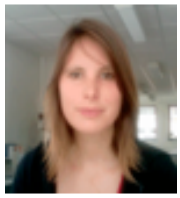
P-019

Control system with two control parameters to avoid agglomeration during coating in fluidised bed

Maudhuit A. [1#], **Prata A.S** [2], **Boillereaux L.** [1], **Poncelet D.** [1]*
[1] ONIRIS, Nantes, France [2] Unicamp - Limeira, Brasil
* Supervisor # audrey_maudhuit@yahoo.fr



INTRODUCTION AND OBJECTIVES

Particle fluidized bed coating is carried out to protect, structure or immobilize active compounds. The process consists in suspending particles in ascensional air stream, sprays on the particles a polymer solution, and by drying, leading to a coating around the particles. This process is largely developed in pharmaceutical and food industries. Despite its industrial importance, it is mainly still control manually and do not run in optimal conditions.

An important parameter for running correctly a fluid bed coating process is the moisture content in the reactor. Too much moisture content leads agglomeration phenomenon. Previous work in our laboratory showed that it was possible to follow the pressure drop in the fluidized bed to predict and then prevent agglomeration (Prata A.S 2011).

To optimize this method and reduce the process time, a relation more obvious between water in the reactor and an other control parameter is needed. To reach that, the capacity of evaporation is studied. After that, the idea is to introduce the evaporation capacity in the reactor as control parameter to regulate the aqueous coating solution flow.

MATERIAL AND METHODS

Cellulose microcrystalline beads (780 µm) (IPC, Germany) were performed. The coating suspension was an aqueous dispersion of gum Arabic or HPMC in distilled water (136 g/ L).

A fluid bed laboratory reactor (UNI-GLATT, Glatt, Binzen, Germany) equipped with the Wurster tube (diameter: 75 mm) is used. From the bottom, the coating solution is sprayed by a peristaltic pump through a two-fluid nozzle (SHLICK 970-S3, Germany). Pressures, temperature and relative humidity are collected inlet and outlet of the bed. Two thermo-hygrometers were used to measure relative humidity, respectively located at the inlet gas and at the top of the fluidization bed. To measure the pressure drops, differential pressure sensors (Delta_P, Halstrup Walcher, Germany) with a range of 0-12,5 mbar were installed (Figure 1). The airflow is fixed at 150 kg/h.

The closed loop of the process is carried out using the toolboxes Simulink and RTI software Matlab (MathWorks, USA), equipped with a card entry/exit ControlDesk (dSPACE GmbH). Pressure drops, relative humidities and temperatures are connected to the analogical entries of the capture card.

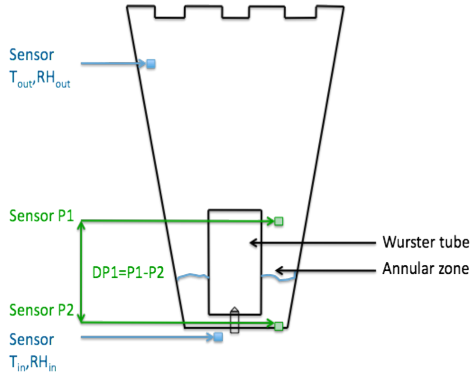


Figure 1: Measures for the automatic control process

RESULTS AND DISCUSSION

Psychrometric chart

The psychrometric chart allows determining drying processes in terms of evaporation capacity and energy. To evaporate water, a low moisture content is introduced on the reactor with the inlet airflow (for instance, A in Figure 2). The moisture content in the outlet airflow is higher due to the evaporation of the water presented on the coating solution (B). The evaporation capacity is the difference between inlet (A) and oulet (B) moisture contents (Δna) multiply by the airflow. The evaporation is realized without energy: the specific enthalpy is constant. Δna is determined in the psychrometric chart following the specific enthalpy line, knowing the T_{in} and HR_{in} (Figure 2).

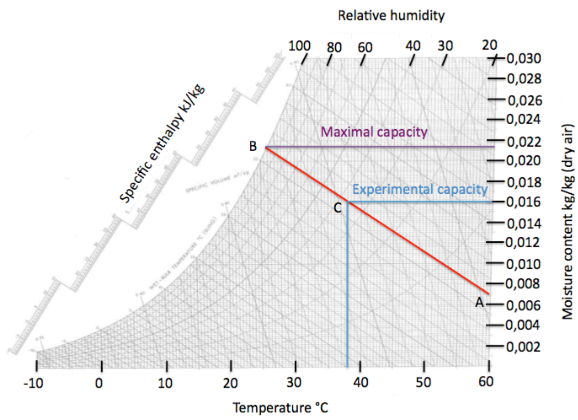


Figure 2: Psychrometric chart

Theoretically, the maximal evaporable capacity is reached at 100% of relative humidity (B). However, in such conditions, particles remain too wet and agglomer-

ation takes place. That is why a lot of processes work with an exit air around 20-30% HR, leading to lower evaporation capacity and then longer process duration.
To optimize the process, the next paragraph studies the real evaporation capacity before agglomeration apparition.

Determination of the capacity of evaporation

Assuming feeding the reactor with air at 5% relative humidity at 60°C and an air leaving the reactor at 100% of relative humidity (in iso-enthalpic conditions), the maximal difference between moisture contents is around 15 g water/kg airflow*150 kg airflow/h, i.e. 2.25 kg water/h (A and B, Figure 2).

The maximal quantity of evaporable water without agglomeration is determined using the pressure drop (DP1). Previous work showed that high-pressure drop fluctuations indicated agglomeration formation (Prata A.S, 2011). The coating solution flow is linearly increased until agglomeration is detected with the pressure drop fluctuation. In this study, the experimental evaporation capacity for Arabic gum and HPMC is around 9 g water/kg dry air*150 kg arflow/h i.e. 1.35kg/h (C in Figure 2). So it is possible to work at 60% of a theoretical quantity i.e. in our conditions until 35%HR.

These first experiments allow knowing that is possible to work at 60% of the theoretical water evaporable capacity (i.e. our conditions 1.35kg/h). This value could be introduced in a control process to regulate the coating solution flow to always have 1.35kg of water/h in the reactor.

Knowing this value, the evaporation capacity (Δna^*Q_f) could be an indicator to the beginning of agglomeration and could be introduced in the control system. A safety interval is defined at 80% reducing the evaporation capacity to 1.08 kg water/h for the following study.

Conception of the double automatic control system

The evaporation capacity of 1.08 kg water/h and the fluctuation of the pressure drop are both integrated as control parameters to avoid agglomeration. They simultaneously work and confirm the agglomeration event.
The real time measurement of the capacity of evaporation (Δna^*Qf) is calculated with inlet and outlet temperatures and relative humidities following this equation:

$$\Delta na_{real} * Q_f = \left(na_{out} - na_{in}\right)Q_f = \frac{M_{water}Q_f}{M_{air}}\left(\frac{1}{\left(\frac{P_{reactor}}{RH_{out}P^{(s)}_{water}(T_{out})} - 1\right)} - \frac{1}{\left(\frac{P_{reactor}}{RH_{in}P^{(s)}_{water}(T_{in})}\right)}\right)$$

M_{water} and M_{air} are respectively molar mass of water and air and $P_{reactor}$ is the pressure in the reactor, and on this study, the value of atmospheric is used. $P^{(s)}_{water}$ is the saturated pressure of water.
The pump regulation allows to reach the Δna^*Qf value to 1.08 kg water/h. The Figure 3 summarizes the new control system.

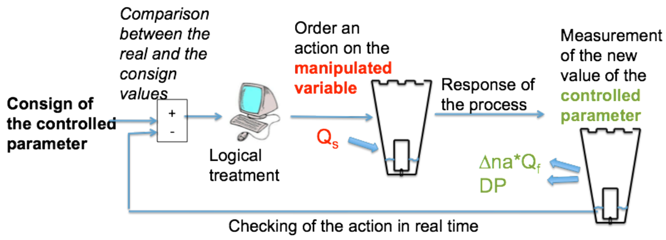


Figure 3: Schematization of the control process

Validation of the control process with two control parameters

The efficiency of this control process is evaluated by comparison with and without using the control process. For that, a disturbance is introduced in the inlet temperature. At 30 minutes of coating process, the heating is stopped during 15 minutes. This procedure is carried out for Arabic gum and HPMC. The Figure 4 shows the percentage of agglomeration obtained in all cases.

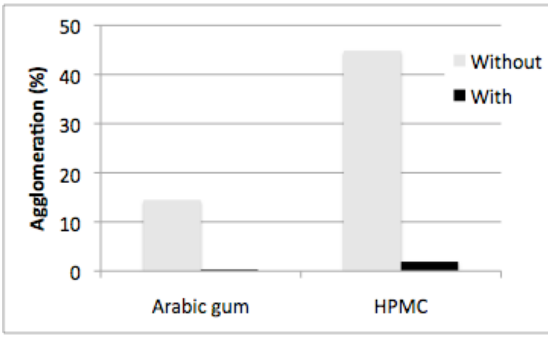


Figure 4: Percentage of agglomeration without and with double control process

It seems clear that the use of the control process allows to reduce considerably the quantity of agglomerates. In the case of Arabic gum, no agglomeration is observed. In the case of HMPC, with the control system, agglomerates contain 2 or 3 particles, whereas, without the control system, agglomerates diameters are bigger than 2 mm.

CONCLUSIONS

On this work, the evaluation of experimental evaporation capacity allows to work at 60% of the maximal capacity (i.e. saturation). This study also shows the possibility to avoid the agglomeration event with two control parameters: evaporation capacity and pressure drop. These first results are very encouraging and will be certainly validated by new comparisons with the use or not of the control system. More validation will be carried out to develop this system at industrial scale.

REFERENCES

- Prata A.S. et al. (2011) *Anticipation of agglomeration in fluidized bed coating process by control system* in *5th International Granulation Workshop* (Granulation Conference, 20-22 june 2011, Lausanne, Switzerland) p212-213.