger mass fractions of fines and large granules [16,18,19]. The effect of changing powder binder wetting thermodynamics has been explored by using formulations with varying ratios of hydrophilic and hydrophobic powder components [26,27]. However, changing the concentration of the two powders also significantly changes the overall primary particle size distribution of the blend. This results in a convolution of the effects of the primary powder particle size as well as blend hydrophilicity on the overall granule properties. Differences in the primary particle size distribution of excipients have been shown to result in subtle differences between granule properties [17,28].

The effect of formulation properties on breakage has not been elucidated in the literature due to the use of multicomponent formulations and the fact that other rate processes occur simultaneously in the granulator. The effects of formulation properties on breakage have been studied in detail in HSWG [6,9,29], but similar studies have not been performed for a TSG. In this work, experiments have been designed to isolate granule breakage in order to study this key rate process.

3. Materials and methods

3.1. Maximum size analysis

The maximum size of granules in CEs and DMEs was determined using computer aided drafting (CAD) files of the screw elements (refer to the Supplementary information for the procedure used to obtain these CAD files). Using the CAD files, the largest diameter sphere that fits between the screw element and barrel was determined, as shown in Figs. 1 and 2.

3.2. Materials

Glass ballotini (Potters Industries LLC, OH, USA) of five different size cuts (0–10 μm, 63–90 μm, 125–180 μm, 180–250 μm, and 355–500 μm) were used as model material due to their spherical shape and well controlled properties (skeletal density = 2.47 g/cc) [3,6]. The particle size distributions of the glass ballotini were measured in a Malvern Mastersizer 2000 using water as the dispersing medium (refer to Fig. S1 and Table S1 in Supplementary information for details of the distributions). Silicone oil (Sigma-Aldrich Corp., MO, USA, viscosity = 64 Pa·s) and a glycerol solution (Sigma-Aldrich Corp., MO, USA, water-to-glycerol ratio = 0.01 by weight, viscosity 0.7 Pa·s) were used as model binders of different viscosities [3,6]. These model powders and liquids have been previously used in mechanistic studies of wet granulation [3,6]. The silicone oil binder was dyed yellow using oil soluble aniline dye (Woodworker's Supply Inc., NC, USA; dye powder-to-silicone oil ratio = 0.01 by weight) and the glycerol solution was dyed using Nigrosin dye (Sigma-Aldrich Corp., MO, USA). Play Doh modeling compound (Hasbro Inc., USA) was used to prepare spheres of different diameters to perform preliminary breakage specific experiments in the twin screw granulator.

3.3. Pellet preparation and dynamic yield strength measurements

To measure the dynamic yield strength (DYS) of the materials, cylindrical pellets with a height and diameter of 25 mm were prepared using a hand punch and die set. The glass ballotini and liquid binder were mixed together such that the binder-to-powder ratio by weight was 0.15. The solid fraction for these pellets was maintained at 0.63. For the 0–10 μm mixture with glycerol, the binder-to-powder ratio was 0.3 and the pellet solid fraction was maintained at 0.6. The pellet solid fractions were chosen to resemble those of granules produced by wet granulation of real powder blends. Cylindrical pellets with the same height and diameter were also prepared using Play Doh.

The DYS of each of the materials was measured using an Instron ElectroPlus E1000 material testing system with a platen impact speed of 10 mm/s. The strength of wet granules is a function of strain rate [3,30,31]. The typical particle impact speed observed in a TSG has been studied to some extent in the literature [18]. However, the results are not directly comparable to this work due to significant differences in

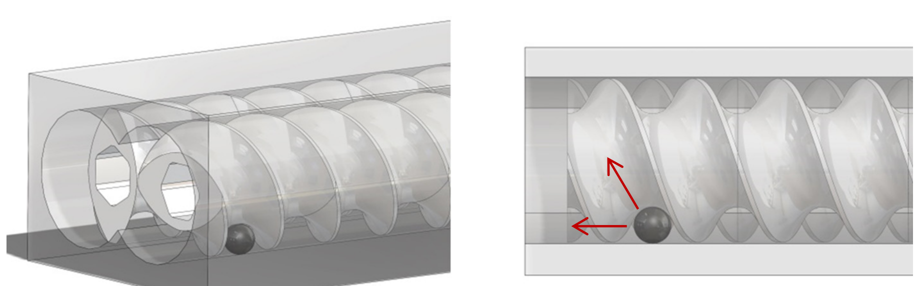

Fig. 1. CAD drawing of sphere of maximum size placed in a conveying element. Arrows indicate possible paths that a pellet can follow in the conveying elements.