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Influence of drying conditions on layer porosity in fluidized bed spray granulation

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ABSTRACT

Particle coating experiments were performed in a lab-scale fluidized bed with varying process parameters, such as spraying rate and air inlet temperature, leading to different drying conditions. Porous (γ-Al_2O_3) and non-porous (glass) initial particles were sprayed with a sodium benzoate solution. For each experiment, the particle size distribution as well as the layer porosity was measured. The results show a dependency of the layer porosity on the drying conditions, represented by the drying potential of the fluidization gas. The obtained relationship is expressed as a linear correlation, which can be used in process models. Apart from the experimental results, a model based on population balances and heat and mass balances is presented. Simulations performed using the obtained empirical correlation are in good agreement with experimental data.

1. Introduction

Particle formulation in fluidized beds includes several sub-processes, such as agglomeration, layering granulation, and coating, which are widely investigated in the literature, e.g. Heinrich et al. [1], Hemati et al. [2], Hede et al. [3], Terrazas-Velarde et al. [4], Dernedde et al. [5], and Li et al. [6]. In these processes, a liquid, containing solid material, is sprayed into a fluidized bed. In the case of agglomeration, the particles collide randomly and stick together by forming liquid bridges at wet spots created by deposited droplets. Due to evaporation, solidified bridges are created. The resulting agglomerates consequently consist of several primary particles. In the case of layering granulation and coating, the dried droplets form a solid shell around the initial particle. Ideally, no agglomerates are formed. The material of the initial particle and the added solid material are the same in granulation, while, in coating, the materials are different. Additionally, a coating layer is relatively thin compared to the particle size, see e.g. Bück et al. [7].

In this work, fluidized bed coating will be treated. This process is widely used in the industry to produce pharmaceuticals, cosmetics, fertilizers, or foods. Applications may be the coating of pharmaceuticals and fertilizers to control the duration and the point of release of active substances, see, e.g. Turton [8], taste and odor masking purposes and obtaining a certain shape or surface structure, e.g. Guignon et al. [9]. In the food industry, coating can be used to separate ingredients from their environment (water, acid, oxygen), if it is detrimental to the uncoated material, see, e.g. Werner et al. [10].

An important product property is the particle size distribution, which influences, e.g., the flow behavior and consequently the handling of the product. If the aim is to shield an active ingredient with a coating layer, the size of the product particle also affects the performance of the product, since the dissolution characteristics depend on the thickness of the coating layer. Additionally, the morphology of the coating (surface structure, porosity) will alter the dissolution behavior and therefore the release times of active substances shielded by the coating. The coating morphology has been investigated in the literature so far only in terms of the surface structure. Dewettinck et al. [11] investigated the agglomeration tendency during fluidized bed coating and performed coating experiments with different materials. They used glass beads as core material and locust bean gum, sodium alginate, carboxylmethycellulose, and κ-carrageenan as coating material. They also investigated the coating morphology by scanning electron microscope images and found differences in the surface structure. In their case, coatings consisting of locust bean gum and κ-carrageenan appeared to be very smooth and non-porous, whereas coatings consisting of the other materials showed imperfections and surface roughness. Tzika et al. [12] investigated the influence of process variables (e.g. spraying rate and fluidization air velocity) on the morphology and quality of a latex coating of fertilizer seeds in a Wurster fluidized bed. The coated particles were examined by scanning electron microscope images and the release rates of the nutrients were measured. By changing the spraying rate, the drying conditions in the fluidized bed were also varied automatically. The results show an influence of the spraying rate on the surface structure of the

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coating layer and also on the release rates. Hede et al. [13] also analyzed the coating morphology for scale-up purposes via scanning electron microscope images. The coating layer porosity was not part of the studies in those publications.

As stated by Tsotsas [14], it is conventionally assumed that the coating layer is compact. In fact, it is possible to produce compact layers, but they can also be porous depending on the drying conditions, namely the liquid flow rate and the gas temperature, in the fluidized bed. In the case of a porous coating layer, particles grow faster than particles with a compact layer, since a larger part of the added volume is void. This means basically that the drying conditions influence the growth kinetics of the particles, which has not been considered in modeling yet.

In publications addressing the modeling of coating and layering processes, e.g., Heinrich et al. [1], Ronsse et al. [15], Hede et al. [3] and Silva et al. [16], the enhancement of growth due to a developing porosity has not been taken into account. There are publications, e.g. Ronsse et al. [15] and Hede et al. [3], where the heat and mass transfer is modeled along with the particle growth. But these models are coupled in only one direction, i.e. particle growth influences the drying conditions but not vice versa.

This work deals with the determination of the coating layer porosity and its dependency on the drying conditions in fluidized bed spray granulation. Therefore we use the following approach:

1. determination of a semi-empirical relationship between the drying conditions and the coating layer porosity based on fluidized bed coating experiments and micro-tomography measurements;
2. development of a model based on population balance equations for the particle growth considering the coating layer porosity;
3. comparison of calculated and measured particle size distributions and evaluation of the model.

2. Experimental work

2.1. Laboratory plant

The fluidized bed coating experiments in this study were performed in a laboratory-scale plant from the Glatt company (type GPCG 1.1), which is shown in Fig. 1. In this experimental plant, only batch processes can be realized. The fluidized bed chamber is cylindrical and has a diameter of 150 mm and a height of 450 mm. The gas distributor plate, having a mean pore size of 100 μm, is made of sintered metal to achieve uniform fluidization conditions.

Sodium benzoate was used as coating material in all experiments. It is sprayed in the form of an aqueous solution into the process chamber with a two-fluid nozzle (Schlick, type 970/0 S4), which can be placed in top-spray or in bottom-spray position. The liquid is dosed by a precision metering pump (Labortechnik Sewald, type: LDP-31) and the amount of injected liquid is detected by weighing of the supply tank. The plant operates with compressed air, which is taken from the local compressed air network. In this way, constant values for the mass flow rate of the fluidization gas and the inlet moisture content can be obtained. The measurement of the inlet and outlet gas moisture content is realized by means of infrared spectroscopy (Rosemount Analytical, type NGA 2000). To ensure constant measurement conditions, several control valves, which guarantee constant air pressure and air flow, are installed. The mass flow of fluidization gas is measured by a mass flow meter (Bronkhorst Mättig, type: F-112AC) and controlled by a throttle valve. Additionally, the gas temperatures at the inlet and outlet were measured. The electric heater allows a maximum temperature of 100 °C for the inlet gas. The plant has a built-in textile filter for the exhaust air, which has a mean pore size of 7 μm.

2.2. Experimental series

Two experimental series with different particle systems (porous and non-porous) were carried out to investigate the influence of different gas inlet temperatures and spraying rates on the particle growth and the layer porosity. Four experiments were performed in each series. All of them were run in batch mode with top-spray configuration in the fluidized bed spray granulator described.

In the first experimental series (experiments numbered from 1 to 4), glass beads with a mean particle diameter of 0.50 mm, a standard deviation of 0.058 mm and a particle density of 2400 kg/m^3 were used. This material has a high sphericity of 0.94, which is close to the model assumption of spherical particles. In addition, the glass beads are non-porous, so during the granulation no droplet imbibition occurs. In the first experimental series, four experiments were performed in which the gas inlet temperature (from 50 °C to 95 °C) and the spraying rate (500 g/h to 1100 g/h) were varied. The specific process parameters of each experiment are listed in Table 1. All other process parameters, such as initial bed mass (1 kg), the amount of sprayed solution (1 kg), mass flow rate of the gas (120 kg/h), and concentration (30 ma-%) of the injected solution are the same for all experiments of the series. The time of the experiments varied between 55 and 120 min, due to the spraying rate and fixed amount of sprayed material.

In the second experimental series (experiments numbered from 5 to 8), porous and hygroscopic γ-Al_2O_3 particles, having a mean diameter of 0.61 mm, a standard deviation of 0.016 mm, and a particle density of 1280 kg/m^3, were used. The sphericity of the particles is 0.97. The process parameters of the second series correspond exactly to those of the first series, except for the mass flow rate of the gas and the initial bed mass. Because of the smaller density of the γ-Al_2O_3 particles, these values have been adjusted to 75 kg/h and 0.5 kg to maintain a similar fluidization regime as in the first series.

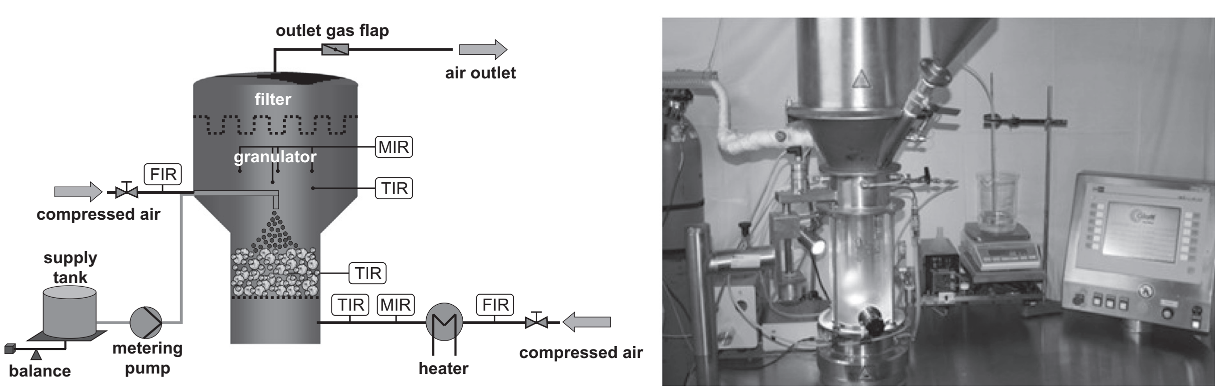

Fig. 1. Scheme and photo of the experimental plant used in this study.