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Spectral Analysis of Pressure Drop Fluctuation in Vibrofluidized Bed Coating

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Fluidized beds are indicated in the literature as the most appropriate equipment for fine particle coating. However, recent findings and the industrial practice show that the high moisture content of the bed during a coating process results in fluid dynamic instabilities and even leads to partial or total collapse of the bed. The employment of mechanical vibration to the fluidized bed has resulted in significant improvement of the process performance when working with fine and cohesive particles that are difficult to fluidize. In the present study, a vibrofluidized bed (VFB) was used to coat sodium bicarbonate with a polymeric suspension. The main objective of this work was to use spectral analysis of pressure fluctuations signals to identify fluid dynamic alterations during the VFB coating. The principal visually observed fluid dynamic regimes during the process were: fixed bed, heavy bed with low particle circulation rate, and partly dry bed with high circulation rate. Loads of 450 g of sodium bicarbonate were used and the process parameters were varied in the following ranges: vibration amplitude and frequency up to 0.02 m and 360 rpm, respectively, and air temperature from 60 to 80ºC. The coating experiments were carried out monitoring in real time the pressure fluctuations signals, at a 400 Hz sample rate using LabVIEW® 6.0 software. Behavior of the pressure spectrums was evaluated applying spectral analysis that showed changes with the fluid dynamic regime of the bed. The results showed the viability of spectral analysis to identify fluid dynamic regimes in vibrofluidized bed coating.

1. Introduction

According to the literature, the most appropriate equipment used for fine particle coating is the fluidized bed (ZANK *et al.*, 2001, CUNHA *et al.*, 2009). In order to improve the fluidization of fine particles, an important possibility is the application of mechanical vibration to the bed, resulting in a vibrofluidized (VFB). The VFB assists in fluidization of particles that present poor contact in conventional fluidized beds, such as materials with wide size distribution, cohesive, clusters, thermoplastics and pastes. The mechanical vibration dominates the interparticle attraction forces, increasing the quality of fluidization of fine particles that tend to cluster. Other advantages of working with the VFB are: mechanical vibration causes a reduction in the amount of air needed for

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fluidization, resulting in lower energy consumption; fluidization is homogeneous, even for binder materials, as the vibration causes breakage of aggregates and reduction of preferential channels; and the product is not subjected to high attrition and shocks as in the conventional bed (GUPTA and MUJUMDAR, 1980;). VFB is applied in industrial drying, heating, cooling and catalytic reactions (MUJUMDAR and ERDEZS, 1988). The application of film coating to a variety of fine particles (chemicals, food and pharmaceuticals) has shown more and more interest to modify the surface properties of the particle or to decrease the material reactivity with the surroundings, such as light, oxygen, water vapor, among others.
The main objective of this work was to use spectral analysis of pressure fluctuations signals to identify fluid dynamic alterations during the VFB coating.

2. Materials and Methods

2.1 Material

Powder of sodium bicarbonate from Synth® was the material chosen to conduct this work, for its characteristics of fine powder with large industrial utility. The powder presents poor or even impossible process condition in conventional fluidized bed, mainly in a coating process, due to its characteristics of fine powder with wide size distribution and to the high moisture inside the bed. Thus, modifications of the conventional bed mode are necessary to insure a good quality fluidization.

2.2 Coating suspension

In this work, coating is obtained by the atomization of a polymeric suspension (NUNES *et al.,* 2010) on the sodium bicarbonate particles that are vibrofluidized by hot air. The air acts also as the drying agent of the suspension on the particle surface. This polymeric coating suspension formulation was developed by Donida *et al.* (2007).

2.3 Particle characterization

The particles were physically characterized before and after coating by: mean diameter, using granulometric analysis in standard sieves and flowability index (FI), obtained by Powder Flowability Test Instrument - Flodex®. This measurement indicates the flowing characteristic of the powder.

2.4 Experimental setup

The coating experiments were carried out in a vibrofluidized bed system. Details of the experimental setup can be found in NUNES *et al.* (2010).

2.5 Experimental Procedure

The bed was loaded with 450 g of powder and the amplitude (a) and frequency (f) of vibration and temperature (T) were fixed and stabilized for each experiment. Flow rate of fluidizing air was adjusted to 0,07 m/s. Next, the coating suspension was fed up at 2 ml/min and at the same time, the atomizing pressure was set to 10 psi. A stopwatch was immediately turned on to count the time of spraying, which was set in 10 min for each experiment. During the tests, the bed dynamics was monitored through a data acquisition system in order to determine instability and the tendency of the bed to saturation.

The process performance was analyzed by evaluation of particle growth and flowability index. Particle growth (δ) was evaluated according to Equation (1):

$$\delta=\frac{d_{s_f}-d_{s_i}}{d_{s_i}} \tag{1}$$

2.6 Spectral analysis methodology

Several methods have been developed for the quantification of fluidization regimes depending on the need to evaluate when the quality of movement of particles is affected by defluidization. Signal pressure fluctuations have been used to identify the defluidization moment and agglomeration of particles in several applications. In the literature there are few studies regarding the monitoring of fluidization regimes during the course of coating particles to prevent the defluidization phenomenon (Parise et al., 2010).

Pressure fluctuation in the frequency domain, known as spectral analysis, has been widely applied to investigate the quality of particles motion in fluidization processes (Trnka *et al*., 2000; Johnsson *et al*., 2000). This analysis can detect fluidization regime transitions through the changes in the spectral amplitude and frequency.

To build the pressure spectrum used to monitor the vibrofluidized coating process, initially 8192 (2^{13}) pressure data points were collected in the plenum chamber at a sampling rate of 400 Hz. The pressure signal was registered by a PCI 6025 (National Instrument) data acquisition system. The software LabView 6.0® was used for the acquisition and processing of the pressure signal.

The pressure signal collected was filtered by a digital filter (IRR - infinite impulse response - Butterworth type) and then normalized.

A Fast Fourier Transform algorithm (FFT) was then applied to the normalized pressure signal, producing a pressure spectrum.

3. Results

3.1 Particles Characterization

By Granulometric distribution, the Sauter mean diameter of the raw particles was of 75 µm, showing that the sodium bicarbonate is a very fine powder. The flowability index observed was of 20 mm (meaning that the particles freely passes through a 20 mm orifice), which indicates good flowing.

3.2 Coating Process

During the experimental development, the influence of the operational conditions both on the fluid dynamics and on the coating process was observed. In some tests, it was not possible to reach the pre-established total spraying time continuously. Intermittences of the coating suspension feeding had to be applied until the dynamic stability restore.

Table 1 shows some results of particle growth and flowability index after coating as a function of some operational conditions.

Table 1: Results of growth and flowability index after coating.

a (cm)	f (rpm)	T (ºC)	δ (%)	FI (mm)
0.40	270	70	26,51	4
0.75	220	75	22.80	4
0.75	320	65	20.95	18
1.25	186	70	9.88	4
1.25	270	70	44.50	8
1.25	354	70	7.79	9
1.75	220	65	25.72	9
1.75	320	75	6.69	10
2.00	270	70	7.83	9

The results show that both the growth and flowability index were influenced by frequency and amplitude of vibration and temperature. This result is an indication of a conjoint effect of the independent variables on the dependent ones (Nunes *et al.,* 2010). Lower flowability index were obtained for the coated particles compared to the sodium bicarbonate without coating for all different operating conditions, showing better flowing characteristic of the powder after coating. It is worth emphasizing that the lower the values for this property, the better the powder flowing.

Growth of particles was observed in all cases, and for the condition of intermediate amplitude, frequency and temperature (1.25 cm, 270 rpm and 70 °C), growth was more pronounced. Also, a low flowability index was obtained for this condition, indicating it as the most suitable to conduct the sodium bicarbonate coating.

3.3 Spectral analysis

Pressure spectra for the coating experiments were evaluated to identify the moment of defluidization of the bed. Since the behavior of the power spectra was similar in all experiments (Table 1), only the results of the experiment with average amplitude, frequency and temperature (1.25 cm, 270 rpm and 70 °C) will be discussed here.

The coating experiment began at bubbling stable fluidization condition. During the process, moisture excess in the bed led to dynamic instabilities. This situation happens commonly in chemical plants and in laboratory, reducing the efficiency of the process.

Figures 1 a, b and c show the evolution of the pressure spectra at different times of the coating process. It can be seen in Figure 1 a, a pressure spectrum characteristic of a bubbling fluidized bed, with the distribution of spectral amplitudes resembling a Gaussian curve. In this case, the spectrum was obtained without the atomization of the coating suspension. Figure 1 b exhibits the initial moments of liquid atomization to the particles. One can observe changes in the spectrum, which reflect a disturbance that occur in the fluidization regime as a function of atomization. At these moments, visual observations showed a decrease of the bubbles and of the movement in the bed. In Figure 1 c, the collapse of the bed was identified because of the significant decrease of spectral amplitudes. As sodium bicarbonate is a very fine powder, the coating process was carried out intermittently. Three intermittent cycles were performed.

Figures 2 a, b and c show the pressure spectra obtained at the third cycle of atomization of the coating suspension and the result is very similar pressure spectrum to the first atomization cycle. Changes in the spectra can be observed.

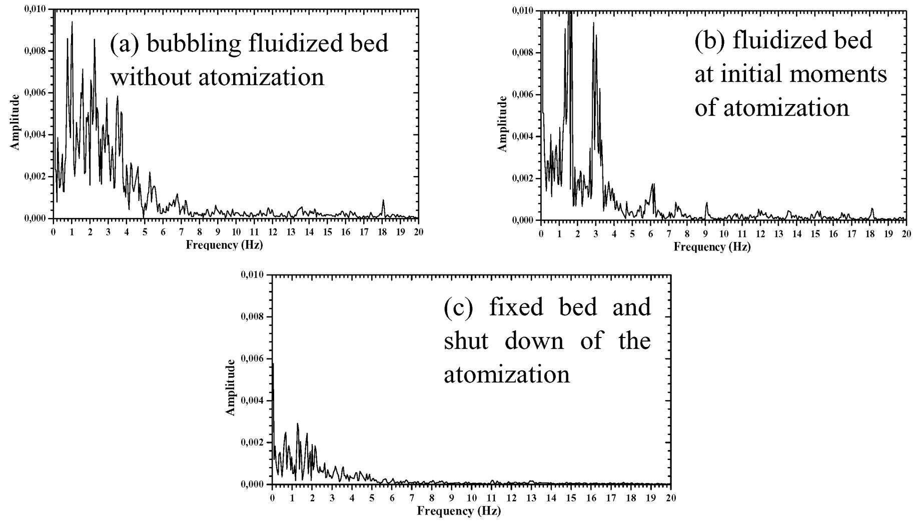

Figure 1: Behavior of the pressure spectrum during the coating process. First cycle of atomization.

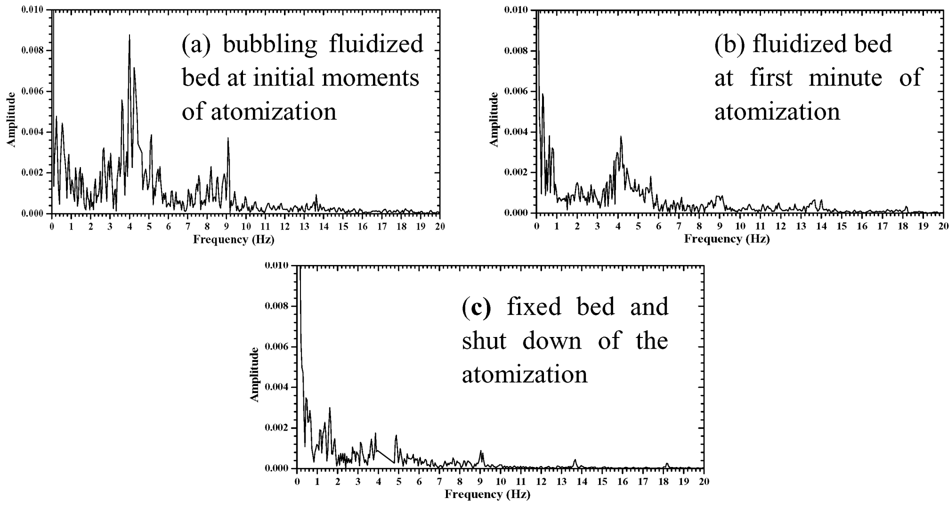

Figure 2: Behavior of the pressure spectrum during the coating process. Third cycle of atomization.

4. Conclusions

The use of the VFB was effective on conducting the coating process of sodium bicarbonate.

Coating of individual particles was obtained, without agglomerates formation. Particle growth was in the range of 6.69 to 44.50%, depending on the process conditions.

The flowability index were lower for the coated particle compared to the sodium bicarbonate without coating for all different operating conditions, showing better flowing characteristic of the powder after coating.

The spectral analysis allowed monitoring changes in the fluid dynamic regime in real time.

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