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Experimental spray zone characterization in top-spray fluidized bed granulation

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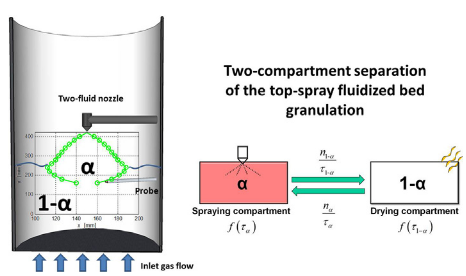
HIGHLIGHTS

- Flat fluidized beds have a shifted regime behavior compared to cylindrical beds.
- Flat fluidized beds show general applicability for the study of solids motion.
- Demarcation of a spray zone and a drying zone by droplet detection.
- Measured mass fractions + particle residence times for population balance modelling.
- Transfer of parameter from the flat fluidized bed to cylindrical beds.

GRAPHICAL ABSTRACT



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ABSTRACT

In fluidized bed granulation wetting and coating of particles depends on the atomization of a liquid binder agent. Droplet distribution and the subsequent micro-scale processes of droplet deposition on particle surfaces as well as drying of layers and liquid bonds between aggregated particles lead to a subdivision of the process space into two major compartments, a spray zone and a drying zone. By using a self-constructed, simple conductivity probe spray patterns inside the fluidized bed are located. The spatial demarcation of the compartments, which is dependent on the fluidization and spray conditions, is deduced. Particularly, nozzle height and nozzle gas flow rates influence the expansion of the spray zone and its intrusion into the bed. The presented results show by means of the particle residence time for the two considered compartments that an increased nozzle mass flow rate leads to significantly accelerated particle flow in the spray zone, and the fluidization velocity of the gas forces a faster particle re-circulation behavior in the entire fluidized bed. Consequently, process time for wetting and drying is reduced. By using a flat fluidized bed with rectangular cross section in combination with image-based acquisition techniques, particle velocities and solid volume fractions have been acquired. Comparing the results of particle circulation patterns with data obtained in cylindrical equipment shows that information can be transferred from the quasi-2D configuration to real 3D geometries.

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1. Introduction

Fluidized bed technology is frequently applied to the large-scale production of granular materials due to its versatility and potential to conduct granulation or agglomeration at low cost. The resulting coated or aggregated powder particles have added-value with advantages in transportation, solubility, protection or controlled release of active ingredients. In industrial applications different fluidized bed technologies are used (e.g. top-spray, bottom-spray, spouted bed) to attain desired granular properties for different products. Particularly for granulation purposes in pharmaceutical production the top-spray fluidized bed is often applied. A better process understanding is essential to improve product quality in manufacturing.

The optimal adjustment of liquid binder atomization into the fluidized bed system plays a critical role in achieving a stable and successful granulation process. Wet-quenching of the particle bed causing a total process breakdown or a spray which does not properly reach the fluidized particles are extreme cases that must be avoided. In order to elaborate the influence of a two-fluid nozzle during fluidization several experimental trials have been conducted, using two different fluidized bed designs. The first one is a cylindrically shaped equipment to capture spray characteristics, like spray expansion or droplet appearance under different fluidization and spray conditions. The acquisition of spray properties has been performed under exploitation of the electrical conductivity of water using a conductivity probe. The technique yields the dimensions of a spray zone in which active binder droplets appear. Consequently in a two-compartment approach, the drying zone can be defined as the part of the process chamber which is not occupied by the spray zone. Such a compartment division of the granulation process is an expedient tool in population balance modeling to improve results and accuracy of model predictions (Hoffmann et al., 2011; Li et al., 2012). The compartment description of fluidized bed granulation goes back to Sherony (1981), who described particle exchange rates and residence times in two considered compartments by a stochastic model of surface renewal. The two compartments were regarded as perfect mixers by Wnukowski (1989) irrespective of spatial process dynamics. Later, a third zone was introduced (Maronga and Wnukowski, 1997), which was a non-active domain with regards to granulation.

Besides the previously described and here applied detection of liquid, another way to identify process specific compartments is based on temperature gradients in the bed (Turchiuli et al., 2011). Three domains are usually defined by temperature measurements: wetting-active zone, isothermal zone and heat transfer zone. However, this process separation takes drying of droplets and wet particle surfaces into account. Such measurements do not provide a clear demarcation between spraying and drying. A further extension to four zones was proposed by Maronga and Wnukowski (1998), applying additional humidity measurements together with the record of temperature profiles. In this approach the drying zone was subdivided into an active and a non-active zone.

In order to capture particle dynamics within the two here considered compartments, a second fluidized bed design has been applied. This fluidized bed is flat with a rectangular shape and transparent walls in order to ensure optical access to the otherwise opaque interior of the multiphase flow. In this way, two-dimensional imaging can be used for tracking of the circulatory motion of particles. Such flat fluidized beds have frequently been used since the early 1990s to investigate system properties, for example, to characterize ascending gas bubbles (Laverman et al., 2007; Busciglio et al., 2008) and particle segregation effects (Goldschmidt et al., 2004; van Bokkers et al., 2004) or to validate numerical simulations (Asegehegn et al., 2011; Schreiber et al., 2011; Taghipour et al., 2005; Link et al., 2005, 2004; Zhao et al., 2008; van Buijtenen et al., 2011b). Particle Image Velocimetry (PIV) or Digital Image Analysis (DIA) is preferred measurement methods for this type of fluidized bed design (Agarwal et al., 1997; van Buijtenen et al., 2011a; Hernández-Jiménez et al. 2011; Shen et al., 2004).

Goal of this study is to quantify the spatial demarcation into a spray and a drying zone for a two-compartment approach of top-spray fluidized bed granulation. The compartment dimensions depend on particle dynamics and droplet appearance. This is a novelty compared to Boerner et al. (2012), where the spray zone was defined in the space between and below the riser draft plates for the case of Wurster fluidized bed granulation.

The experimental investigations comprise the variation of essential process parameters, in particular, nozzle gas flow rate, nozzle height and intensity of fluidization. The results obtained at the flat, rectangular fluidized bed reveal particle-scale features like particle residence times inside the compartments, particle re-circulation behavior or solid distributions in the entire bed.

Furthermore, results from the flat fluidized bed are to be compared with results obtained in the cylindrical bed, in order to dispel doubts concerning applicability of the former. Therefore, both systems have a similar basic geometry, meaning that the width of the flat fluidized bed is equal to the diameter of the cylindrical bed. Measurements of solid volume fraction inside the entire bed and pressure drop analysis have to be conducted, and the results shall be compared to show similarities and differences in flow properties.

2. Experimental methods

The experimental investigations have been performed using two different fluidized bed systems. In order to investigate the spray pattern for compartment demarcation a cylindrical fluidized bed has been used, schematically shown in Fig. 1. The simplified perspex construction was supplied with air at an inlet temperature of 30 °C. A two-fluid nozzle in top-spray mode has been adjusted in different heights, spraying pure water into the system. The Schlick 970-S8 nozzle operates at a maximum spray pressure of 2 bar. A gas flow rate of 3.6 kg/h passed the nozzle at this pressure. The amount of water was adjusted at the nozzle by a micrometer screw keeping the water throughput constant at 3.8 ml/min for the different gas throughputs. The spray patterns were measured

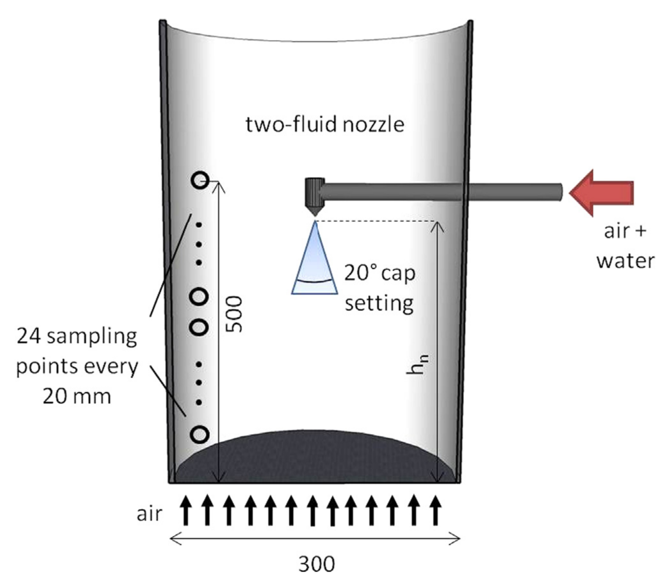

Fig. 1. Illustration of cylindrical fluidized bed setup; all dimensions in mm.