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Identification of thermal zones and population balance modelling of fluidized bed spray granulation

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ABSTRACT

Air temperature measurements in a fluidized bed of glass beads top sprayed with water showed that conditions for particles growth were fulfilled only in the cold wetting zone under the nozzle which size and shape depended on operating conditions (liquid spray rate, nozzle air pressure, air temperature, and particles load). Evolution of the particle size distribution during agglomeration was modelled using population balance and representing the fluidized bed as two perfectly mixed reactors exchanging particles with particle growth only in the one corresponding to the wetting zone. The model was applied to the agglomeration of non-soluble glass beads and soluble maltodextrin particles spraying respectively an acacia gum solution (binder) and water. Among the three adjustable parameters, identified from experimental particle size distributions evolution during glass beads agglomeration, only one describing the kinetics of the size distribution evolution depended on process variables. The model allowed satisfying simulation of the evolution of the particle size distribution during maltodextrin agglomeration.

1. Introduction

Fluidized bed agglomeration is widely used in food and pharmaceutical industry to improve handling properties of powders (flowability, wettability, density, and reduction of dust formation) by changing the physical properties of individual particles (size, shape, density, porosity, and structure) [1].

Agglomeration is obtained by fluidizing particles with hot air, to allow their individualization and circulation, and spraying a liquid (solvent or binder solution) to wet their surface and render it sticky. If particles are soluble, the spray of pure solvent is sufficient to get sticky particles due to some local dissolution. If particles are non-soluble, a binder solution must be used. Collisions between wet particles occur due to the high mixing provided by the fluidized bed and allow the viscous layers at their surface to come into contact and to form dynamic pendular liquid bridges between them. Depending on the liquid viscosity, bridges will manage or not to dissipate the relative kinetic energy due to the collision preventing or not rebound of the colliding particles [2]. In the first case, solvent evaporation by the hot fluidizing air will lead to a consolidated solid bridge between particles. And the repetition of the different steps of wetting, collision and drying will give progressively rise to larger and larger agglomerates (agglomeration growth). In the second case, the binder layer at the particle surface will solidify and finally coat each particle (layer growth). Anyway, if drying is insufficient, a high humidity is generated and the bed risks collapse due to wet quenching. Conversely, when drying is too intense the solvent evaporates either before wetting particles or before collision between particles without any agglomeration. In the fluid bed, particles are also subjected to rupture and/or abrasion due to collisions between them or with the equipment walls [3,4]. The growth(s) mechanism(s) involved will depend on the apparatus, process and product parameters [5] such as fluidizing air temperature and flow rate; nature, concentration and feed rate of the sprayed solution; spraying system and particle properties.

A proper control of the obtained powder properties would require understanding of the mechanisms prevailing in the process. But it is impossible to expect that these mechanisms (e.g. wetting, collision, consolidation, and rupture) will occur singly or simultaneously or sequentially. Population balance equations (PBE) are convenient to describe the particle size distribution evolution during granulation process. They allow taking into account the different growth modes (layering, agglomeration) and rupture mechanisms. Momentum and

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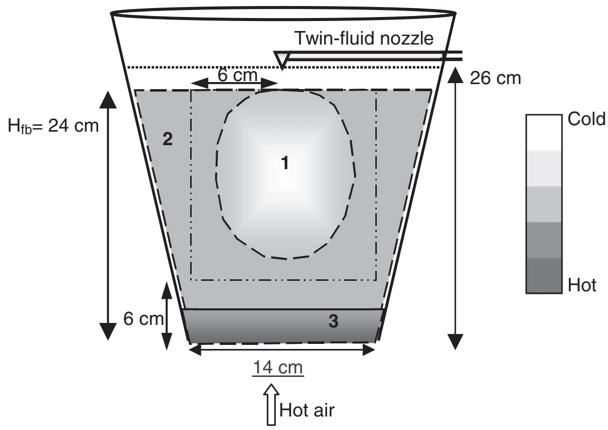

Fig. 1. Thermal zones in the conical fluidized bed granulator. 1. Wetting-active zone, 2. Isothermal zone, 3. Heat transfer zone. —— Thermal zones boundary. -·-·-·· Measurement region boundary.

heat balances are implicit. For an open well-mixed system where agglomeration, layering and rupture occur, the population balance equation can be written as Eq. (1):

$$\underbrace{\frac{1}{V}\cdot\frac{d(V\cdot\Psi(v,t))}{dt}}_{Accumulation} = \frac{1}{V}\cdot\underbrace{[Q_{in}(t)\cdot\Psi_{in}(v,t)-Q_{out}(t)\cdot\Psi_{out}(v,t)]}_{Input-Output} \underbrace{-\frac{\partial}{\partial v}\left[\frac{dv}{dt}\cdot\Psi(v,t)\right]}_{Layering} + \underbrace{F-E}_{\substack{\Uparrow\\ Agglomeration}} + \underbrace{C-D}_{Rapture} \quad (1)$$

where V is the system volume, Ψ(v,t) is the number density function at time t for particles with volume v, Q_{in} and Q_{out} are the volume flow rates of particles at the inlet and outlet of the considered volume V, F and E are the birth and death terms due to particles agglomeration, C and D are the birth and death terms due to rupture.

Due to bulk mixing of the particles, the fluidized bed is often represented as a fully mixed bed. But, when liquid is sprayed at the top of the bed, it is assumed that an "active" zone is formed, close to the surface, where both deposition of the spray on particles and solvent evaporation take place. Significant temperature gradients measured in that region of the bed confirmed the existence of this zone near the nozzle. And, air temperature distributions measured in fluidized bed equipment with different scales (laboratory, pilot) and geometries (conical, cylindrical) with top spraying of liquid (water or methanol) by means of different nozzles (single or twin fluid) led to consider three regions in the fluidized bed (Fig. 1) [6–11]:

1. The *wetting-active zone*, low temperature and high humidity region, near the spraying nozzle at the topmost part of the bed. It is characterized by high humidity and temperature gradients due to the wetting of the fluidized particles by the liquid sprayed and the evaporation of the solvent. In this region, symmetrical to the nozzle axis, air temperature increases from the centre sideways.
2. The *isothermal zone*, near the walls and around the wetting-active zone. In this region there is equilibrium between heat and mass transfer and air temperature is homogeneous.
3. The *heat transfer zone*, situated right above the bottom hot air distributor plate. In this narrow area, the hot air temperature decreases drastically due to the energy absorbed by the colder particles coming from the upper zones.

The size of the three zones varied with the operating parameters also affecting agglomeration efficiency, especially that of the wetting-active zone, depending on the diameter and penetration depth of the spray into the bed of particles. Particles will grow only if they circulate through this zone where conditions for agglomeration (wet sticky particles, collisions) are fulfilled [12]. The size of this zone and the rate of transfer of particles to this part of the fluidized bed will therefore determine particles growth and agglomeration efficiency.

In this study, air temperature measurements throughout the fluidized bed are performed at steady state, spraying water on inert glass beads to measure the fraction of the bed volume occupied by the wetting-active zone for different process conditions (air temperature, liquid spray rate, nozzle air pressure and initial particle load). A model of the granulator is then proposed and combined with population balance equation to simulate particles agglomeration. Parameters of the model are identified using results of experiments with model glass beads (non-soluble) and maltodextrin particles (soluble) agglomerated respectively with acacia gum solution and water.

2. Materials and methods

Experiments were performed in a pilot batch fluidized bed of conical shape UNI-GLATT (Glatt GmbH Process Technology, Germany) (Fig. 1). Liquid (20 °C) was top sprayed by means of a bi-fluid nozzle in a full cone. Model inert glass beads (d_{50} = 160 μm, ρ = 2490 kg m^{-3}, DUP, France) and soluble maltodextrin particles (d_{50} = 180 μm, ρ = 1424 kg m^{-3}, Glucidex 12, Roquette, France) were fluidized and heated with a constant air flow rate (157 m^{3}h^{-1} for glass beads and 120 m^{3}h^{-1} for maltodextrin particles) allowing keeping the same fluidized bed height (h_{fb} ≈ 24–25 cm) with a constant gap (1–2 cm) between the top of the fluidized bed and the tip of the nozzle.

For the measurement of air temperature profiles, at steady state, in the central zone of the fluidized bed, glass beads were top sprayed with water (20 °C, spray angle between 20 and 40° (Table 1)). Three series of six thermocouples were positioned at 0, 3 and 6 cm from the axis of the apparatus and, for each position, at 6, 10, 16, 20, 22 and 24 cm from the air distribution grid. Assuming symmetry of temperatures around the bed axis, it allowed the characterization of a cylindrical measurement region right below the nozzle representing 32% of the fluidized bed volume (Fig. 1).

For agglomeration trials, the initial load of particles was first heated by the hot fluidizing air (10 min, constant temperature). The

Table 1
Spray angle, bed average temperature T_b and wetting-active zone characteristics (volume fraction of fluid bed α, height and shape) (reference: 500 g glass beads, air 70 °C, water 5.33 ml min^{-1}, 1 bar).

Variables	Reference	250	750	60	80	2	3	2.65	7.75
		g	g	°C	°C	bars	bars	ml min^{-1}	ml min^{-1}
Spray angle (°)	38	38	38	38	38	27	23	34	39
T_b (°C)	52	53	53	46	60	52	52	54	50
α (%)	29	>31	24	>30	22	18	28	14	29
Height (cm)	16	17.5	12	18	14.5	17	18	14	17
Shape	2	1	2	1	2	2	2	2	2