P-005 **Developement of process-control for coating in fluid bed**

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INTRODUCTION AND OBJECTIVES

Fluidized bed coating is a commonly applied technique to microencapsulate food powders. The basic principe of fluidized bed coating is to atomize a fine liquid spray into a bed of fluidized particles. The spray consists of a solute that acts as a coating medium which is dried by the fluidized air. There are several type of process for the fluidized bed coating. One of them is the Wurster process which is recognized for precision application of the film coat to particulates materials (Teunou 2002). This process is characterized by the location of the spray nozzle at the bottom of the fluidized bed and the presence of the Wurster tube to induce a cyclic flow of the particles (Figure 1a).

The most operational problem encountered during fluid bed coating is the agglomeration. It could be due to lack of drying or temperature over the glass transition. In both case, particles are sticking leading to agglomeration. A previous work studied which parameters (T, air humidity, pressure,...) fluctuate during fluid bed coating and showed that only the pressure drop over the reactor allowed to predict agglomeration. Based on this observation, an automatic control system was set-up consisting in measuring the pressure drop in the reactor and switching on and off the spray flow when the fluctuation is too high (Poncelet 2010). To optimize this system, we need to understand why the pressure drop is a signal to avoid agglomeration.

The idea is to break down the process step by step. This work presents the first step without coating. The two goals are to define which operating parameters are represented by the pressure drop and to find the part of the reactor where the measure of the pressure drop is the most signifiant.

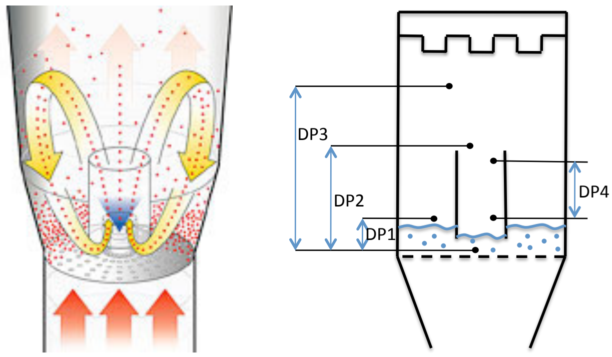
MATERIALS AND METHODS

Fluidized bed equipment

All measures are performed in the bottom spray reactor of the Uni-Glatt (Glatt, GmbH, Binzen, German). The capacity of this fluidized bed is 1 kg. For pressure drop measures, microcrystalline cellulose beads (IPC, Dresden, Germany) are used as fluidized particles. Three different diameter ranges were selected: 212-300 µm, 710-850 µm and 1000-1400 µm. For all of these three dimensions of particles, three amounts of powder (300 g, 600 g and 900 g) are fluidized at relatively low and high air flow rates. These air flow rates are included between 80 and 220 $m^3.h^{-1}$.

Pressure drop measurements

The pressure drop are measured at different operating conditions using four transmitters of differential pressure that are placed in the fluidized bed (Figure 1b).



a) b)

Figure 1: a) Schematic representation of the Wurster coating process, b) Position of the measures of differential pressure

The pressure drop DP1 allows to measure the effect of the particles bed in the annular zone. DP2 is the measure of the pressure drop generating into and below the Wurster tube. DP3 gives the total pressure drop of the system. And, DP4 is the measure of the pressure drop in a part of the Wurster tube on 10cm-length. This last pressure drop allows to calculate the porosity into this tube.

Experimental design

A box-Behnken design (Goupy 1999) are performed to improve the influence of three operating conditions on the pressure drop. The coded and actual values of the investigated process variables (fluidization flow (Q_f), particle diameter (d_p) and particles amount (m_p)) are illustrated in Table 1. The software JMP®8 is used to fit polynomials to the response.

Table 1: Actual and coded values of the investigated process variables

			Actual values (coded values)		
Factor		Units	Low	Medium	High
(A)	d_p	µm	260 (-1)	780 (0)	1300 (+1)
(B)	m_p	g	300 (-1)	600 (0)	900 (+1)
(C)	Q_f	$m^3.h^{-1}$	80 (-1)	150 (0)	220 (+1)

RESULTS AND DISCUSSION

The results obtain with experimental designs are analyzed for each pressure drop and are presented in figure 2. This figure shows two groups of results. The first group is constituted by DP1, DP2 and DP3 and the second group by DP4.

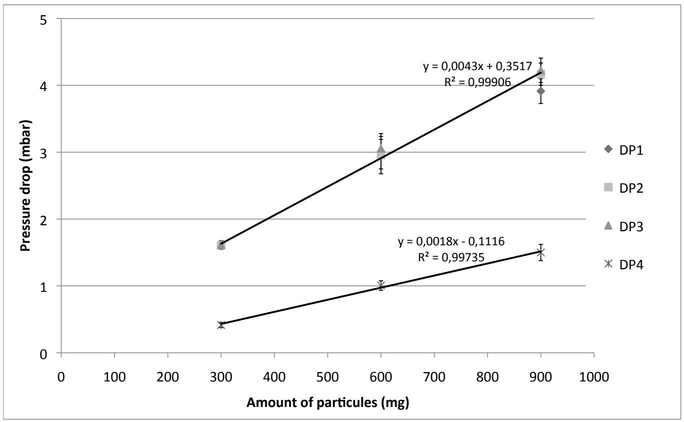


Figure 2: The four pressure drops for different amounts of particles

The values of the pressure drops DP1, DP2 and DP3 are nearly. DP2 = DP3 means that the pressure drop between the top of the Wurster tube and the superior part of the system is equal to zero. The concentration of particles in this part is negligible. DP1 = DP2 means the concentration of particles falling on the annular zone is too lower than these of the bottom of the annular bed. The measure of these three DP shows that the difference of the pressure drop is only significant at the bottom of the annular zone.

DP4 is three times lower that the others pressure drops but theirs values is not negligible. This pressure drop can give the porosity of the tube and moreover the amount of particles on the Wurster tube. One hypothesis is: if an agglomeration phenomena appears, the quantity of particles would change in the tube; in this case, DP4 is a signal to stop the spray flow.

These result allow to conclude that the signal to the agglomeration phenomena is located in the bottom of the annular tube and/or on the Wurster tube.

An experimental design is performed to understand and find the parameters which are measured by the pressure drop. Among the three parameters (fluidization flow (Q_f), particle diameter (d_p) and particles amount (m_p)), the pressure drop is only in function of the particles amount. The figure 2 shows that this relation is linear: the pressure drop increases when the quantity of particles in the system increases. This observation is in relation with the definition of the fluidization state. Indeed, the fluidization state takes place when the pressure drop induced by the air flow is equal to the mass of the bed by surface unit of the reactor.

The pressure drop is independent on the fluidization flow. This result means that all experiments are performed in the stable fluid bed regime (Teunou 2002). In this case, the pressure drop is defined by the following equation:

$$\Delta P = (1-\varepsilon)(\rho_p - \rho)gH$$

with ε the porosity, ρ_p the density of particles (kg.m^{-3}), ρ the density of air (kg.m^{-3}) and H the bed level (m).

An other parameter that could be play a role on the pressure drop is the partition gap (Shelukar 2000). To evaluate this effect, the table 2 presents DP4 in function of the partition gap and the amount of particles.

Table 2: DP4 in function of partition gap and particle amount (mbar) with d_p=780µm and Q_f=150m^3.h^{-1}

	300 g	600 g	900 g
5 mm	0.49	0.63	1.04
10 mm	0.42	1.00	1.50
20 mm	0.36	0.65	1.55

Firstly, it appears with the table 2 that the partition gap is also a critical parameter. Secondly, it seems that partition gap and amount of particle are dependent: to maximize the pressure drop in the tube, it need to increase the partition gap when the particle amount increases.

CONCLUSIONS

Previously work showed that the pressure drops monitoring permits to avoid agglomerations. Four measures of the pressure drop are performed to understand why the pressure drop in the signal of agglomeration. The first results show that the measure of the pressure drop is only sensitive in the bottom of the annular tube and/or on the Wurster tube. Among the proceeding parameters, the pressure drop is in relation with the among of particles in the bed and with the partition gap. A variation of the mass of the bed in the annular zone and on the Wurster tube should be the signal that agglomeration begins. Others measure of pressure drop should be performed. The next studies are focused on the partition gap zone.

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