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Development of a control system to anticipate agglomeration in fluidised bed coating

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ABSTRACT

Fluidised bed coating is a technology commonly used to modify the properties of pharmaceuticals or powdered food. However, too high moisture content during the process, due generally to insufficient drying or the use of a temperature above glass transition of the polymer solution, usually induces particle agglomeration. Various processing elements can be involved however, and this paper provides an analysis of these variables in order to determine which parameters should be controlled and which can be manipulated. It was found that the pressure must be controlled and that the flow of the coating solution can be manipulated to do this. Since a bang-bang controller can manipulate the flow of the coating solution, it was adopted here, with agglomeration avoided by maintaining the pressure in an adequate range. The system was used successfully to coat microcellulose beads with gum Arabic; moreover, it was possible to increase the quality of production.

1. Introduction

Fluidised bed technology can be used to coat particles by spraying them with a solution of any desired material. Industrial companies use this technology to mask undesirable tastes, control the release of a compound, or change the physical properties of products. Coating can also improve handling conditions and storage stability, as well as reducing sensitivity to light, oxygen and moisture. The coating can be sprayed on using either a top-down or a bottom-up configuration, with the former being especially adapted for dealing with a large number of particles, although the coating will not necessarily be homogeneous. When the spray nozzle is placed at the bottom, the coating material raises the particles, preventing the premature drying of the solution before reaching and coating the particles. The addition of a cylindrical central tube (the draft tube) in a bottom spray configuration, known as the Wurster process [1], allows better control of particle movement, thus facilitating modelling and control. Indeed, the airflow can be concentrated in this tube, reducing the drying time of the coating droplets and enhancing the homogeneity of the coating. Fig. 1 shows the movement of particles in such a system. They are driven up by the air current into zone A (spraying zone) of the inserted cylinder and are wet by the coating liquid. In zone B (cylinder zone), the wet coated particles are transported by pneumatic conveyance with most of the drying process taking place in this area. In the annular zone (zone C), the particles fall downwards to the bottom of the fluidising chamber and, in the buffer zone at the bottom (zone D), they are recirculated into the spraying zone.

One of the main problems in the fluidised bed process is uncontrolled agglomeration of particles. This agglomeration is the result of the coalescence of the wet coated material, which forms liquid bridges between the particles [2]. Solvent evaporation then leads to the solidification of these bridges, thus forming agglomerations. This phenomenon can also occur when the temperature of the particle surface is above that of the glass transition of the coating substance [3]. Such agglomerations can be desirable for increasing the solubility of fine powders, but they tend to reduce the quality of the finished product and can even lead to defluidisation. Detection of the formation of agglomerations is difficult in real time and requires the constant attention of an experienced operator. Also, the few sensors installed in industrial processes are not used for the detection of agglomerations, and production is therefore limited. These problems partially explain the high cost of production and the slow development of this technology.

Some authors have suggested that establishing optimal operating conditions can prevent agglomeration. Jiménez et al. [4] studied agglomeration as a function of inlet temperature, coating solution flow, atomisation pressure and particle type for a top spray configuration, but the correlations established varied with specific conditions and are valid only for that chamber design. Hede et al. [5] also studied the influence of operating parameters on the percent of agglomeration. They showed that an increase in atomisation pressure and coating solution concentration favoured the formation of agglomerations, whereas inlet temperature and airflow have less influence. Zhonghua and Mujumdar [6] developed mathematical models of agglomeration based on the instability of the fluidisation the bed as a function of

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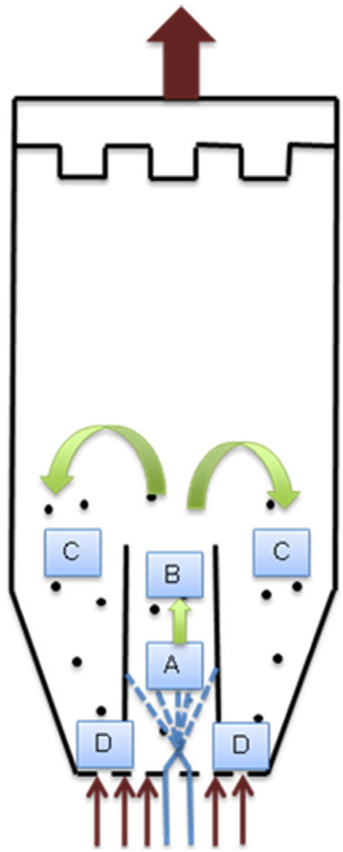

Fig. 1. Definition of different zones of the Wurster fluidised bed coating. A. Spraying zone, B. Ascendant zone, C. Deacceleration zone, D. Tampon zone.

pressure and airflow. The large number of inter-related parameters, however, complicates the prediction of agglomeration [7]. For instance, an increase in airflow facilitates drying, but can induce unstable fluidisation. Consequently, initial conditions must be chosen to assure that the agglomeration phenomenon does not occur, even though these may prevent maximal efficiency. Another approach consists of using real-time measurements to detect default values [8], for example, the presence of clogging can be detected by a drop in pressure [9,10]. Bed defluidisation can also be detected by such drops in pressure thus indicating the presence of agglomeration [11,12]. In fact, pressure is a good indicator of the state of fluidised beds [13]. Croxford and Gilbertson [14] used a linear proportional–integral–derivative (PID) controller to achieve the steady state of the bed by manipulating airflow rate, but without any coating. In all of these proposals, however, the detection of the problem leads to the termination of the process and the loss of the batch. The most efficient solution is to employ an automatic control process. Some authors such as Larson et al. [15] and Wang et al. [16], used an automatic control to measure the moisture content in a fluidised bed dryer or coater to optimise the process. However, this approach needed extensive previous calculations.

Control theory aims at maintaining optimum or reference settings of specific output variables for the process, as well as establishing a protocol to be followed in the face of eventual problems. In principle, certain variables (called inputs) are manipulated to maintain the output variables as close to the reference values as possible. These output variables are measured using software sensors or estimated using indirect measurements. Controllers must thus modify the manipulated variable so that it approaches the reference value.

This paper proposes a Single Input Single Output (SISO) control strategy allowing the rapid detection of agglomeration and the adjustment of the operating variables to be manipulated to facilitate successful completion of the coating process. Initially, the variables adequate for manipulation and reference (control) were determined; the feasibility of this manipulation was then tested using an automatic controller to coat microcrystaline cellulose beads with gum Arabic.

2. Materials and methods

Microcrystalline cellulose (MCC) beads (780 μm, IPC, Germany) were coated with an aqueous suspension of gum Arabic (CNI, Rouen, IRX 40693) in distilled water (300 g/L).

A fluid bed laboratory reactor (UNI-GLATT, Glatt, Binzen, Germany) equipped with an inner Wurster tube (diameter: 75 mm) was used. This conical vessel presents an angle of 9.8° with respect to the vertical axis; it has a base diameter of 150 mm and a height of 460 mm (Fig. 2). The bottom grid directs 80% of the hot airflow into the central tube and the coating solution is sprayed up by a peristaltic pump (1001-SR/65, Petro Gas Ausrustungen, Berlin) through a two-fluid nozzle (SHLICK 970-S3, Germany).

Pressure, temperature and relative humidity were determined at intervals of 5 s, at different points in the fluidisation chamber. Fig. 2 shows the placement of the thermo-hygrometers (HUMICAP HMP 110, Vaisala, Finland) at the gas inlet and at the top of the fluidisation bed, as well as at the differential pressure sensors (0–12.5 mbar, Delta_P, Halstrup Walcher, Germany). Most of the experiments were repeated 3 times with good reliability.

Experimental design can reduces the number of experiments and yield a statistical analysis revealing the effects of parameters and their interactions. The Box Behnken design, which consists of variations of three inlet factors, for a total of 15 experiments, was adopted here. The range of these factors was selected based on preliminary assays for atomisation pressure and airflow. The range of coating flow was determined as a function of the drying capacity of this reactor.

A closed loop control of the process was carried out using the Simulink toolbox and RTI Matlab software (MathWorks, USA), equipped with a card entry/exit ControlDesk (dSPACE GmbH). Each pair of pressure measurements is linked to two analogic entries on the capture card. The Simulink software permits the use of various control algorithms. For this paper, a simple bang bang controller was employed [17,18].

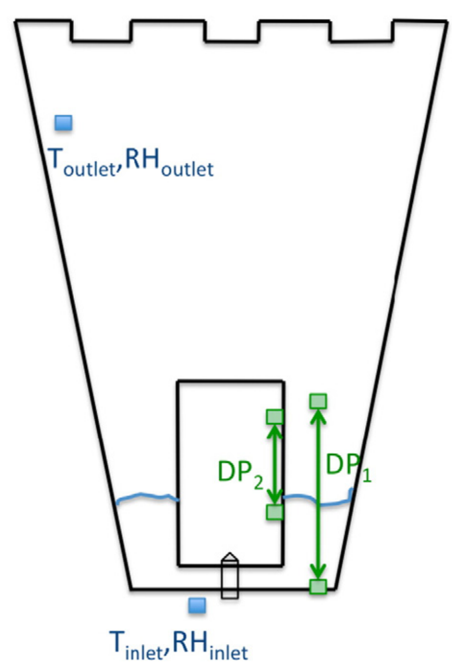

Fig. 2. Location of transducers for evaluation of coating process. Temperatures, relative humidity in outlet: (T_{outlet}, RH_{outlet}) and at inlet of air: (T_{inlet}, RH_{inlet}), as well as pressure in annular region (DP_1) and in the draft tube (DP_2).

3. Results and discussion

The control process was established on the basis of the identification of a controlled reference parameter and the determination of a variable to be manipulated.

3.1. Controlled reference parameter identification

Premature agglomeration should ideally be detectable from the presence of specific signals, i.e., the most adequate parameter should be selected based on its ability to indicate quickly the initiation of

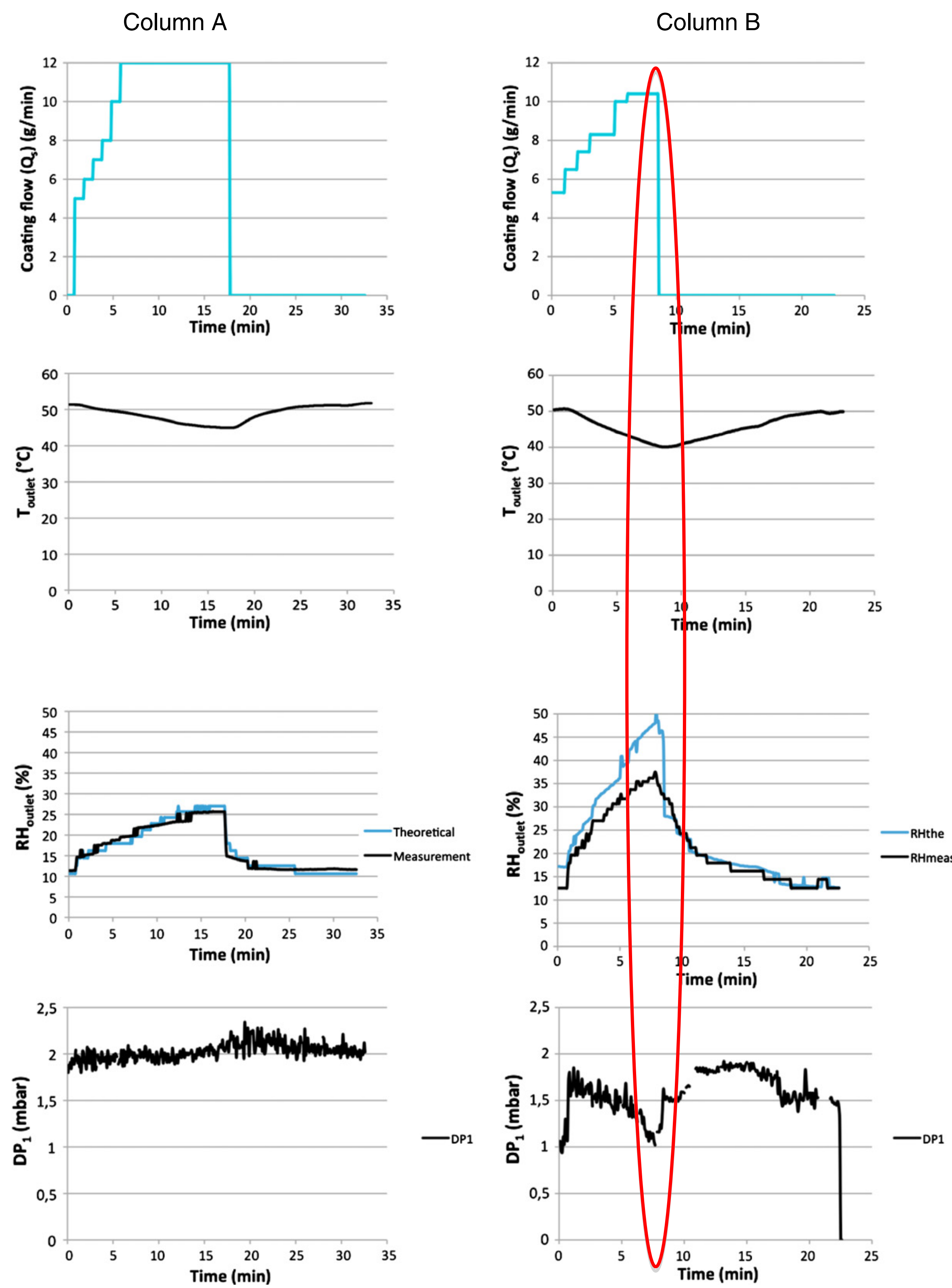

Fig. 3. Relation between coating flow and outlet handling parameters of temperature, relative humidity and pressure with 150 $m^3.h^{-1}$ of airflow (not agglomeration: column A) and 50 $m^3.h^{-1}$ (with agglomeration: column B).

agglomeration. It must be chosen from the handling parameters of outlet temperature, outlet relative humidity and the pressure at two points.

Experiments were initially carried out for a wide range of coating rates to evaluate the mechanism of agglomeration. For these experiments, the three handling parameters mentioned earlier were measured. The coating was sprayed on the beads at two different rates of airflow, with the coating solution rate was increased by 1 g.min^{-1} until coating equilibrium was reached (or until 20% by weight had been added). Fig. 3 summarizes the handling parameters during the spraying and drying phases of the coating process for 600 g of spherical MCC particles, a coating flow of 12 g.min^{-1} and an atomisation pressure of 1.5 bar. With an airflow of 150 m^3.h^{-1}, no agglomeration was observed, although when this was reduced to 50 m^3.h^{-1} agglomeration did occur.

The outlet temperature decreased during spraying but increased when the coating flow was stopped. However, the curve was the same for the two airflows tested. Outlet temperature therefore does not represent a relevant indicator of agglomeration.

For the relative humidity, the actual experimental value was compared to the calculated theoretical value. If the experimental value is superior of the theoretical one, drying is insufficient and the presence of liquid on the surface can cause agglomerations to form.

The theoretical value is obtained from the following equations. The first step is the determination of the absolute humidity at the inlet (w_{inlet}):

$$w_{\text{inlet}} = \frac{M_{\text{water}}}{\left(\frac{P_{\text{reactor}}}{\text{RH}_{\text{inlet}} P^{(s)}_{\text{waterinlet}}}\right) M_{\text{air}}}$$

where M_{water} and M_{air} are molar mass of water and of air, respectively; RH_{inlet}, represents the measured inlet relative humidity, and $P_{reactorinlet}$, the pressure on the reactor. In this study, the value of atmospheric pressure is used. $P^{(s)}_{water}$ is the saturated pressure of water at the inlet temperature.

The second step involves the mass balance equation, which makes it possible to determine the absolute humidity at the outlet (w_{outlet}):

$$w_{\text{outlet}} = \frac{Q_s}{Q_f} x_{\text{coating}} + w_{\text{inlet}}$$

where $x_{coating}$ is the water concentration of the coating solution (% w/w), Q_s the coating solution rate (g.min^{-1}) and Q_f the airflow rate (kg$_{dryair}$.min^{-1}).

The third and final step is the calculation of the theoretical relative humidity at the outlet :

$$\text{RH}_{\text{outlet}} = \frac{w_{\text{outet}} M_{\text{air}}}{w_{\text{outlet}} M_{\text{air}} + M_{\text{water}}} \frac{P_{\text{reactor}}}{P^{(s)}_{\text{wateroutlet}}}$$

The relative humidity for the two rates of airflow (Fig. 3) shows that when the airflow is reduced, the relative humidity clearly does not accompany the theoretical curve, although for the faster rate of flow, the curves are similar.

The pressure is maintained constant throughout the spraying and drying phases with a rapid airflow, but when this flow is reduced, the pressure decreases as long as spraying is continued, although it rises again when spraying is finished. These results corroborate the conclusions of El-Mafadi and Felipe et al. [9,10], who discuss the influence of pressure measurements on the movement of particles.

Fig. 4 represents the development of pressure as a function of airflow past the two sensors (DP_1 and DP_2). The coloured areas indicate variations of these pressure values for a given airflow. El-Mafadi [9] obtained similar results and interpreted the profile as consisting of four distinct zones. Although his experimental conditions were clearly different from those used here, results of both studies show that controlling the airflow limits the instable movement of the particles, i.e., fluctuations in pressure. In this study, fluctuations in pressure were monitored on the basis of the pressure inside of the Wurster tube (DP_2) and that in the annular region (DP_1). Four fluidisation zones were delimited: S1, S2, S3 and S4.

S1 is characterized by very low airflow rates, below 50 m^3.h^{-1}; in this area, the particles remain relatively static, and the value of DP_2 is close to zero, with limited variations in pressure. This is the result of the insufficiency of the airflow to induce particle movement. When the airflow increases, in S2, some particles will leave the tube and be transported to the annulus, but the velocity is not homogeneous and fluctuation at DP_1 and DP_2 are observed. In S3, at a flow rate between 100 and 150 m^3.h^{-1}, there is little fluctuation in pressure at either sensor, indicating that equilibrium has been reached and the circulation

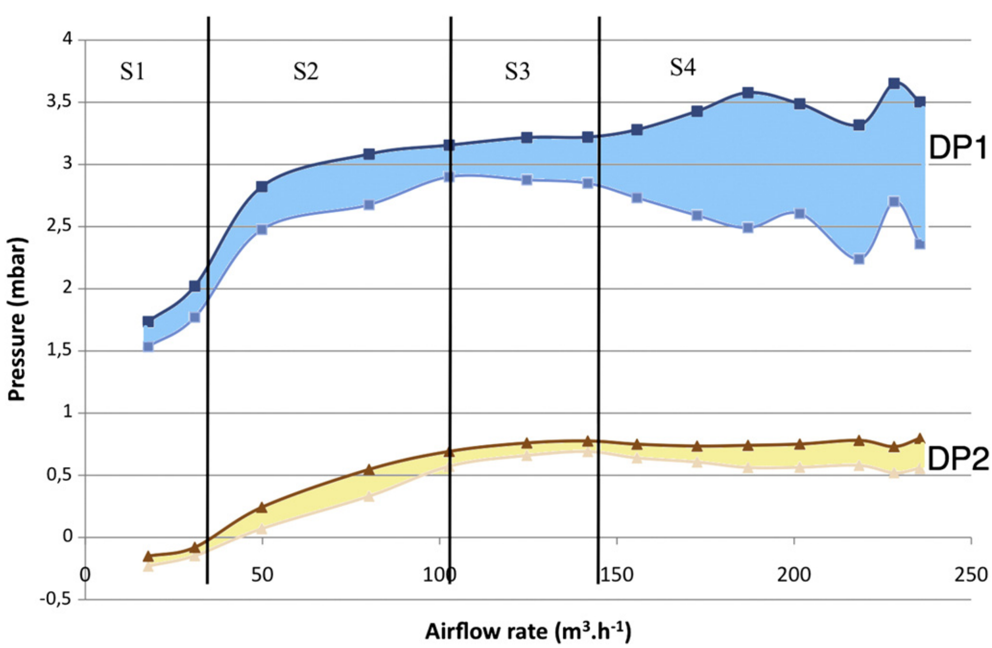

Fig. 4. Variation of the pressure DP_1 and DP_2 at the same airflow (coloured areas present variations between the minimal values (light colour) and maximal (dark colour)).

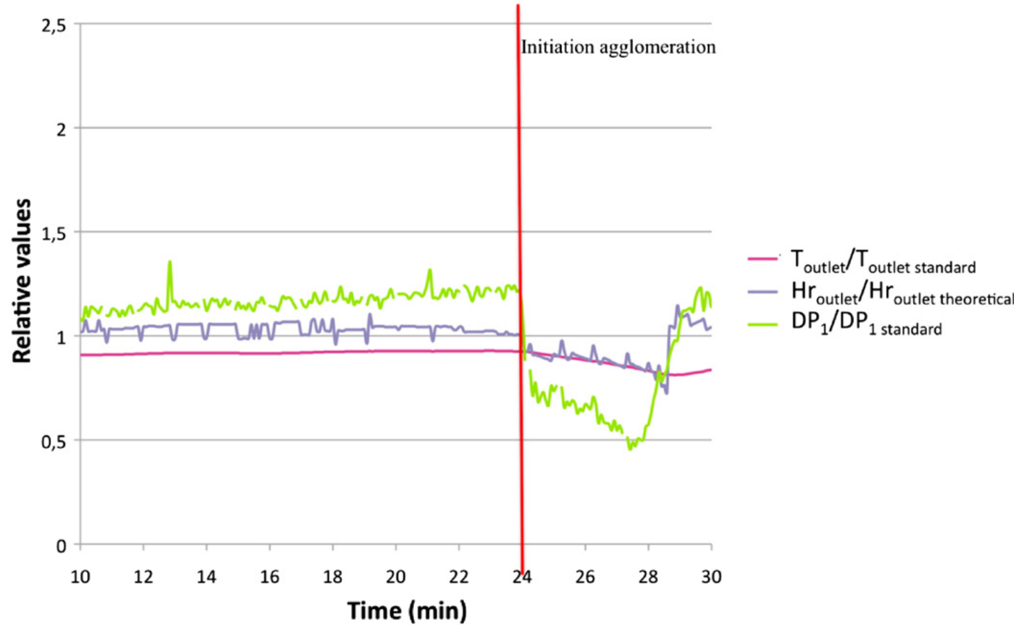

Fig. 5. Sensibility of the controlled parameters ($T_{outlet}/T_{outlet\ standard}$, $RH_{outlet}/RH_{outlet\ theoretical}$ and $DP_1/DP_{1standard}$) to detect agglomeration phenomenon.

of particles is relatively uniform. For very high airflows, fluctuations in pressure at DP_1 increase drastically.

Zone S3 provides the best conditions for the detection of agglomeration, because fluidisation is homogeneous, and pressure is relatively constant. Furthermore, the pressure at DP_2 shows the ascending movement inside of the inserted tube, although for detection of agglomeration, the pressure at DP_1 in the annular region is more sensitive. We propose to operate under these conditions, and to consider the behaviour the pressure at DP_1 to be an indicator of agglomeration.

Based on these experiments it was concluded that both relative humidity and pressure at the outlet vary upon initiation of agglomeration, and that both could serve as potential indicators of agglomeration. The final selection was thus based on sensitivity response of the two measurements that are reported in the next section.

The sensitivity of the two parameters, relative humidity and pressure, was evaluated by introducing a disturbance in the airflow so that agglomerations would form. Fig. 5 shows their response when airflow was decreased from 150 $m^3.h^{-1}$ to 50 $m^3.h^{-1}$, after 24 min. The parameters are presented as relative values to facilitate comparison of the three parameters.

This experiment confirms that the outlet temperature is not sensitive to agglomeration. On the other hand, the outlet relative humidity and pressure vary considerably once agglomeration starts. The pressure at DP_1 drops 0.5 units at this point, whereas the relative humidity decreases about 0.1 units, suggesting that the pressure is more sensitive to the appearance of agglomeration. Therefore the pressure at DP_1 was selected as the controlled variable.

3.2. Manipulated variable determination

The next step was the determination of a variable which, when manipulated, would be capable of maintaining the system at a set pressure at DP_1, so that once agglomeration is detected, it can be modified to return the system to the desired reference pressure. The choice of the variable to be manipulated depends on its influence on the system.

Since agglomeration is due to a temperature above that of the glass transition of the polymer being applied or the presence of high humidity in the fluidisation chamber, the manipulated variable must be related to the conditions of drying and/or atomisation. The main drying parameters are the airflow and the inlet temperature. Airflow, however, is easier to work with, because many products are extremely sensitive to high temperatures. Moreover, responses of the fluidised bed to change in temperature are slow, whereas an increase in airflow leads to a rapid increase in mass transfer by convection. Atomisation, on the other hand, is associated with the flow of the coating solution, as well as the pressure of atomisation. The flow of the coating solution affects the supply of water in the fluidised bed, whereas the pressure of atomisation influences the size of droplets.

These three parameters (airflow, atomisation pressure and coating solution flow) were evaluated in relation to the percentage of agglomeration and the amount of coating adhering to the particles. An experimental design with a total of 15 experiments based on the Box Benkhen approach, outlined in Tables 1 and 2, was used. A statistical model based on JMP software (SAS, USA) was used to determine surface responses, presented in Fig. 6, with the effects on the potential for agglomeration analysed.

The correlation coefficients obtained for the percentage of agglomeration and for the amount of adhering coating are 98.0 and 98.9%,

Table 1
Values for parameters in experimental design.

Factors	Units	Real values (coded values)		
		Low	Medium	High
Airflow: Q_f	$m^3.h^{-1}$	50 (−1)	100 (0)	150 (+1)
Atomisation pressure: P_p	Bar	1 (−1)	1,5 (0)	2 (+1)
Coating solution flow:Q_s	$g.min^{-1}$	6 (−1)	12 (0)	18 (+1)

Table 2
Experiments included in Box Behnken plan with encoded values as established in Table 1.

Experiment	Coded values			Experiment	Coded values		
	Q_f	P_p	Q_c		Q_f	P_p	Q_c
1	−1	−1	0	9	0	−1	−1
2	−1	+1	0	10	0	−1	+1
3	+1	−1	0	11	0	+1	−1
4	+1	+1	0	12	0	+1	+1
5	−1	0	−1	13	0	0	0
6	−1	0	+1	14	0	0	0
7	+1	0	−1	15	0	0	0
8	+1	0	+1				

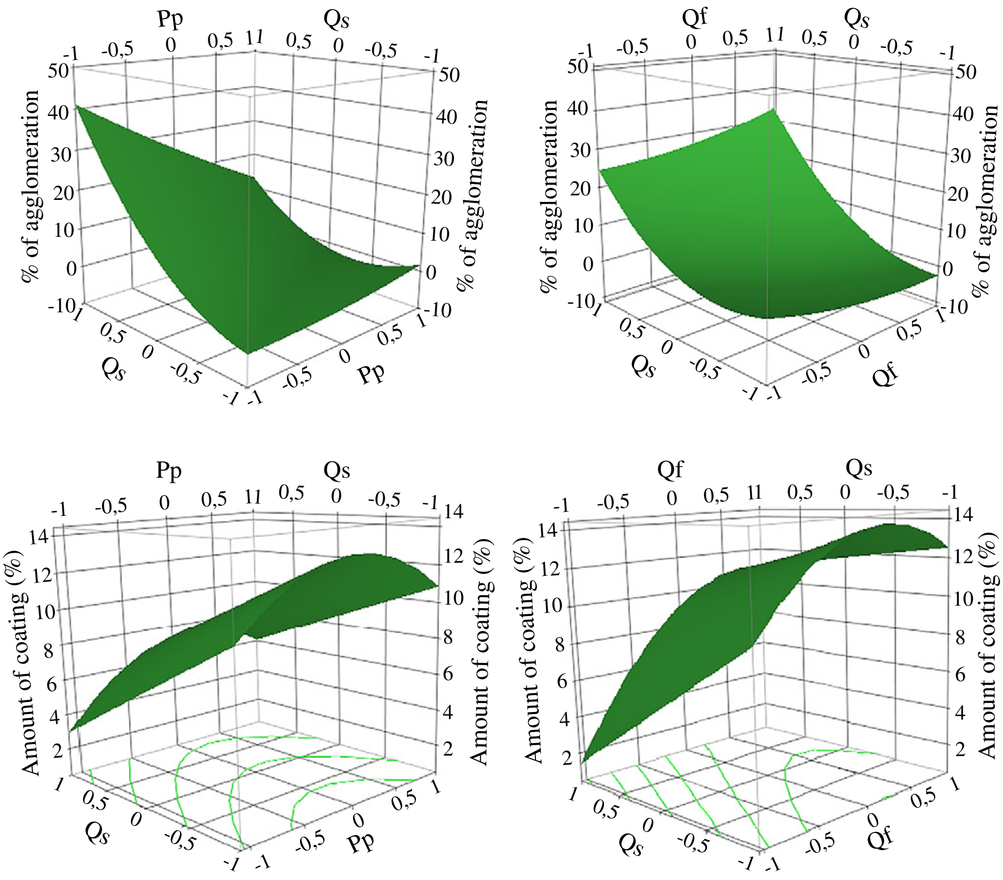

Fig. 6. Response surfaces showing effect of manipulated parameters of atomisation pressure (Pp), airflow (Qf) and flow of coating material (Qs) on percentage of agglomeration and the amount of coating deposited.

respectively. Fig. 6 shows that all these parameters influence the responses.

An increase in airflow leads to a greater potential for evaporation as well as decreasing the amount of water in the fluidisation chamber. This variable thus affects mainly the amount of coating deposited. Its action is crucial when the coating solution flow is maximal, as in this study. Airflow seems, however, to have less influence on the percentage of agglomeration than does coating solution flow.

An increase in atomisation pressure reduces the size of droplets and, consequently, improves the efficiency of drying, thus limiting the risk of agglomeration. Indeed, rapid drying of droplets reduces the percentage of agglomeration by 50% especially when the coating solution flow is maximal. However, too great an atomisation pressure leads to faster drying of the spray and a greatly reduced level of coating.

The flow of the coating solution is the only parameter that affects positively both coating level and percentage of agglomeration. Reducing the coating flow leads to a decrease in agglomeration, as well as the obtaining of the maximum amount of coating (12%). Moreover it is a key parameter in relation to time of processing and must to be used at optimal levels during the process to avoid reformation of agglomerates. It was thus chosen as the variable for manipulation for the closed-loop control presented in the following section.

3.3. Closed-loop control design

The control process is based on closed-loop control, with the action on the flow of coating solution designed to minimise the difference between measured pressure and reference value. Pressure was measured every 0.5 s, and the coating flow modified as a function of this value. The control strategy is very simple, and is based on the following principle: maximum and minimum values for pressure DP_1 (the controlled variable) are established so that the difference between them will be maintained within a specified range. If the value of pressure measurements are outside of the defined zone, the coating flow is switched to the lower level and when the pressure value returns to the defined zone, the coating flow is switched to the higher level. The final tuning of the controller is linked to the range between the minimal and maximal values (bandwidth) as well as the actual high and low settings for the coating flow (manipulated variable).

The selection of the bandwidth depends on a consideration of the fluctuation in pressure in zone S3 (Fig. 4). The bandwidth of the controller chosen here was between 2.9 and 3.2 mbar. For the coating flow, the minimal value was set at zero, whereas the maximum level was arbitrarily fixed at the maximum capacity of evaporation of the bed (18 g/min).

The adequacy of this strategy was evaluated for the maximum evaporative capacity of the bed by reproducing three sets of conditions that led to agglomeration in the original experiments (Table 3). The process of coating in each was followed to determine when agglomeration would begin and whether control of the manipulated variable would be sufficient to prevent its evolution.

Once the initiation of agglomeration was verified (by a drop in pressure at DP_1), the injection of the coating solution was paused until the pressure returned to the optimal range.

For each of the experiments, the percentage of agglomeration and actual amount of coating were determined after enough coating solution had been injected into the system to increase the particle weight by 20% (or until defluidisation).

The control strategy was able to avoid agglomeration for all three sets of conditions and was more efficient than manual observation.

Table 3
Comparison of results of control process and unmonitored procedure for sets of conditions leading to agglomeration.

Experiments	Q_f ($m^3.h^{-1}$)	P_p (bar)	Q_s ($g.min^{-1}$)	% of agglomeration		Coating level	
				Without control	With control	Without control	With control
A	150	1,5	18	37.0	0	10.1	11.4
B	100	1	18	42.9	0.2	5.4	11.4
C	50	1,5	18	21.2	23.5	2.6	4.0

Q_f: Airflow, P_P: Atomisation pressure, Q_S: Flow of coating solution.

Fig. 7. Pictures of coating particles without (left) and with (right) using automatic control process (Experiment B: $Q_f = 100$ $m^3 \cdot h^{-1}$, $P_p = 1$ bar, $Q_s = 18$ $g.min^{-1}$).

For the third experiment (C), the percentage of agglomeration was quite similar to that of the other two, but no emergency stop was required to avoid damages. However, this high percentage of agglomeration confirms that operating in zone S2 is not recommended. The level of coating, however, suggests that the use of the control strategy was more efficient than without control, especially with the conditions in B (Table 3): 100 $m^3.h^{-1}$, 1 bar atomisation pressure. Fig. 7 shows the difference in the coated particles when the automatic control process and no control are used for the same experiment B. It clearly shows that the batch obtained without automatic control could not be utilized due to the presence of large agglomerations and a heterogeneous coating. The right-hand illustration in Fig. 7 shows the batch obtained using the control process, with a homogeneous coating and separated particles.

4. Conclusion

The determination of the importance of a drop in pressure in the annular region as a signal for the onset of agglomeration makes it possible to intervene in the process by pausing the injection of the coating solution until the pressure has returned to an adequate level thus preventing the loss of entire batches due to agglomeration. Moreover, automated manipulation of the flow of coating makes it possible to maximize the efficiency of the operation by providing a more homogeneous coating, increasing the amount of coating adhering to the particles and reducing processing time.

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