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Original Research Paper

# Shell porosity in spray fluidized bed coating with suspensions

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ABSTRACT

An experimental study regarding spray fluidized bed coating with aqueous suspensions is presented. The dependency of coating shell morphology on drying parameters, atomization pressure and composition of suspension is investigated. The results are compared to existing work regarding spray fluidized bed coating with aqueous solutions of crystalline material. Contrary to coating with solutions, coating shell smoothness and porosity does not depend on drying conditions. Nevertheless, atomizing pressure and mass fraction of solids in suspension have large influence on coating shell morphology. High atomization pressures, leading to small droplets, result in smooth coating surfaces and low shell porosities. A similar trend is observed for a low mass fraction of solids in the suspension.



## 1. Introduction

Spray fluidized bed coating is a technology used in particle formulation. Core particles are sprayed onto with a solid-containing liquid, which dries on the particles, creating a coating shell. This technique is applied in food, pharmaceutical and chemical industries for encapsulation (e.g. protection from degradation or oxidation) [1,2], for improvement of flow properties, i.e. reducing dustiness, stickiness and attrition, for controlled release of drugs [3] or fertilizers [4,5] or to mask unpleasant tastes or odors. In most applications, the coating layer has to cover the whole particle surface uniformly and the morphology has to fulfill additional specifications. For improvement of the flow properties, coating must be firm, durable, non-friable, and often smooth to reduce interparticle forces. For the controlled release of drugs or fertilizers solubility or porosity of the coating layer are the governing product quality parameters, e.g. high porosities lead to high release rates [6]. Thus, controlling the porosity of coating layers is necessary, especially for tablets containing drugs and for fertilizer granules.

In spray fluidized bed coating with aqueous solutions the morphology of coating layers is strongly influenced by process conditions. Ebey [7] first derived the so-called Environmental Equivalency (*EE*) model to describe all psychrometric process variables with one parameter. For a constant value of *EE*, two separate experiments with different process conditions should produce an equal film coating quality. Strong [8] modified the *EE* factor as the inverse of the vaporization efficiency *E*, which has a finite range of zero to unity. The modified model of Strong is often used to describe and scale up pharmaceutical tablet coating in practice. Additionally, Rieck et al. [9] use a similar approach and define the drying potential $\Pi$ as governing factor for coating shell quality. They link the porosity of the coating shell formed by evaporation of aqueous solutions of crystalline material to the drying potential during the coating process. The goal of the present paper is to investigate experimentally, if the morphology of the coating layer is influenced similarly for coating with aqueous suspensions.

First, a short overview about the process of spray fluidized bed coating is presented. Then, the psychrometric description of spray fluidized bed coating with aqueous solutions will be explained, and the equivalency of the models of Strong [8] and Rieck et al. [9] will be shown. Following, a series of experiments regarding aqueous coating with suspensions will be presented, and conclusions on the dependency of coating shell quality on process conditions will be drawn.

## 2. Process description

Particle formation during spray fluidized bed granulation (Fig. 1) can take place along three different processing routes. When particles are sprayed onto with a liquid, containing different solid material than the particles, the process is called coating (Fig. 2A). As stated before, the coating shell protects particles, e.g., against mechanical stress, too fast dissolution or too fast release of drugs. Spraying the same solid material onto particles is called spray fluidized bed layering granulation (Fig. 2B). Here, the main goal is an increase in particle size or the transfer of a certain material from the liquid to the solid granular state. This pro-

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**Nomenclature**

| | |
|---|---|
| E | vaporization efficiency, – |
| EE | environmental equivalency factor, – |
| $EE^*$ | environmental equivalency factor modified by Strong [8], – |
| M | mass, kg |
| $\dot{M}$ | mass flow rate, kg $h^{-1}$ |
| N | number, – |
| p | pressure, bar |
| $q_3$ | normalized particle size distribution density with respect to particle volume, $m^{-1}$ |
| $Q_3$ | normalized cumulative particle size distribution with respect to particle volume, – |
| w | mass fraction, w-% |
| x | particle size, m |
| $x_{50}$ | particle size, where $Q_3(x_{50}) = 0.5$, m |
| Y | absolute gas moisture content per kg of dry gas, $kg_{H2O}$-$kg^{-1}_{dry\ gas}$ |

*Greek letters*

| | |
|---|---|
| ε | porosity, – |
| Π | drying potential, – |
| ρ | density, kg $m^{-3}$ |
| σ | standard deviation |

*Subscripts*

| | |
|---|---|
| AlOx | gamma alumina oxide spheres |
| avg | average value (for constant atomization pressure and constant limestone mass fraction) |
| bed | bed material |
| dry | dry sample after drying oven (including glass vessel) |
| end | at end of experiment |
| exp | from experiment |
| g | fluidizing gas |
| HPMC | hydroxyl propyl methyl cellulose |
| in | at inlet |
| lime | limestone |
| noz | nozzle |
| out | at outlet |
| p | particle |
| s | solid |
| sat | at saturation |
| shell | coating shell |
| spray | spraying suspension |
| start | at start of experiment |
| th | in theory (without porosity) |
| tot | total |
| ves | empty glass vessel for drying oven |
| void | void/pore |
| water | water/moisture |
| wet | wet sample for drying oven (including glass vessel) |

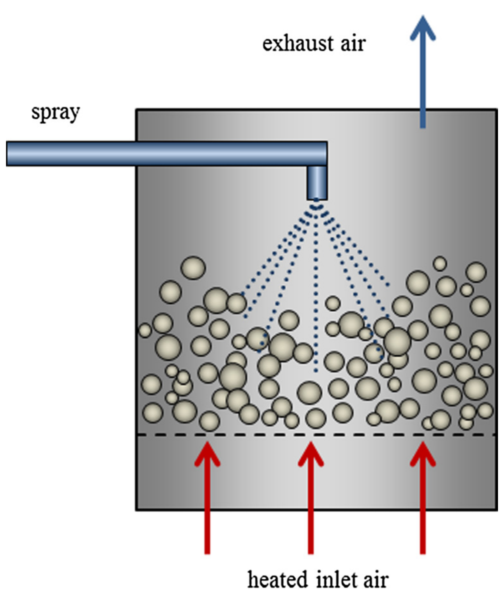


**Fig. 1.** Scheme of fluidized bed granulator with top spray nozzle.

cess is used for instance for salts, e.g. ammonium sulfate as a fertilizer [10]. Spraying binder with the intention of aggregating fluidized particles is called spray fluidized bed agglomeration (Fig. 2C, [11]). This technique is often used to improve the redispersibility of instant food powders and detergents [12], to reduce the dustiness or improve the flow properties of powders. In case of coating processes, agglomeration is unwanted, because large particle clusters may lead to de-fluidization of the bed and are hindering the complete coating of particles. Especially in the pharmaceutical industry, agglomerates of drug-containing particles may lead to overdoses and need to be avoided.

## 3. Psychrometric characterization of aqueous fluidized bed coating

An aqueous film coating process is characterized by moisture and temperature of inlet and outlet gas. On this basis, Ebey [7] derived the Environmental Equivalency factor *EE*, which was modified by Strong [8] to the following description:

$$EE^* = \frac{Y_{sat} - Y_{in}}{Y_{out} - Y_{in}} = \frac{1}{E}, \quad (1)$$

where *E* is the vaporization efficiency, which was originally derived by Reinald et al. [13], and $Y_{sat}$, $Y_{in}$ and $Y_{out}$ are the absolute gas moisture contents of fluidizing gas at saturation, inlet and outlet, respectively. The magnitude of $EE^*$ ranges from unity (saturation) to infinity (no evaporation), making it mathematically impracticable. Converting the $EE^*$ factor to the vaporization efficiency seems more practical, as the latter ranges from zero (no evaporation) to unity (saturation). Rieck et al. [9] characterized their spray fluidized bed coating experiments with the drying potential *Π*, describing the remaining capacity of the outlet gas to evaporate water. Accordingly, drying potential is calculated as follows:

$$\Pi = 1 - E = 1 - \frac{1}{EE^*} = \frac{Y_{sat} - Y_{out}}{Y_{sat} - Y_{in}}. \quad (2)$$

The drying potential has a range from zero (completely saturated outlet air, no remaining drying capacity) to unity (completely dry air, full remaining drying capacity). The concept of drying potential will be used throughout this paper.

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