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Experimental investigation of process stability of continuous spray fluidized bed layering with external product separation

M. Schmidt*, C. Rieck, A. Bück, E. Tsotsas

NaWiTec, Thermal Process Engineering, Otto von Guericke University Magdeburg, Universitätsplatz 2, 39106 Magdeburg, Germany

HIGHLIGHTS

- Experimental investigation of dynamics in continuous layering with external classification.
- Proof of oscillations in particle size distribution (stable limit cycle).
- Similar periodic behavior for significant variation in mean diameter of milled particles.
- Milling behavior determined as key process influence.

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ABSTRACT

The present work investigates experimentally continuous spray fluidized bed layering with external product separation. In this process configuration, growing particles are continuously withdrawn and classified externally by two sieves into product fraction, undersize and oversize fraction. The oversize fraction is milled and fed back to the fluidized bed alongside with the undersized fraction. New seed particles for the layering process are provided by recycling of milled particles and agglomeration of dust particles, formed by dried droplets (overspray) and attrition. Until now, this process configuration has been investigated by simulations only. For certain parameter regimes, these models predict oscillations of the particle size distribution. Based on these predictions, even theoretical controllers have already been developed to diminish these oscillations. An experimental investigation of these oscillations of the particle size has not yet been published. Our work presents a systematic study of the process and proves the existence of oscillations of the particle size distribution in the fluidized bed.

1. Introduction

Spray fluidized bed granulation is commonly used in chemical, pharmaceutical and food industries. Solid-containing liquids like solutions, suspensions or melts are transferred to the solid state by spraying into a fluidized bed of particles (for example Kunii and Levenspiel, 1991; Mörl et al., 2007). In most cases, this phase transition involves growth of the particles. Three different growth mechanisms can be distinguished. If the sprayed liquid acts as a binder, and clusters of initial particles are formed, the process is called spray fluidized bed agglomeration. This mechanism is used, for example, to agglomerate food powders (Avilés-Avilés et al., 2015; Sanders et al., 2003), to agglomerate laundry powder (Boerefijn and Hounslow, 2005) or to produce pellets for a controlled release of pharmaceuticals (Pauli-Bruns et al., 2010). The second mechanism is called spray fluidized bed coating. There, a different material is sprayed onto carrier particles, forming a layer on the surface of the particles. This process is used, for example, to ensure a controlled release rate of pharmaceutical ingredients (Kulah and Kaya, 2011) or for encapsulation of anti-oxidants (Coronel-Aguilera and San Martín-González, 2015). If the sprayed liquid contains the same material as the initial particles, the process is called spray fluidized layering. This mechanism can be used, for example, to produce large fertilizer granules from small initial crystals (Wang et al., 2013). By right choice of parameters, the process can be guided towards the desired granulation mechanism (for example Ennis et al., 1991; Iveson et al., 2001). Following, layering growth is regarded to be the governing mechanism, and thus, the present work will focus on layering growth only. Size reduction processes, such as attrition or breakage, are considered to be either negligible or in equilibrium with aggregation processes. On a much smaller size scale,

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* Corresponding author. Tel.: +49 391 67 12 319.
E-mail address: martin.schmidt@ovgu.de (M. Schmidt).

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nucleation takes place, which describes the formation of new granulation seeds through wetting and aggregating of small dust particles in the process (Iveson et al., 2001).

To ensure high product throughputs, spray fluidized bed layering processes are operated in continuous mode. Processes for continuous particle formation often show highly dynamic behavior: continuous crystallization processes with classifying product removal are one example in a solid/liquid environment (Lei et al., 1971; Motz et al., 2003; Vollmer and Raisch, 2002). There, small crystals are dissolved and re-fed as supersaturated solution to increase the size of the product crystals. This removal of fines and refeeding to the crystallization vessel may induce self-sustained oscillations of the crystal size distribution. Also, a configuration, in which the largest crystals are crushed by an ultrasonic attenuator and re-fed to the crystallizer, shows oscillatory behavior (Mangold et al., 2015). In most cases, these oscillations become visible in a plot of mean diameter over process time, featuring a certain period time and amplitude. Interaction of classifying withdrawal of single fractions, nucleation in the vessel, continuous crystal growth and recycling causes this oscillating behavior. Similar dynamic behavior is expected for the gas/solid/liquid system of continuous spray fluidized bed layering with classifying product removal. Here, nucleation (seed particle formation), particle growth, classifying product removal and recycling also take place. Thus, the occurrence of oscillatory process behavior is to be expected. Problems linked to oscillatory particle size distribution (PSD) are varying particle sizes and oscillating bed mass, bed height and product mass flow rate. The discharged particles can be separated internally through a classifying tube, which has been extensively investigated experimentally in previous work (Schmidt et al., 2015). The process setup with internal separation showed highly oscillating process behavior for certain discharge and drying parameters. Another configuration is external classification. Here, the particles are discharged from the bed and classified externally by two sieves. The oversized particles are crushed by a mill and re-fed to the granulator together with the undersized particles. The product fraction is withdrawn. This setup yields highly dynamic process behavior due to complex interaction of seed particle formation, particle growth, removal and recycle, as was shown in simulations and bifurcation analysis, for example, by Heinrich et al. (2002), Radichkov et al. (2006) and Dreyschultze et al. (in press), predicting oscillations of the PSD for certain mean diameters of milled particles. Simulated temporal evolutions of the normalized PSD density q_3 showing oscillatory and stable process behavior are shown in Fig. 1. The mean diameter of milled particles is identified as key process parameter in these works to influence process stability. Besides, changing fluidization and drying conditions can also influence process stability (Schmidt et al., 2015), changing also product properties like particle structure and consequently particle size (Rieck et al., 2015). In most cases, constant product properties are desired, leaving the mean diameter of milled particles as a free parameter to influence process behavior.

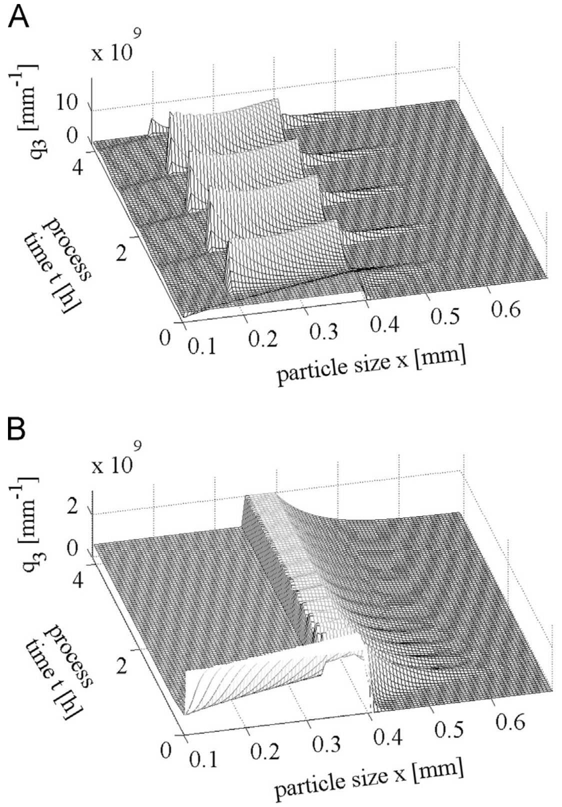

Fig. 1. Simulated temporal evolution of the normalized particle size distribution density q_3 for a continuous layering process with different mean diameters of milled particles, showing A: a process with oscillating PSD and B: a process with stable PSD.

So far, the only documented case of oscillations of PSD in practice is shown in Fig. 2. There, the mean particle diameter (there: d_{50}) and the standard deviation of the PSD (there: s) are pictured over time. Schütte et al. (1998) presented their data in a patent without further interpretation and without explanations regarding reproducibility. Besides Heinrich et al. (2002), Radichkov et al. (2006) and Dreyschultze et al. (in press), a wide range of authors deal with these oscillations of PSD in simulations (for example Bück et al., 2015; Cotabarren et al., 2010; Dosta et al., 2010; Drechsler et al., 2005; Heinrich et al., 2003; Palis and Kienle, 2012; Palis and Kienle, 2014) or use them to motivate the need of process control or better models (for example Bertin et al., 2013; Cotabarren et al., 2011). But none of these references includes an experimental proof of the occurrence of PSD oscillations.

On the other hand, industrial processes conducted are usually run with high recycle rates of product or temporary product buffering, retarding product throughput and reducing capacity of the plant, to realize stable operation. A better understanding of the existence and regime of occurrence of oscillations and possible control would therefore enhance plant capacity and product throughput.

In this work, we present a methodical study, proving the existence of PSD oscillations experimentally and reproducibly, providing the missing link between theoretical studies, simulations and process control schemes for continuous spray fluidized bed layering with external product separation. We present a series of four experiments with varying rotational speed of the mill, crushing oversized particles, discuss the influence of this parameter variation and compare the experimental results to expectations from simulations already existing.

2. Process description

In spray fluidized bed layering processes, solid-containing liquids are sprayed on fluidized particles in a granulator. A schematic view of this setup is shown in Fig. 3A. The layering mechanism is shown in more detail in Fig. 4. The spray droplets spread on the surface of a particle and form, due to drying, a solid layer around the initial particle. Repetition of this mechanism results in an onion-like granule. The evaporated spray liquid is carried out by the fluidizing gas, and the remaining solid increases the bed mass. For a continuous process, also some way of product discharge has to be implemented. In this case, bed material was