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Fluidised bed drying of powdered materials: Effects of operating conditions

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ABSTRACT

The fluidized bed drying of powdered materials was studied with a focus on the modifications of the agglomerate population as a result of abrasion and fragmentation. A number of test were carried out in a Lexan® lab-scale fluidised bed with different granular materials, namely a pharmaceutical powder supplied by Novartis-Farma S.p.A., wheat bran, and a molecular sieve 13X, selected to effectively surrogate powders of interest in various industrial applications such as pharmaceuticals pills and adsorption pellets manufacture. The process was monitored to correlate the temperature and the flow rate of the fluidising gas, the temperature and the moisture level in the bed, the qualitative fluidisation patterns. Experimental results were further worked out to highlight the effects of inlet air temperature and gas velocity on process thermal efficiency. Bed material was characterized to assess the modifications of the population of agglomerates as a function of the operating conditions.

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1. Introduction

Wet granulation is a unit operation frequently used in a number of industrial applications, from food to pharmaceutical industries. Larger granules of primary particles are commonly produced by high-shear granulation mixers wherein wetted powder particles are mixed, agglomerated, and densified under the action of shear imposed by impellers. Wet granulation can be seen as a combination of several series/parallel processes as defined by Iveson et al. [1]:

- *wetting and nucleation*, where the liquid phase (binder) is brought into contact with the original powder and liquid bridges formed among contacting particles give rise to strong-dense granules (micro-aggregates);
- *consolidation and growth*, where plastic collisions among micro-aggregates lead to denser macro-aggregates (granules);
- *attrition and breakage*, where granules diameter decreases as a consequence of wearing (attrition) or structure collapse due to impacts (breakage).

The recognition of the role played by granulator design and granulation conditions on the attributes of the produced granules has been extensively addressed in the literature [1–14].

Granulation phase is often followed by a drying stage carried out in gas-solid fluidized beds operated as continuous or batch devices [15]. In batch drying operations, the dryer is charged with wet material and the flow of the drying medium is started. Once the material is sufficiently dry, flow is stopped and solids are discharged. In continuous dryer, both the wet and the dry material streams are continuously fed/removed to/from the unit. Obviously, unit outlet is a mixture of solids characterized by features distribution close to that of a continuous stirred tank. Batch operations are commonly applied to sensitive materials such as pharmaceuticals, given that homogenous batches can be easily set apart if quality standards are not met [16].

Despite the long history of research on fluidized bed drying of wet materials [15–23], to authors' knowledge there is still a lack of understanding on the complex interplay of phenomena active during the drying stage, when abrasion and fragmentation of solid particles are active and affect size distribution and morphology of agglomerates. The effects of key variables - superficial velocity (U_g) and temperature (T_{IN}) of the fluidizing gas - on produced granules is poorly characterized.

The present study addresses the fluidized bed drying of powdered materials. The attention was focused on both the characterization of the population of agglomerates as a function of the operating conditions and the thermal efficiency of the process. The experimental tests were carried out in a Lexan® lab-scale fluidised bed to assess the modifications of the population of agglomerates as a function of the operating conditions. Data were processed to correlate the temperature and the flow rate of the fluidising gas, the temperature and the moisture level in the bed, and the qualitative fluidisation patterns. The analysis of the effects of inlet air temperature and gas velocity on thermal efficiency

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was also proposed to provide energy savings and optimum processing conditions.

2. Experimental

2.1. Apparatus

The dryer fluidised bed apparatus used for the tests is represented in Fig. 1 and basically consisted of: a lab-scale fluidization column; a gas-drying feeding system; diagnostic devices and an acquisition unit.

The lab-scale fluidization column was made of Lexan® and was of two sections: a conical fluidisation chamber (ID_{min} = 0.11 m; ID_{max} = 0.14 m; height = 0.10 m) and a freeboard (I.D. = 0.14 m; height = 1 m). The fluidisation column was equipped with a purposely designed gas distributor, a perforated plate with 110 holes ($d_h = 2 \times 10^{-3}$ m) laid out on seven concentric circumferences and inclined at 45° with respect to the horizontal plane.

The gas-drying feeding system consisted of a mass flow controller and of a 2.5 kW tubular air heater (I.D. = 0.1 m; height = 1 m).

Several diagnostic devices were used to continuously measure the pressure at the bottom of the bed, the temperature profile along the plant and the humidity of the exhaust gas stream. Pressure was measured by means of a Druck resistive pressure transducer (mod. PDCR130). Temperature was measured by means of T-type thermocouples. Gas relative humidity was measured by means of a Hanna-Instruments hygrometer (mod. HI8064). Uncertainties of pressure, temperature and moisture measurements are evaluated as 0.05%, 0.25% and 1.5%, respectively. All signals were logged on a data acquisition unit consisting of a PC equipped with a data acquisition board (NI-ATI-MIO16X).

2.2. Materials

Three granular materials were investigated: a pharmaceutical powder (PP) supplied by Novartis-Farma S.p.A., wheat bran (WB), and a molecular sieve 13X (ZX). Table 1 reports main properties of the granular materials used in the experiments. Solids are characterized by different densities, Sauter mean diameters and angles of repose. In order to accomplish the granulation phase, WB was pre-stirred in a high-shear mixer with an aqueous solution of HPM-Cellulose; ZX granulation was accomplished in the same high-shear unit using an aqueous solution of Kaolin as binder. PP was from the on-line sampling port of a production unit. The solids were sampled just after the granulation step and collected in twist-seal bags.

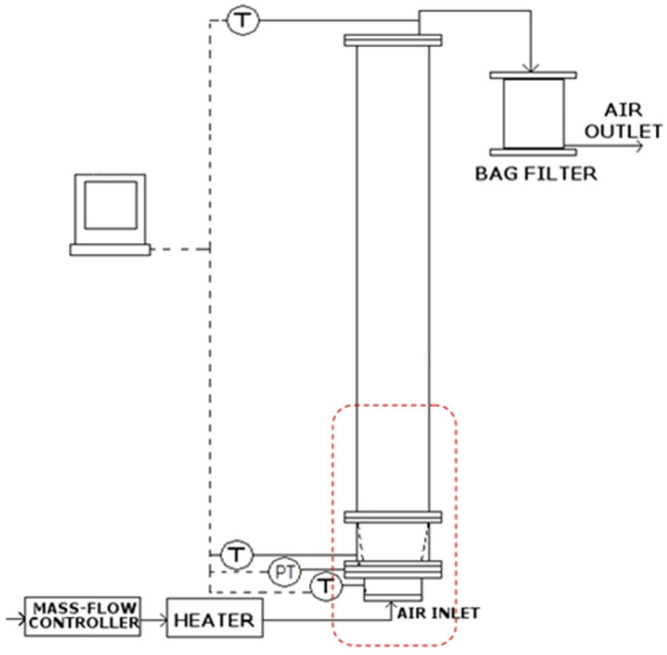

Fig. 1. Experimental apparatus. T - temperature; PT - pressure transducer. The red dashed line marks the control volume of the energy balance. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Table 1
Properties of the granular solids investigated. (1) According to [24]. (2) According to [25].

Acronym	WB	ZX	PP
Material	Wheat bran	Molecular sieve	Pharmaceutics
Sauter mean diameter (d_p), μm	50	15	550
Initial size range, μm	30–50	10–20	200–700
Particle density (ρ_s), kg/m^3	440	2140	1600
Incipient fluidization velocity (U_{mf}) (1), m/s	5×10^{-4}	2×10^{-4}	1.4×10^{-1}
Terminal velocity (U_t) (2), m/s	3×10^{-2}	1×10^{-2}	3.16×10^{0}
Angle of repose, °	35	55	55

2.3. Operating conditions and procedure

Solids inventory was 0.2 kg in all tests. The inlet temperature of fluidising gas (T_{IN}) ranged between 40 and 60 °C. Superficial gas velocity (U_g) ranged between 0.12 and 0.24 m/s for PP and WB, between 0.12 and 0.35 m/s for ZX. For any fixed value of U_g and T_{IN}, ten runs were performed and it was verified that the majority of experimental points were within the ±2.5% error. Each run was characterized in terms of pressure and temperature at the bottom of the bed of solids (T_{bed}), and relative humidity of the outlet gas stream (rH). Data were collected until rH approaches:

- A minimum required level (20%) in tests carried out on PP and WB (as required in most pharmaceuticals manufacture);
- its final value (rH_{res}) after a fixed amount of time (7200 s) in tests carried out on ZX, as in most continuous pellets manufactures.

At the end of each run, fluidising gas was shut off and solids in fluidised bed were collected. Solids were characterized in terms of granular size distribution by means of a Malvern Instruments laser-light-particle-size analyser. Measured data were worked out to correlate temperature and superficial gas velocity of the fluidising gas, drying time, Sauter mean diameter (d_{SM}) of the granules, and thermal efficiency of the drying process.

3. Results

3.1. Relative humidity

Fig. 2 reports values of relative humidity measured during runs carried out with PP. In particular, Fig. 2A refers to runs carried out at T_{IN} = 60 °C for different values of U_g, Fig. 2B to runs carried out at U_g = 0.18 m/s for different values of T_{IN}. Relative humidity decreased with time under all the operating conditions investigated, while the dynamic of drying regime changed with the operating conditions. The analysis of the rH vs. t plots in Fig. 2 pointed out the following issues:

- For a set value of T_{IN} (Fig. 2A), the drying regime remarkably changed with U_g. At the lowest value of U_g (0.12 m/s) investigated, two drying regimes were observed in a single run: the constant-rate and falling-rate regimes as defined by [10]. At higher U_g, the constant-rate regime

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