Effects of various process parameters on fluidized bed particle coating

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INTRODUCTION AND OBJECTIVE

In fluidized bed particle coating processes, many physicochemical phenomena occur simultaneously within various spatial and time scales (Lettieri et al 2009). Therefore it is hard to evaluate the net impact of the process parameters on the system's efficiency and product. In the present study, we perform a parametric study to observe the effects of inlet air flow rate, inlet air temperature and material properties, on the process yield of a batch bottom spray coating system, and to find optimal operating parameters among these variables.

MATERIALS AND METHODS

Materials

3 types of maltodextrins were purchased (DE6, DE12 and DE19) were purchased from Roquette (France). Coating experiments were performed on 500 µm mean diameter inert particles. The oil used in the emulsions is a mixture of essential oils(confidential).

Maltodextrin samples were kept in desiccators with saturated solutions of salts with different water activities. The samples were weighed regularly until constant weight change was reached and their final water content % was measured. The glass transition values corresponding to the respective samples with various water content % were measured via DSC (Q100-TA – 5 °C/min ramp, 2 cycles of heating).

Preparation of Coating Emulsions

The emulsions consist of 20% oil 30% maltodextrin 50% water w/w. Maltodextrin was dissolved in water and oil was added to this solution. The emulsions were prepared mixing the aqueous and oil phases by a high-speed disperser (Ultra-Turrax, IKA T25) at 20000 rpm for 5 minutes.

Coating of Particles

The coating experiments were performed with a lab-scale, bottom-spray fluidized bed (Uni-Glatt, Germany). The pressure used to pulverize the coating emulsion was 1.5 bar and the spray was fed to the reactor at a rate of 10 g/min.

As shown in figure 1, hot and dry inlet air is supplied to the reactor through a perforated bottom plate, causing a circulating motion of particles. Meanwhile, the coating emulsion is pumped into the reactor by a peristaltic pump and it is sprayed on the particles inside the reactor by a pneumatic nozzle. The droplets deposited on particles are then dried by means of the airflow and a layer of coating is formed. Particle agglomeration and spray drying are two main problems encountered in fluidized bed coating processes. The extent of these problems is strongly related to the material characteristics such as glass transition temperature and the rate of drying in the system. Therefore the process parameters studied in this work were chosen to reflect these aspects.

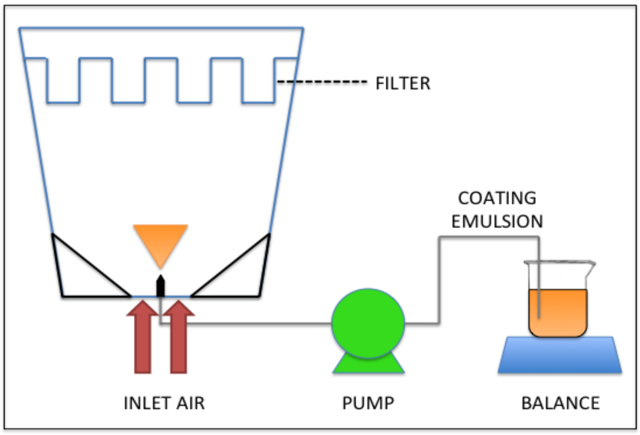

Figure 1: Coating Setup

To determine the effects of process parameters on the coating yield a Box-Behnken experimental design for 3 factors / 3 levels (Table 1) was performed.

Table 1 : Experimental Design Parameters

Factor	Level		
	-1	0	+1
Inlet temperature (°C)	48	54	60
Air flow rate (m^3/hr)	100	140	180
Maltodextrin type (DE number)	6	12	19

At the end of the experiments, the initial mass of the particles was subtracted from the final mass of the particles inside the reactor. The ratio of this value to the theoretical maximum dry material in the coating solution gives the yield of coating.

RESULTS AND DISCUSSION

The response surface statistics obtained through the experiments are as given in table 2. As seen in the table, material properties (DE) has a strong impact on the process yield while the other 2 factors, their interactions or their 2nd order terms s do not affect the response significantly within given factor levels.

Additionally, the R^2 value of 0,82 implies a reasonable fit for these findings. However, large relative standard errors are observed for all statistical terms except DE.

Table 2. RSM statistics for the parameters

Term	Estimate	t Ratio	Prob>|t|
DE	5,05±1,34		0,0130*
T	-2,75±1,34		0,0953
T*T	2,99±1,97		0,1902
DE*T	1,98±1,90		0,3450
Qf	-1,25±1,34		0,3936
DE*Qf	1,08±1,89		0,5950
Qf*Qf	0,74±1,97		0,7238
DE*DE	0,69±1,97		0,7416
T*Qf	0,03±1,89		0,9900

Process yield prediction profiles for each parameter are given in figure 2. The yield, for any given parameter, does not exceed the level of 85,8%. Visual confirmation of the product powder after each experiment was done and no agglomeration was observed in the coating processes. Therefore the loss of 14,2% material loss is attributed to spray drying of the coating material and evaporation of the essential oil from the emulsion. However, the losses from these sources could not be calculated separately.

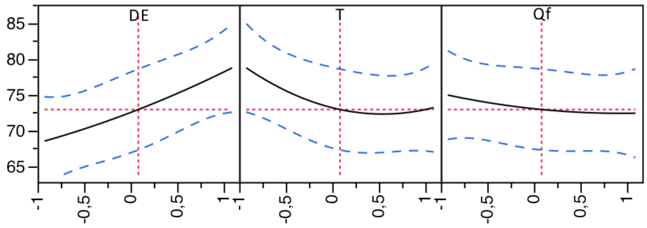

Figure 2. Prediction profiles vs % material yield

The rate of drying in a fluidized bed depends on the properties of the inlet air. It should be sufficiently low to allow coating droplets reach the particles and sufficiently high to prevent particle agglomeration. Clearly, within the given range of airflow rates and inlet temperatures, the drying rate of the system is too high, such that the coating droplets are spray dried. Additionally, the same trend is observed in figure 2 when the temperature and airflow rate are increased.

Maltodextrins are obtained by hydrolysis of starch syrups. The DE number depends on the extent of hydrolysis. Low DE maltodextrins have longer glucose chains and higher molecular weights resulting in a higher T_g at given water content level. When an amorphous coating droplet cools down it passes through a “sticky zone”. When passing through these conditions the viscosity of the coating solution significantly increases and the coating becomes adhesive (Gianfrancesco 2009). This change allows the coating solution to stick to the surface of the particles. However, if the coating solution remains long enough in sticky conditions, particle agglomeration will occur due to enhanced adhesivity.

The glass transition vs the water content curves for 3 types of maltodextrins used in the study are given in figure 3. The coating material is transformed from a liquid droplet to a solid deposited on the particle surface from point A to point E.

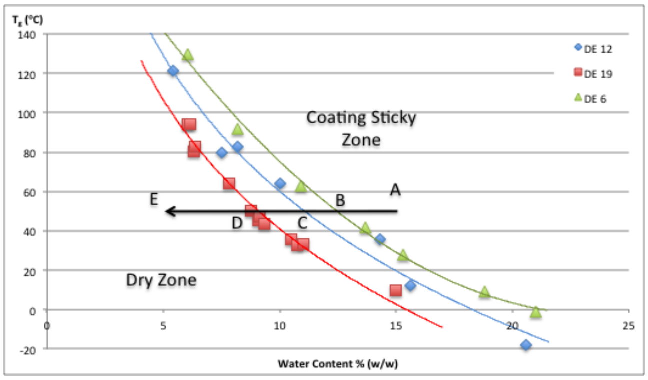

Figure 3. T_g vs water content %(w/w)

A coating material with a high glass transition temperature allows operating on a higher inlet temperature decreasing process time. On the other hand, if a fast drying is supplied in the system, the droplets will pass through the sticky zone, and will not attach to the particle surface easily. If we consider the line AE in figure 3 as a time scale, the segments AB, AC, AD represent the time it takes to cross the conditions where the coating solutions are sticky. Clearly, as the time required to dry the coating droplets increases, a higher material yield as pointed out in figure 2 is obtained. It should be noted however, to determine the true extent of the effect of the difference in DE values on the process yield, additional material characterization tests (ex: emulsion stability) should be performed.

CONCLUSION

A maximum material yield of 85.8% within the given experimental design parameters, point to a high drying rate provided by the airflow and inlet temperature. Material properties were found to be the only statistically significant parameter in the response surface study. Further physicochemical characterization of the coating emulsion is required to verify the extent of the impact of glass transition on coating material yield.

REFERENCES

- Gianfrancesco A. (2009) *Spray drying engineering : particle stickiness in relation with agglomeration*. Agro Paris Tech, Thesis Dissertation.
- Lettieri et al (2009) *Challenges and Issues on the CFD Modeling of Fluidized Beds: a Review*. The Journal of Computational Multiphase Flows 1(2) 83-131