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Fluidised bed agglomeration of particles with different glass transition temperatures

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ABSTRACT

Fluidised bed agglomeration of particles consists in spraying liquid drops (water or binder solution) on the surface of particles, fluidised by hot air, in order to create sticky regions to allow formation of agglomerates when the sticky particles collide. Many parameters influence agglomerate growth, especially those controlling the particle circulation, and the water and temperature conditions within the bed that determine drying and particle stickiness linked to the glass transition of amorphous components and to the viscosity of moist zones at the particle surface.

Maltodextrin DE12 and DE21 particles with different glass transition temperature domains were agglomerated in a batch bench scale fluidised bed, under constant mechanical constraints, changing the sprayed water feed rate and the fluidisation air temperature in order to investigate the influence of particle stickiness on agglomerate growth kinetics and mechanism.

The two powders showed a different sensitivity to the water and temperature constraints applied. Whilst for maltodextrin DE12, the size and growth rate increased significantly with the sprayed water flow rate, only a small variation was observed for maltodextrin DE21. In both cases, agglomeration occurred in two stages: firstly, the association of initial particles and secondly the agglomeration of intermediate agglomerates into larger and more porous structures. The change from one growth mechanism to the other depended on the conditions, and influenced the size distribution and structure of agglomerates.

1. Introduction

Agglomeration is widely used in the food and pharmaceutical industry where a great amount of the raw materials, intermediate or final products are in powder form. Agglomerates consist in the assembly of fine particles (~200 μm) linked by solid bridges to create large porous structures (~800 μm). By modifying the physical properties of the powder particles (size, shape, porosity, density), agglomeration allows modifying the powder end-use properties (flowability, wettability, instantaneity).

Fluidised bed agglomeration consists of spraying a liquid (water or binder aqueous solution) on a bed of solid particles fluidised by hot air. Sprayed liquid droplets collide with the individualised and moving particles, allowing to create sticky zones on their surfaces, either due to the local wetting of the particle surface (water soluble particles) or to the deposit of binder solution on the particle surface (non soluble particles). Agglomerate formation takes place when the sticky moving particles collide with others (sticky or not) and form liquid or viscous bridges that are dried and consolidated by the fluidisation hot air. The repetition of these steps (spraying, wetting, collision, adhesion and drying) allows progressive agglomerate growth.

Fluidised bed agglomeration is therefore a complex process resulting in different phenomena. Agglomerate growth, structure and properties depend on many parameters linked to the equipment, to the operating conditions, and to the particle and sprayed liquid properties (Table 1) [1]. These parameters determine the mechanical, temperature and water constraints applied inside the particle fluidised bed. Mechanical constraints are mainly due to the agitation of particles by the fluidising air allowing both contacts between particles by friction or collision, and the distribution of the sprayed liquid within the bed. For a given device and geometry, mechanical constraints mainly depend on the fluidising air flow rate; on the particle load in the chamber; and on the particle size, size distribution and density influencing fluidisation. Temperature constraints are linked to the temperature and flow rate of both the hot fluidising air and sprayed liquid. They determine the temperature and the drying conditions within the particle bed and therefore also influence the water constraints. Controlled agglomerate growth occurs

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Table 1
Main parameters in fluidised bed agglomeration and their contribution to the mechanical, thermal and water constraints.

Geometry of granulator	Operating parameters	Product parameters
Chamber	Particle load	Particles
* Size	Fluidising air	* Composition
* Shape	* Flow rate	* Size/size distribution, density
Air distributor	* Temperature	* Surface
Nozzle	Spraying	Sprayed liquid
* Type	* Liquid feed rate	* Composition/concentration
* Position	* Liquid drop size	* Temperature

when the conditions allow bridges to create between colliding particles without over wetting and collapse of the bed. This requires controlling at the same time agitation, wetting and drying of the particle bed (i.e. intensity and duration of the constraints applied).

The wetting of the particle surface by the sprayed liquid allows the formation of liquid or viscous bridges between colliding particles thanks to the viscous dissipation of the collision kinetic energy. Actually, a collision between two particles is effective only if the Stokes number St is below a certain critical value St* [2,3]. Above St*, particle rebound is observed and the collision does not allow agglomerate growth:

$$St = \left(8 \cdot \rho_p \cdot r_p \cdot u_c\right)/(9 \cdot \mu_l)$$

and

$$St* = (1 + 1/c)\ \ln(e/r_u)$$

Where ρ_p is the particle density (kg m^{-3}), r_p is the mean particle radius (m), u_c is the relative collision velocity (m s^{-1}), μ_l is the viscosity of the viscous film (Pa s), c is the collision restitution coefficient (-), e is the viscous film thickness (m), and r_u is the particle surface rugosity (-).

However, the formed liquid or viscous bridges must also be able to give rise to solid bridges by the evaporation of the solvent (water) during hot-air drying. For the agglomeration of water insoluble particles the spraying of an aqueous solution containing a binder (e.g. amorphous component) is needed to form a viscous adhering film on the particle surface and render it "sticky". When particles are soluble in water and contain amorphous components, they can be agglomerated by spraying pure water. In this case, water can cause both some partial dissolution and plastification, decreasing the glass transition temperature of the amorphous components on the particle surface [4]. In the specific temperature conditions of the fluidised bed, these plasticized amorphous components undergo glass transition causing a decrease of their viscosity and the appearance of stickiness when they reach the rubbery state. This corresponds to a critical viscosity range between 10^8 and 10^{12} Pa s [5,6]. For a given water content, temperatures for sticky conditions range between the glass transition temperature (Tg), and the "sticky temperature" (Ts); from 20 to 30 °C above Tg for the main food carbohydrates (maltodextrin, lactose, fructose, sucrose, etc.) [7,8]. Tg and Ts depend on the composition of the product considered.

During fluidised bed agglomeration, the particle surface water content and temperature permanently vary along the bed and with time due to simultaneous wetting and drying of the particles (1–4, Fig. 1) and to the agitation causing their circulation through the thermal zones within the bed [9,10]. The presence of sticky particles able to agglomerate when colliding therefore depends on their position, on temperature and water conditions within the bed and on the particle surface composition [4,8,11]. Different studies were performed on the effect of parameters such as fluidising air temperature and flow rate; nature, concentration and feed rate of the sprayed solution on agglomerates growth [12–17]. Most of them were performed with non soluble particles and only few investigated the behaviour of particles undergoing glass transition.

The aim of this work was to study the agglomerate growth during fluidised bed agglomeration of particles with different glass transition temperature evolution with the water content. Two model powders e.g. maltodextrins with different dextrose equivalent (DE12 and DE21), were agglomerated spraying water. Similar mechanical constraints (same air flow rate, particle load and initial size distribution) were applied, but temperature and water constraints were changed by modifying the sprayed water feed rate and the fluidising air temperature. Agglomerate growth was studied following the evolution of both the median diameter and particle size distribution in order to consider the influence of the studied parameters on both the growth kinetics and growth mechanisms.

2. Materials and methods

2.1. Powders

Maltodextrin DE12 and DE21 (Glucidex, Roquette, Fr) were used for agglomeration trials (Table 2). Prior to each trial, powders were sieved (Analysette 3 Spartan, Fritsch, Ge—80 g, 12 min, amplitude of vibration 2.5 mm) to obtain a narrow size distribution. The fraction between 100 and 315 μm was kept in order to have a median diameter d_{50} of about

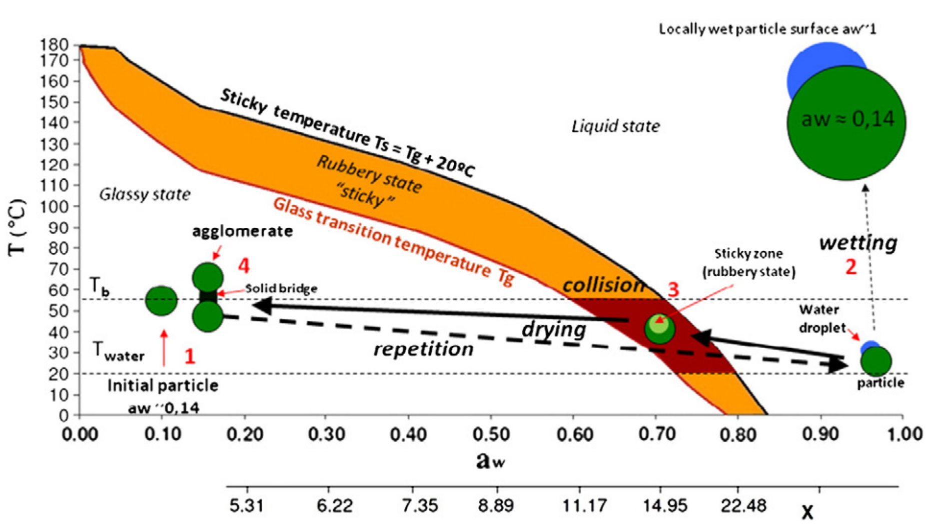

Fig. 1. Glass transition and sticky temperatures (Tg and Ts) as a function of water content X and water activity a_w for maltodextrin DE12 and evolution of temperature and water content of moist zones on the particle surface during the different steps of fluidized bed agglomeration (1. initial particle; 2. wetting; 3. drying/collision; 4. solid bridge/dry agglomerate).