



## Influence of zone formation on stability of continuous fluidized bed layering granulation with external product classification



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### ABSTRACT

Continuous fluidized bed layering granulation with external product classification and a sieve-mill cycle can show instability in the form of self-sustained nonlinear oscillations of the particle size distribution. In the present study, the stability and bifurcation analysis of this process is presented. The underlying process models explicitly account for compartmentalization of the fluidized bed into a granulation and a drying zone, which is an important feature of many technical processes. Implications for plant operations are discussed with the help of stability diagrams as a function of zone size, residence time within different zones, the addition of external seeds and particular properties of the sieve-mill cycle.

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### Introduction

Fluidized bed layering granulation is used for the production of high quality particles from liquid suspensions or solutions in the chemical, pharmaceutical, and food industries (Kunii & Levenspiel, 1991; Mörl, Heinrich, & Peglow, 2007). During large scale applications, these systems are operated as continuous processes with throughputs of up to several tons per hour. A characteristic flow-sheet is shown in Fig. 1. Typical systems employ a granulation chamber, where particles are fluidized by heated gas. Briefly, a solution or suspension is sprayed into the chamber by a nozzle. Once inside the chamber, the droplets collide with the particles, spread, and then interact with the heated gas, which causes the liquid to evaporate and the solid to remain on the particle surface, resulting in layer-wise growth in particle size. Particles are continuously discharged and classified into three fractions. The oversize fraction,  $\dot{n}_{\text{over}}$ , is ground in a mill and returned to the granulation chamber, which provides new nuclei,  $\dot{n}_{\text{mill}}$ , for granulation. In addition, external nuclei,  $\dot{n}_{\text{enuc}}$ , can be added. The intermediate fraction is

the desired product,  $\dot{n}_{\text{prod}}$ , which is removed from the process. The undersized fraction,  $\dot{n}_{\text{fine}}$ , is returned directly into the granulation chamber.

These processes are highly nonlinear and can give rise to instability in the form of self-sustained oscillations, most prominently in the particle size distribution (Heinrich, Peglow, Ihlow, Henneberg, & Mörl, 2002; Schütte, Ruhs, Pelgrims, Klasen, & Kaiser, 1988). Similar patterns of behavior have been observed for other particle processes, including granulation processes with internal nucleation (Vreman, van Lare, & Hounslow, 2009) and continuous crystallization processes (Randolph, 1980; Randolph & Larson, 1988). These oscillations result in varying product properties, which are usually not acceptable. Oscillations can be avoided by suitable selection of operating and design parameters in the stable regime, or by means of stabilizing feedback control applied in the unstable regime (Bück, Palis, & Tsotsas, 2015; Chiu & Christofides, 1999; Christofides, 2002; Palis & Kienle, 2012, 2014; Vollmer & Raisch, 2001, 2002). The first strategy requires reliable prediction of parameter combinations leading to instability.

Radichkov et al. (2006) generated a preliminary model based analysis of this cyclic behavior for the above process based on a simple model assuming a homogeneous granulation zone within the granulation chamber. This model assumes that the injected liquid is equally distributed on all particles, giving rise to a uniform growth that is proportional to the overall available particle surface

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**Notation**

$G$	growth rate (m/s)
$K$	drain (1/s)
$L$	particle diameter (m)
$m$	mass (kg)
$\dot{m}$	mass flow rate (kg/s)
$n$	number density (particle number/m)
$\dot{n}$	distributed particle flow rate (particle number/(m s))
$N$	total flow rate of particles (total particle number/s)
$q$	normalized number density (particle number/(m·total number of particles))
$t$	time (s)
$T$	separation function

**Greek Letters**

$\alpha$	relative size of granulation zone
$\mu_i(x)$	$= \int_0^\infty L^i x(L) dL$ ; $i$ -th order moment of argument $x = \{q, n, \dot{n}\}$ ( $m^{i+1}$ -dimension of argument $x$ )
$\sigma$	variance
$\rho$	particle density (kg/m <sup>3</sup> )
$\tau$	residence time (s)

**Sub- and Superscripts**

1	granulation zone
2	drying zone
enuc	external nuclei
fine	undersize fraction
in	inlet of granulation chamber
mill	mill
out	outlet of granulation chamber
over	oversize fraction
prod	product fraction
sc1	coarse screen
sc2	fine screen

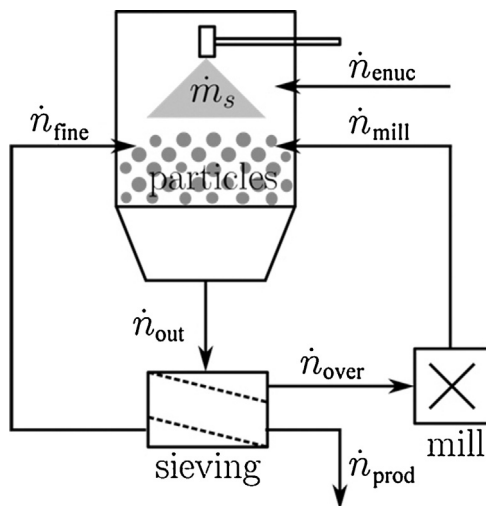


Fig. 1. Fluidized bed spray granulation with external product classification.

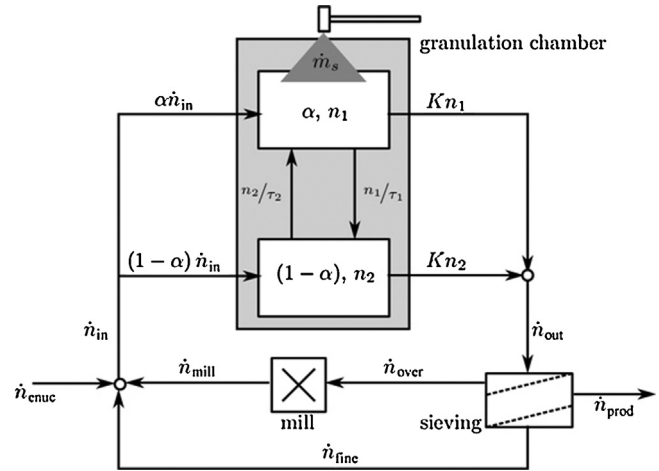


Fig. 2. Model configuration and nomenclature.

experimental investigations have shown that a significant change in the shape of the particle size distribution can actually occur, and the particles do not grow uniformly (Börner, Peglow, & Tsotsas, 2011; Grünewald, Westhoff, & Kind, 2010; Hoffmann, Peglow, & Tsotsas, 2011; Li, Kessel, Grünewald, & Kind, 2013; Zank, Kind, & Schlünder, 2001). This is attributed to only a portion of the particles being wetted by the injected solution owing to the position of the nozzle and the construction of the granulation chamber, and therefore the fluidized bed is separated into a spraying or granulation zone and a drying zone (Heine et al., 2013; Maronga & Wnukowski, 1997; Sherony, 1981; Wnukowski & Setterwall, 1989). Turchiuli, Jimenez, and Dumoulin (2011) detected these zones by measuring the temperature profiles in a fluidized bed. Additionally, Hoffmann et al. (2011) were able to explain length dependent growth observed in their batch layering experiments using a two-zone population balance model. They then related the change in shape, which was quantified by the increase or decrease in standard variation of the size distribution, to the size of the zones and the particle exchange rates between them while keeping the model assumption of size-independent growth in the spray zone.

In the present study, an extended stability and bifurcation analysis of continuous fluidized bed layering granulation processes is presented by means of a two-zone model. Implications for plant operation are then discussed with the aid of stability diagrams as a function of zone size, residence time within the different zones, addition of external seeds, and particular properties of the sieve-mill cycle.

**Mathematical model**

To study the influence of zone formation on the dynamics and stability of continuous fluidized bed layering granulation with external product classification, a two zone model as introduced by Hampel, Bück, Peglow, and Tsotsas (2013) for a Wurster coating process with internal product classification was employed. The model structure is illustrated in Fig. 2. The granulation chamber in Fig. 2 consists of a granulation zone (index 1) with relative size  $\alpha$  and a drying zone (index 2) with relative size  $(1 - \alpha)$ . It is assumed that both zones are well mixed. Solid material is injected with flow rate  $\dot{m}_s$  into the granulation zone.

Exchange rates between the granulation and the drying zones were described with characteristic residence times,  $\tau_1$ , and  $\tau_2$ , which are related to each other according to Hampel et al. (2013):

$$\frac{1}{\tau_1} = \frac{1}{\tau_2} \frac{1 - \alpha}{\alpha}, \quad (1)$$

according to Mörl et al. (Mörl, Mittelstraß, & Sachse, 1977; Mörl et al., 2007). Based on the assumptions of this model, which is also used by Bertin, Cotabarren, Buccala, & Pina (2011) and Vreman et al. (2009), the shape of the initial particle size distribution is conserved over the process (i.e., no narrowing or widening of the particle size distribution can occur in the model). However, recent

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