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Coating of finely dispersed particles by two-fluid nozzle

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ABSTRACT

Particle coatings are used extensively to generate dispersed solids with well-defined properties, e.g., to protect active ingredients, with most coating processes using core particles of a diameter larger than 200 μm. This work contributes to the development of a coating process for fine dispersed particles (diameter less than 50 μm) by combining two particle-formulation processes, namely, coating and spray drying. The feasibility of the operation is based on and demonstrated by the innovative application of a two-fluid nozzle. Experiments were conducted by using glass particles as core particles and sodium benzoate as the coating agent. The coating of finely dispersed particles is achieved by the spraying of particles and coating solution as a homogeneous suspension. The aim is to create droplets with only one contained particle at the nozzle outlet. After evaporation of the water in the droplet, a thin solid film is built on the particle surface. The suspension viscosity was measured and compared with empirical equations from the literature. The liquid-film thickness on the particle surface was calculated to predict the building of a uniform coating layer or agglomerates. In this study, the feasibility of pneumatic transport through the nozzle and an investigation of the process were illustrated. The agglomeration fraction and degree of coating of the particle surface were analyzed optically by scanning electron microscopy. In this way, the influence of different processes and suspension parameters on the product quality were determined.

Introduction

The coating of fine dispersed particles is becoming increasingly important in the food and pharmaceutical industries, and in chemistry. Coating processes are motivated by the fact that different functional properties can be achieved, the release of active ingredients can be manipulated, adhesion and compatibility between various components of the composite materials can be improved.

A fluidized bed coating without severe agglomeration is typically limited to particle sizes that are appreciably larger than 100 μm (Dewettinck & Huyghebaert, 1998). In this case, however, fine particles are extremely difficult to fluidize because of their high cohesion (Geldart, 1973). In general, the fluidization of such powders leads to the formation of channels or ratholes. To overcome these processing issues, fine particles are granulated with larger, easily fluidized powders to enhance processing (Ehlers et al., 2009; Takano, Nishii, & Horio, 2003; Thiel & Nguyen, 1982; Shen, 1996; To & Davé, 2016). Such approaches can produce coated products, but they have the disadvantage for limited application ranges, because of a need for high concentration of well fluidized excipient, which is problematic for products that require high active-ingredient loadings.

Several methods exist for fluidized bed coatings or the agglomeration of fine cohesive particles without the addition of significant amounts of excipient materials. However, most methods require complicated and potentially expensive modifications to the typical fluidized-bed configuration. Kawaguchi et al. (2000) coated cohesive active-pharmaceutical-ingredient particles in a rotating fluidized bed and prepared reproducible acetaminophen granules (~500 μm). The production of this product in a conventional fluidized bed is difficult. Later, Watano et al. (2003, 2004) reported that 15-μm cornstarch particles could be granulated or coated individually in a rotating fluidized bed. In this case, the centrifugal forces exerted onto normally cohesive cornstarch particles increased the apparent particle weight and made them behave like well fluidized particles.

In other studies, Hamashita et al. (Hamashita, Nakagawa, Aketo, & Watano, 2007; Hamashita et al., 2008, 2009) presented that micronized core particles that contain ibuprofen could be granulated using an impeller-agitated fluidized bed. Miyadai, Higashi, Moribe, and Yamamoto (2012) used a similar apparatus to produce microgranules of ibuprofen particles. These methods could produce agglomerates with high loadings of active materials reproducibly. Ichikawa and Fukumori showed in a series of studies that micronized and cohesive powders could be coated and agglom-

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Nomenclature

Latin symbols

A	Surface area (m^2)
d	Diameter (m)
d_{32}	Sauter mean diameter (m)
F	Force (kg m/s^2)
h	Calculated liquid-film thickness (m)
M	Mass (kg)
$\dot{M}$	Mass flow rate (kg/s)
q	Normalized density distribution (m^{-1})
Q	Normalized cumulative distribution
t	Time (s)
T	Temperature (°C)
V	Volume (m3)
X	Loss of product/solid (%)

Greek symbols

α	Particle fraction in suspension
η	Viscosity (Pa s)
μ	Total moment (m^i)
ρ	Density (kg/m^3)
σ^2	Variation (m^2)
ϕ	Solute mass fraction in coating solution

Subscripts

1	Single particle
ad	Adhesion
calc	Calculated
CS	Coating solution
dr	Droplet
dry	After drying in oven
exp	Experimental
g	Gas
gr	Gravitational
inlet	Inlet gas
M	Mass fraction
max	Maximum
N	Nozzle
p	Particle
s	Solid
V	Volume fraction

erated in a bottom-spray Wurster-type spouted bed (Ichikawa, Shin, & Kang, 1999; Jono, Ichikawa, Miyamoto, & Fukumori, 2000; Fukumori et al., 1991). The particles could not be fluidized in a strict sense; hence, high superficial velocities were required to make them spout and a draft tube was used to enhance their circulation.

Watano et al. (2003, 2004) compared cohesive forces to other forces such as buoyancy or a resultant drag force, indicating that to counteract cohesion one may need to increase the body weight of the powders. A cohesion reduction by introducing flow additives to fine powders could be used to promote fluidization as was shown in US patent 6,833,185 (Zhu & Zhang, 2004).

Yang, Sliva, Banerjee, Dave, and Pfeffer (2005) showed that surface modification through dry coating with nanosized flow additives is a simple and practical method to provide more significant and reliable results compared with additive blending. Cohesive powders were made to flow and be potentially fluidizable. In dry coating, a discrete, fairly uniform layer of nanosilica particles is applied onto the surface of the cohesive host particles. Yang et al. derived a simple equation that showed that a reduction in cohesion because of the introduction of nanosized surface asperities as a nanosilica coating is inversely proportional to the particle size, and hence, the coating of nanosilica would improve the flow properties.

Chen et al. (Chen, Jallo, Quintanilla, & Dave, 2010; Chen, Yang, Dave, & Pfeffer, 2008; Chen, Yang, Mujumdar, & Dave, 2009) showed in a few studies that surface asperities, the level of coating, and the surface energy play a major role in cohesion reduction. The addition of a nanosilica surface coating can reduce the granular bond number (the ratio of cohesive forces to inertial forces) by more than an order of magnitude for aluminum particles below 5 μm. This cohesion reduction can allow for fine cohesive particles to be fluidized in a conventional fluidized bed and has led to a fluidized-bed-coated product without significant agglomeration.

Work on the dry coating of drug powders indicates that their cohesion can be reduced, albeit less significantly than the cohesion of materials such as cornstarch via dry coating with silica; however, their fluidization behavior has not been investigated (Jallo, Ghoroi, Gurumurthy, Patel, & Dave, 2012).

Recent work of To and Davé (2016) shows that silica nanoparticles can be dry coated onto the surface of as-received and micronized ibuprofen particles with a median size of 41–74 μm and a corresponding Sauter mean diameter of 21–41 μm. Ibuprofen was selected as a model drug because of its poor flowability and hence poor fluidizability. Dry coating with nanosilica particles allows previously cohesive particles to be fluidized-bed coated with minimal agglomeration.

The main objective of this contribution is to investigate the application of an innovative process for the coating of fine dispersed particles (d_p < 50 μm). The newly developed method combines the advantages of two particle formulation processes, namely, spray drying and coating.

Spray-drying processes are applied widely in numerous industrial fields, particularly in dairy and food production. They produce a dry powder by the rapid evaporation of water from droplets that are atomized in a stream of hot air. The main advantage of this method is the short residence time of particles inside the drying chamber. Thus, spray drying is a suitable drying technique for thermally sensitive products (Tran, Jaskulski, & Tsotsas, 2016). All spray dryers use some type of atomizer or spray nozzle to disperse liquid into a controlled-drop-size spray. Most common are rotary disks and single or multiple fluid pressure nozzles. Depending on the process needs, drop sizes from 10 to 500 μm can be achieved with appropriate choices (Kröll, 1978). In wet coating, particles are contacted with droplets from the liquid, which deposit on the particle surface. After the evaporation of water from the droplets, a coating layer is built. The liquid can be a solution or a suspension (Hampel, 2015). The most-used equipment for wet coating includes the pan coater, drum coater, and the fluidized-bed coater (Hampel, 2015).

In this study, the change in suspension viscosity with particle mass and solute was determined experimentally and calculated. Experimental studies were conducted with a variation in operating parameters, such as nozzle pressure, mass fraction of core particles in suspension, and mass fraction of solute in the coating solution.

The aim in this study was to establish the influence of process and suspension parameters on the individual processes, namely, coating (partial or complete), agglomeration, and spray drying. It is desirable to maintain a maximal amount of completely coated particles, with a minimal amount of agglomerated particles and spray-dried droplets. For this reason, dependency of the formation of agglomerated particles on process parameters and properties of the suspension was analyzed.

An outline of this study is as follows. The general principles of the proposed process are discussed. The suspension viscosity was measured and the dependency of the particle and coating solute mass fraction were determined and the results were compared. An experimental plant and the feasibility of the coating process in this