Refereed Proceedings

The 13th International Conference on Fluidization - New Paradigm in Fluidization Engineering

INFLUENCE OF PROCESS PARAMETERS ON FLUIDISED BED DRYING OF POWDERED MATERIALS

Piero Bareschino[*]   Antonio Marzocchella[†]

Piero Salatino[‡]

[*]Università degli Studi del Sannio, piero.bareschino@unisannio.it
[†]Università degli Studi di Napoli 'Federico II', antonio.marzocchella@unina.it
[‡]Università degli Studi di Napoli 'Federico II', salatino@unina.it

INFLUENCE OF PROCESS PARAMETERS ON FLUIDISED BED DRYING OF POWDERED MATERIALS

Piero Bareschino[1], Antonio Marzocchella[2], Piero Salatino[3]

[1] Engineering Department, Università degli Studi del Sannio, P.zza Roma 21, 82100, Benevento, ITALY; T:+39 0824305589, F:+39 0824325246, e-mail: piero.bareschino@unisannio.it
[2] Chemical Engineering Department, Università degli Studi di Napoli 'Federico II', P.le Tecchio 80, 80125, Napoli, ITALY; T:+39 0817682541, F:+39 0815936936, e-mail: antonio.marzocchella@unina.it
[3] Chemical Engineering Department, Università degli Studi di Napoli 'Federico II', P.le Tecchio 80, 80125, Napoli, ITALY; T:+39 0817682258, F:+39 0815936936, e-mail: salatino@unina.it

ABSTRACT

The present study reports preliminary characterization about the fluidised bed drying of powdered materials. Tests were carried out in a Lexan® lab-scale fluidised bed with solids selected to effectively surrogate powders of interest in the manufacture of pharmaceuticals. 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. Bed material was characterized to assess the modifications of the population of agglomerates as a function of the operating conditions.

INTRODUCTION

The granulation process is particularly relevant in pharmaceutical industries for its relevance in solids attributes, significant for solids applications and handling. Larger granules of primary particles are commonly produced by high-shear granulation typically coupled with a drying stage carried out in gas-solid fluidised beds.

The influence of granulator design and granulation conditions on the attributes of the product granules has been extensively addressed in the literature (1-4). At authors knowledge, few studies can instead be found addressing the complex interplay of phenomena active during the drying stage, where abrasion and fragmentation of solid particles determine the agglomerates size distribution and morphology. The way key variables, like superficial velocity (U_g) and temperature (T_{IN}) of the fluidising gas, affect product granules is poorly characterized.

The present study aims to characterize the modifications of the population of agglomerates occurring in fluidised bed drying of powdered materials as a function of the operating conditions.

EXPERIMENTALS

Apparatus

The Lexan® lab-scale fluidised bed dryer, represented in figure 1, basically consisted of a conical fluidisation chamber (ID_{min} = 0.11 m; ID_{max} = 0.14 m; Height = 0.10 m) and a freeboard (ID = 0.14 m; Height = 1 m). The fluidised bed was equipped with a purposely designed gas distributor consisting of a perforated plate with holes inclined at 45 degrees with respect to the horizontal plane.

Pressure was measured by means of an electronic pressure transducer, temperature by a K-type thermocouples located along the plant, relative humidity by a Hanna-Instruments hygrometer (mod. HI8064).

Signals were logged on a data acquisition unit consisting of a PC equipped with a data acquisition board.

Materials

Table 1 reports the main properties of the solids tested. Two different granular materials were investigated: a spent FCC catalyst (FCC) and wheat bran (WB). Solids are characterized by different densities, Sauter mean diameters and angles of repose. Both solids were pre-stirred with an aqueous solution of HPM-Cellulose in a high-shear mixer in order to accomplish the granulation phase.
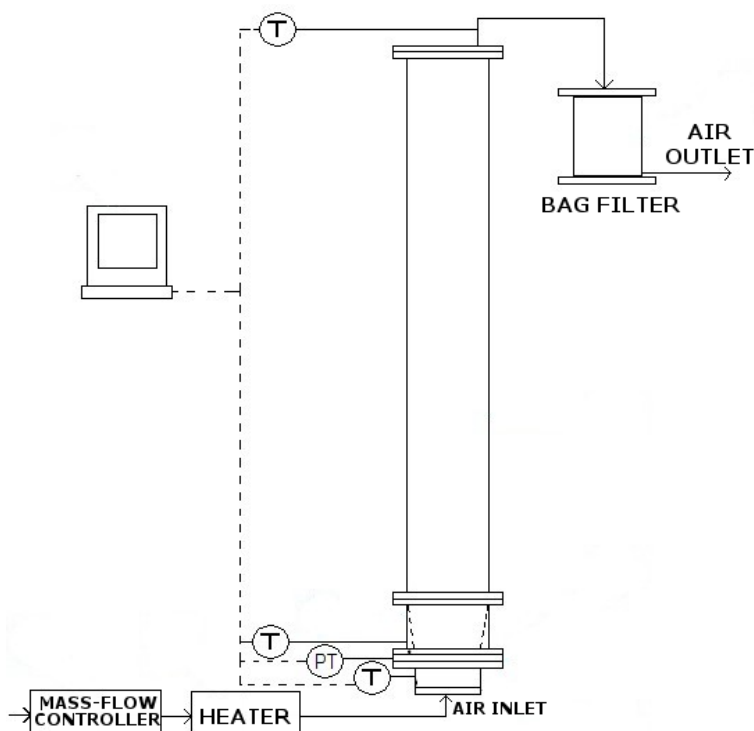

Figure 1: Experimental apparatus.
T – temperature; PT – pressure transducer

	FCC	WB
Material	FCC catalyst	Wheat bran
Sauter mean diameter (d_p), μm	40	50
Particle density (ρ_s), kg/m^3	1600	440
Incipient fluidization velocity (U_{mf})[*], m/s	$1.3 \cdot 10^{-3}$	$5 \cdot 10^{-4}$
Terminal velocity (U_t)[**], m/s	$7 \cdot 10^{-2}$	$3 \cdot 10^{-2}$
Angle of repose, °	35	45

[*] according to Wen and You [5]; [**] according to Haider and Levenspiel [6].

Table 1 - Properties of the granular solids investigated.

Operating Conditions and Procedure

Solids inventory was 0.2 kg in all tests. Superficial gas velocity (U_g) ranged between 0.12 and 0.24 m/s, inlet temperature of fluidising gas (T_{IN}) between 40 and 60 °C.

For any U_g and T_{IN}, the run was characterized in terms pressure and temperature at the bottom of the bed of solids (T_{bed}) and relative humidity of the outlet gas stream (Hr). Data were collected until Hr approaches the minimum required level (20%). At the end of the run fluidising gas was shut off and solids in fluidised bed were collected and analysed by means of a Malvern Instruments laser-light-particle-size analyser (LLPSA).

Data were further worked out to correlate drying time, Sauter mean diameter (d_{SM}) of the granules, temperature and superficial gas velocity of the fluidising gas.

RESULTS

Relative humidity

Figure 2 reports values of relative humidity measured during runs carried out with FCC. In particular: Fig. 2A refers to runs carried out at T_{IN}=60°C for different values of U_g; Fig. 2B to runs at U_g=0.18 m/s for different values of T_{IN}. The analysis of the Hr vs. t plots in Fig. 2 and those obtained during tests carried out with WB solids (data not reported) pointed out the following issues.

- For a fixed value of T_{IN} (Fig. 2A), the drying regime changes notably with U_g alters. At low value of U_g (0.12 m/s) two different drying regimes may be observed successively in a single run, i.e. the constant-rate and falling-rate regimes as defined by [7]. At higher U_g, the constant-rate regime disappears rather abruptly and only the falling-rate regime can be observed.
- For a fixed value of U_g (Fig. 2B), the drying regime does not change with T_{IN}.

Bed temperature

Figure 3 reports values of bed temperature measured during runs carried out with FCC. In particular: Fig. 3A refers to runs carried out at T_{IN}=60°C for different values of U_g; Fig. 3B to runs at U_g=0.18 m/s for different values of T_{IN}. The analysis of the

T_{bed} vs. t plots in Fig. 3 and those obtained during tests carried out with WB solids (data not reported) pointed out the following issues: i) for a fixed value of T_{IN} (Fig. 3A), at low value of superficial gas velocity (U_g=0.12 m/s) and in the constant-rate regime T_{bed} value remains stationary while, at higher U_g, as the constant-rate becomes a falling-rate regime a gradual growth of T_{bed} can be observed; ii) for a fixed value of U_g (Fig. 3B), the time resolved bed temperature profile remains unaltered whatever the value of the temperature of fluidising gas.

Bed temperature – superficial gas velocity maps

Drying time and Sauter mean diameter are reported in the (T_{IN}, U_g) phase space shown in Figure 4. An interesting result comes from the analysis of the surface morphology of the two plots: the drying time decrease monotonically with one or
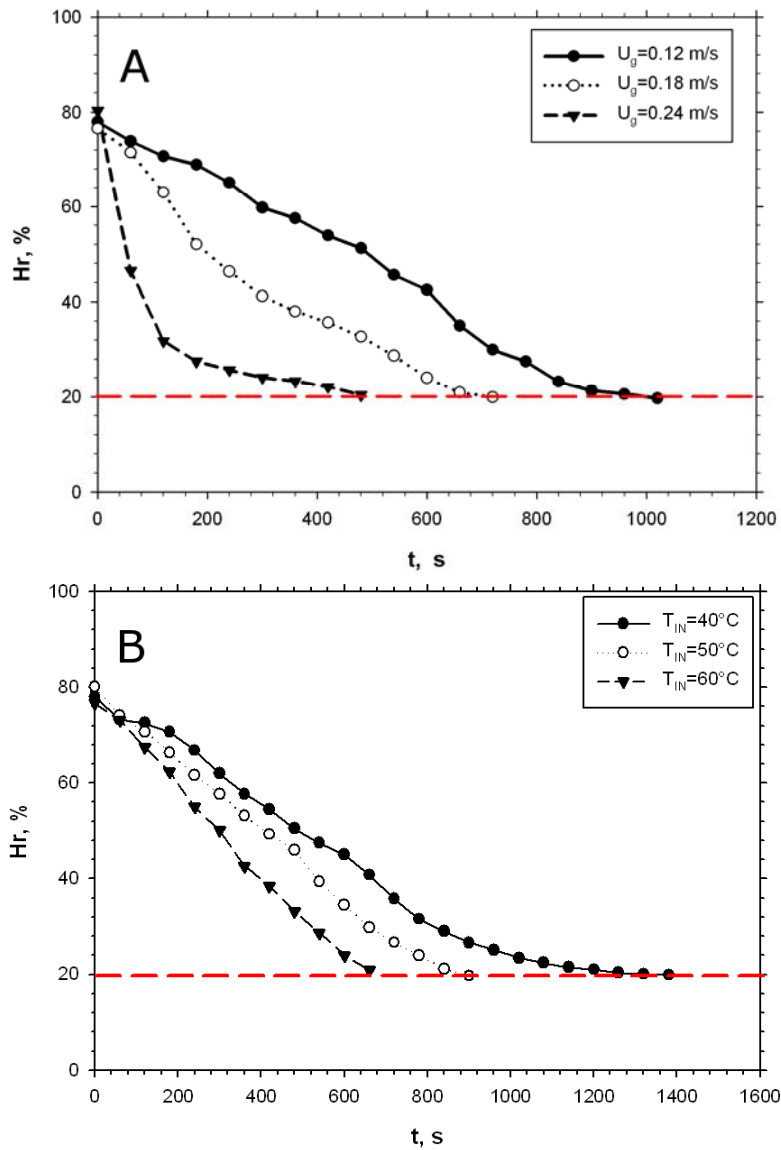

Figure 2: Relative humidity as a function of time. Solids: FCC.

A) T_{IN}=60 °C; B) U_g=0.18 m/s.

both T_{IN} and U_g; the Sauter mean diameter is characterized by a multi maximum surface. As a consequence, it results that: i) whatever the value of U_g, increasing T_{IN} produces a reduction of d_{SM} because the particles fragmentation due to temperature gradients boosts; ii) whatever the value of T_{IN}, Sauter mean diameter increases as U_g increases because drying time reduction prevails over augmented comminution phenomena in determining granule size distribution.

FINAL REMARKS

The fluidised bed drying of powdered materials was successfully carried out in a lab-scale unit. Agglomerates of FCC and wheat bean - solids selected to effectively surrogate powders of interest in the manufacture of pharmaceuticals - were investigated. The drying process was characterized in terms of drying time and of
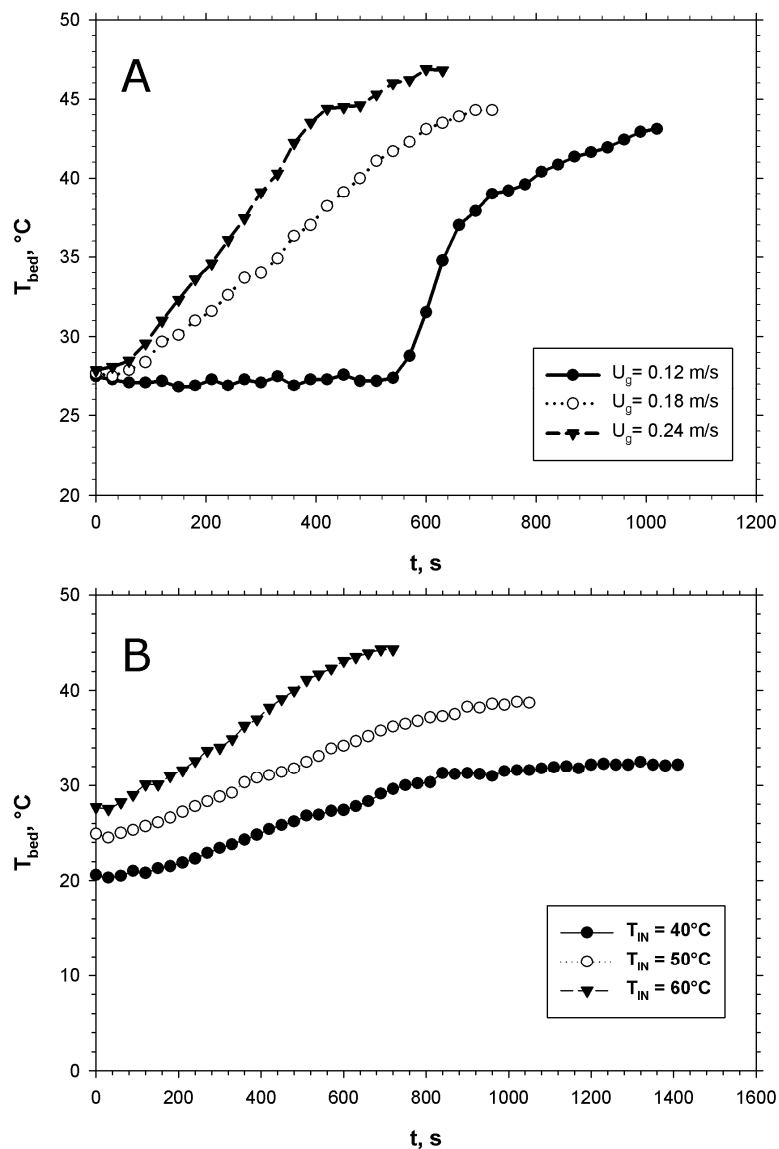
Figure 3: Bed temperature as a function of time. Solids: FCC.

A) T_{IN}=60 °C; B) U_g=0.18 m/s.

the modifications of the population of agglomerates as a function of the operating conditions.

Maps of the Sauter mean diameter and of the drying time in gas superficial velocity vs fluidising gas temperature phase plane were outlined. The maps showed that:
- the drying time decrease monotonically with one or both the gas superficial velocity and the fluidising gas temperature;
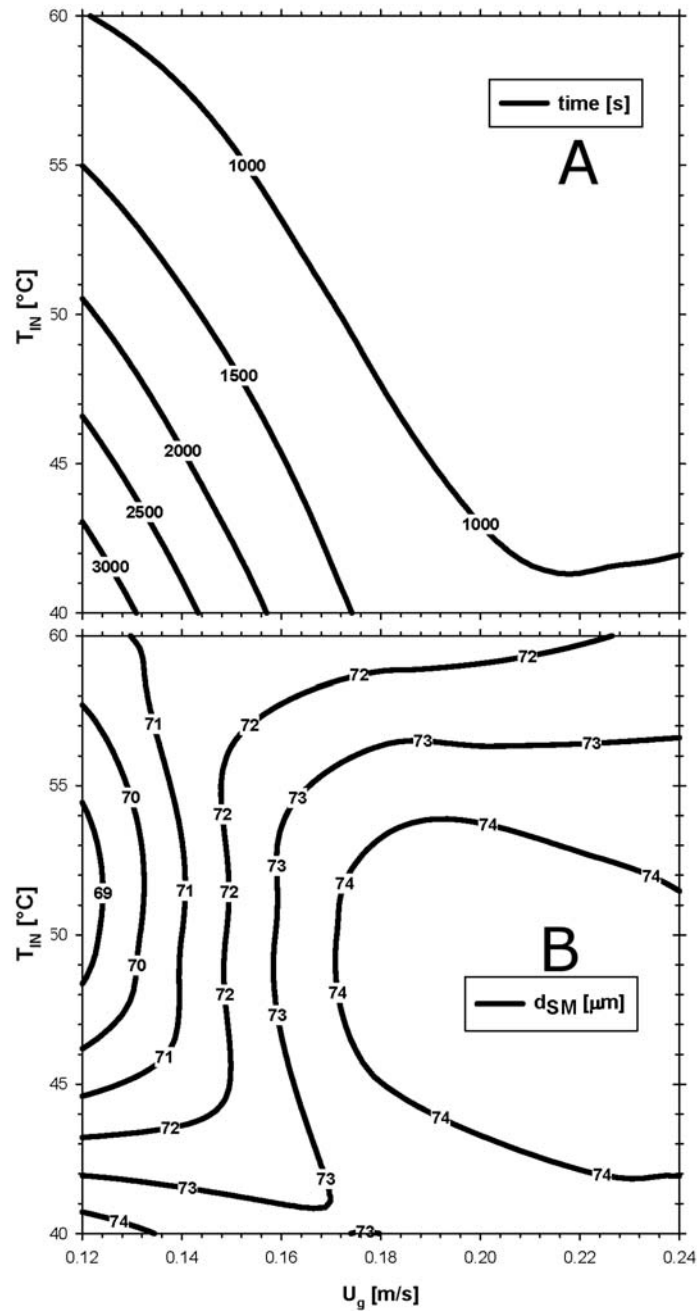- the Sauter mean diameter is characterized by a multi maximum surface.

Figure 4: Drying time (A) and Sauter mean diameter (B) as a function of superficial gas velocity U_g and inlet temperature of fluidising gas T_{IN}. Solids: FCC.

NOTATION

d_{SM} Sauter mean diameter, μm
Hr   relative humidity of the outlet gas stream, %
T_{bed} temperature of the solids bed, °C
T_{IN} temperature of the fluidizing gas, °C
U_g superficial gas velocity, m/s

REFERENCES

1) Borruto, A., Crivellone, G., Marani, F., Influence of surface wettability on friction and wear tests, Wear 222 (1998), pp. 57–65
2) Holm, P., Jungersen, O., Schæfer, T., Kristensen, H.G., Granulation in high-speed mixers. Part I. Effects of process variables during kneeding, Pharm. Ind. 45 (1983), pp. 806–811
3) Holm, P., High shear mixer granulators, In: D.M. Parikh, Editors, Handbook of Pharmaceutical Granulation Technology vol. 81, Marcel Dekker, New York (1997), pp. 151–204.
4) Bouwman, A.M., Visser, M.R., Eissens, A.C., Wesselingh, J.A., Frijlink, H.W., The effect of vessel material on granules produced in a high-shear mixer, Europ. J. of Pharm. Sciences, 23 (2), (2004), pp. 169-179.
5) Wen, C.J., Yu, Y.H., A generalized method for predicting the minimum fluidization velocity. AIChE Journal 12(3):610-612 (1966).
6) Haider, A., Levenspiel, O., Drag coefficient and terminal velocity of spherical and nonspherical particles. Powder Technol., 58, 63 (1989).
7) Vanecek, V., Drbohlav, R., Markvart, M., Fluidized Bed Drying, Leonard Hill Books: London (1966).