Optimisation and Process control of fluid bed coating

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INTRODUCTION

Coating is a powerful process to modify properties of particles. Sticky powder could be converted in free flowing powder. Coating may constitute a barrier to oxygen, water, allowing protection of the encapsulated materials. Well selecting the coating materials allows controlled release at the right place following the adequate profile.

Fluid bed coating, especially Wurster process (see description below) are one of the most usual technologies for particle coating. However, the process driving is still based on empirical data and good quality coating requires an experimented operator. The following paper reports work done in our group to understand the different processes involved in fluid bed coating and proposes some approaches to optimize and to control the process.

MATERIAL AND METHOD

Equipment : Coating was realized in a fluid bed reactor (Uniglatt, Glatt, Binzen, Germany) equipped with a Wurster inner tube (8.5 cm). In some experiments, an insert was introduced at the bottom of it to convert the reactor to a spouted bed (Figure 2) and force all air to pass through the central tube. Coating was sprayed at the bottom of the reactor with help of peristaltic pump.

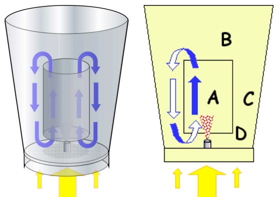

Figure 1 : Wurster reactor

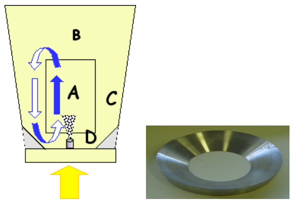

Figure 2 : spouted bed reactor and insert

Protocol *:* Particles were introduced in the reactor. Air ventilator was run. Spray was started when temperature was stabilized in the reactor (generally 60 °c). After end of spraying, reactor was let stand running to finalize particle drying. Then particles were collected and analysed.
Beads : Microcrystalline cellulose beads (IPC, Dresden, Germany) of different sizes (350 and 600 µm) were used as support for coating. Particles were dried in autoclave at 120 °c for 15 minutes before used for micro-organisms coating.

Coating : Coating solutions were generally dispersions of arabic gum (300 g/L, IRX40693, CNI, Rouen, France) in distilled water or dispersions of skim milk (400 g/L) and sucrose (100 g/L) in distilled water.
Probiotics **:** Freeze dried powders of *lactobacillus casei Lc1* (CNCM MA43/6V, Lesaffre, Marcq en Baroel, France) and *Lactobacillus acidophilus R0052* (CNCM I-1722 OI 5062, Lallemand, Blagnac, France) were used as model of sensitive materials. In both cultures, powder was added to the coating solution to reach a concentration of 2 10^9 cfu/ml and let stand 15 min for re-hydration before spraying.

RESULTS

General observation of the process

In the Wurster process (Figure 1), reactor is equipped of a central tube. Thank to a hole distribution in the bottom plate, 80-90% percents of air pass through this tube provoking a particle upward motion (A). On top of the reactor (B), particles are slow down and fall back in the annular zone (C). Finally, they are transfered from annular zone to the central zone and accelerated again (D).

Conventional reactor design : Wurster process

Figure 3 shows one record of the pressure drop over the reactor in function of the air flow rate through the reactor (El Mafadi, 2003). For low air flow rate, particles remain static and pressure drop stable over time. When increasing the flow rate, the particles start to move. One could observe an expansion of the bed volume to get a fluid bed (particles are suspended in air and behave like a fluid). At still low rate (50 m^3/h), the fluidisation seems unstable (large variation of the bed height, bubbling in the bed) and pressure drop are fluctuated. For a higher flow rate (90 m^3/h), both fluidisation and pressure drop stabilize. However, while flow rate (> 120 m^3/h) is increased too much, pressure drop again fluctuates and fluidisation is difficult to control.

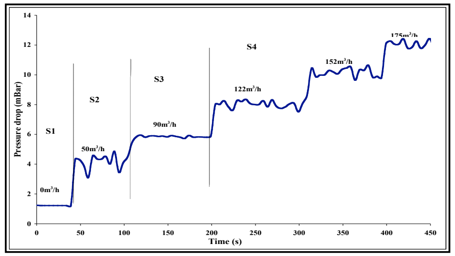

Figure 3 : Reactor pressure drop in Wurster reactor

Zone	Residence	Temperature
A	0.15 s	43 °c
B	0.9 s	
C	6 s	60 °c
D	0.05 s	

Table 1: Wurster reactor residence times and temperature distribution

Pressure drop stability is a good indicator of the homogeneity of fluidisation. To get a correct coating, the concentration in the central zone must in a certain range favour a good probability of impact between the particles and the sprayed droplet. This condition could only be reached well if the fluidisation is stable over time. In the Wurster process, observations shown that the "window" of flow rate value is quite limited.

Using a particle dynamic model (Femlab and matlab simulation) and particle velocity measurement (Laser velocimetry, LDSystems), the residence time in the different zone of the Wurster reactor was evaluated (Table 1). It appears that the particles resides 80 % of the time in the annular zone. In the central zone, particles received sprayed liquid droplets. The heat effect of the air drying flow is mainly compensated by the cooling effect of the droplet evaporation. However, in the annular zone, as particles has to be dried to avoid agglomeration, no evaporation compensates the heating effect and one could expect particle warming. This assumption is confirmed by the temperature measurements (Table 1) proving a real cooling effect in the central zone while annular zone is at air drying temperature. For thermo-sensible material, the Wurster process may then not be totally adequate.

Modifying the reactor design : spouted bed.

To overcome this problem, the reactor has been modified by introducing an insert (Figure 2) (El Mafadi, 2003) to avoid air flow through the annular zone. To avoid risk of agglomeration, this insert was designed to form a cone promoting flowing of particles to the central zone.

Initial observation of the particle motion shows a similar circulation in the reactor but more homogeneous flow. Figure 4 shows the pressure drop over the reactor for different flow rates. If a minimum flow rate is required for homogenous fluidisation, the system remains stable even for high flow rate. This allows more freedom in selecting the conditions adapting the air flow to reach enough drying capacity and avoiding agglomeration. Further experiments shown that the central tube could be removed with effecting the particle circulation. Removing central tube presents a real advantage as part of the coating sticks to it, reducing the efficiency and quality of the coating.

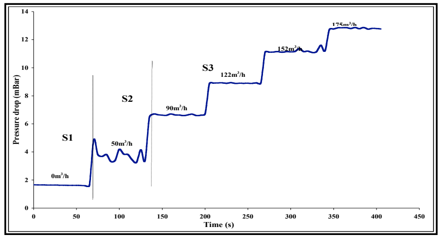

Figure 3 : Reactor pressure drop in spouted bed

Probiotic strain	Survival (%)		impro-vement factor
	Wurster	Spouted bed	
L. casei Lc1	40.1 ± 6.9	53.6 ± 7.2	1.34
L. acidophi-lus R0052	7.6 ± 1.3	15.3 ± 1.4	2.02

Table 2 : Coated probiotic survival

In the modified system, the temperature is homogenous over the reactor. As no air circulates into the annular zone, no over heating is observed. To confirm the impact of the reactor design, beads were coated with a suspension of cells in milk/saccharose solution using either the conventional Wurster reactor and the modified spouted bed reactor (Elmafadi, 2005). Depending of the probiotic strains, survival was improved by a factor up to 2 (Table 2).

Prevention of agglomeration using a bang-bang control.

The most operational problem encountered during fluid bed coating is the agglomeration. It could be due either to lack of drying or temperature over the glass transition. In both case, particles are sticking leading to agglomeration.

An experimental plan has been performed to see which parameters (T, air humidity, pressure, ...) fluctuate during fluid bed coating (Prata, 2009). Only pressure drop over the reactor allows to predict agglomeration. Mainly all other parameters only change after agglomeration has started.

Generally, coating flow rate is fixed at the beginning of the process at a level that avoids risk of agglomeration (usually at 50 % of maximum drying capacity of the reactor). When operator "sees" a risk of agglomeration (based on process sound, sample taken from the reactor), he stops the pump and restarts after a "while". Based on previous observations, an automatic control system was set-up consisting in measuring the pressure drop in the reactor and switching on and off in function of its value. Coating solution pump speed was fixed at 100 % of the maximum drying capacity of the reactor. Several conditions were tested (air temperature, atomization pressure, particle load ...) without observing any agglomeration but a process time mainly 2 times shorter than with "manual" control. Globally, the process has been run at 90 % of its maximum capacity.

CONCLUSIONS

Fluid bed coating has been used for more than a half century. Thousand tons of coated material are produced every years but it still lets place for innovations. This paper reports that changing slightly the design or using simple process control may improve quality of the coating (better fluidisation), integrity of the encapsulated material (survival factor) and the process cost (coating time).

Other approaches may be considered to innovate in the fluid bed coating process such as dry powder coating (Bilancetti, 2008), quality control (Depyrere, 2009) or continuous coating process (Tenou, 2002).

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