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Wet Granulation of Cereal Grains in a Tapered Fluidized Bed

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Wet granulation of cereal grains has been studied in a conical fluidized bed designed and realized in our laboratory. This test facility consists essentially of a conical vessel, a two-fluid nozzle and an electric heater for drying. Powders of durum wheat semolina, barley flour and durum wheat flour were the solid materials used. The hydrodynamic study, conducted for different static bed heights and air flows of 2 - 30 m^3/h, led to the determination of the minimum fluidization velocity and maximum pressure drop. For different operating conditions, granulation tests were carried out in batch. Particle size, flowability, friability and residual humidity of grains were the parameters investigated.

1. Introduction

Powders wet granulation process which consists to agglomerate and enlarge size grains, in the aim to modify their properties (size, shape, density, porosity, flowability), finds many applications in several industries like chemical pharmaceutical and food industries.

The modern theory of granulation distinguishes three stages in the granulation process: wetting and nucleation, consolidation and growth, breakage and attrition (Tardos et al., 1997; Iveson et al., 2001). In recent years, several studies have been conducted to investigate the influence of process variables and physicochemical properties on granulation mechanisms (Bouffard et al., 2005; Abberger, 2001).Various technologies exist and are distinguished by the means used to ensure the movement of solid particles. Thus we can distinguish:

- Agglomeration by mechanical agitation made in a fixed granulator.
- Agglomeration by rolling carried out in rotating plates and drums.
- Agglomeration in a fluidized bed: particles motion is performed by an air flow passing upward and the three steps (mixing, granulation and drying) take place in the same equipment. (Benali et al., 2009; Palzer, 2011). Tapered beds are particularly indicated for the fluidization of materials with a wide particle size distribution because superficial fluid velocity is reduced when cross-sectional area increase with the bed height.

According to currently available knowledge in the literature it emerges that fluidization offers to the wet granulation several advantages in particular intense mass and heat transfers with vigorous mixture of solids in bed. However, the mechanisms of particles growth remain still insufficiently understood because of the complex interactions between the thermodynamic and process parameters.

In this optic, to study wet granulation of cereal grains in a tapered fluidized bed, an experimental set-up was conceived and realized.

Granulation tests were carried out in batch with pure water, NaCl saturated water and starch solution as the binding liquids during wetting steps.

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2. Materials and experimental procedure

2.1 Products

Powders of durum wheat semolina, barley and durum wheat flour were the products used in experiments. The particle size of the powders was measured by normalized sieves (AFNOR) and flow proprieties were evaluated by determining Carr's index and Hausner ratio values. Characteristics of these solid particles are presented in Table 1.

Optical microscopic (OM) images of primary particles are compared in Figure 1. The three cereals have different sizes and irregular shape.

Three binder solutions were used; water (density ρ = 980.0 kg/m^3), NaCl saturated aqueous solution (density ρ = 1,121.67 kg/m^3) and starch saturated aqueous solution (density ρ = 1,008.0 kg/m^3).

Table 1: Physical proprieties of primary solid particles

Solid material	Cereals		
	Durum wheat flour	Barley flour	Durum wheat semolina
Median mean size d_{50} (µm)	68	341	584
Sauter mean diameter d_{32} (µm)	78.93	291.34	585.7
Span = $(d_{90}-d_{10})/d_{50}$	0.97	1.78	0.78
Density (kg/m^3)	1,218.0	1,318.91	1,437.7
Apparent density (kg/m^3)	491.33	498.0	710.67
Carr's index (%)	39	20	14
Hausner ratio	1.65	1.25	1.16
Porosity		0.55	0.48
Geldart classification	C	A	B

Durum wheat flour Barley flour Durum wheat semolina

Figure 1: OM images of cereal grains (X 50) Nikon Epiphot 300

2.2 Equipment

Figure 2 presents the experimental set-up conceived and realized for the present study. Air flow from the air compressor (5) is measured (4) and heated to the required temperature as it flows over an electrical heater (3) before it enters the calming zone (2), filled with ceramic balls, and then the conical fluidized bed granulator (1). The fluidized bed granulator of 11 cm bottom diameter and 24 cm top diameter is a brass column with a tapered angle of 9.5 °. The granulator is fitted with a 3 % perforated plate distributor and a filter bag at the top (8). Thermocouples (T) are provided to control temperature of inlet gas and fluidized bed.

A two-fluid nozzle (7) fed by a peristaltic pump (9), is used to top spray binder solution on solid particles.

2.3 Procedure

A weighed amount of powder was charged in the column and air was allowed to pass through the bed. Then the airflow was slowly increased till the bed was fluidized.

Pressure drop across the bed was measured by two static pressure taps and U-tube manometer.

For tests granulation, during each trial, process parameters (air inlet temperature, air flow rate, liquid feed rate and spraying pressure) were kept constant.

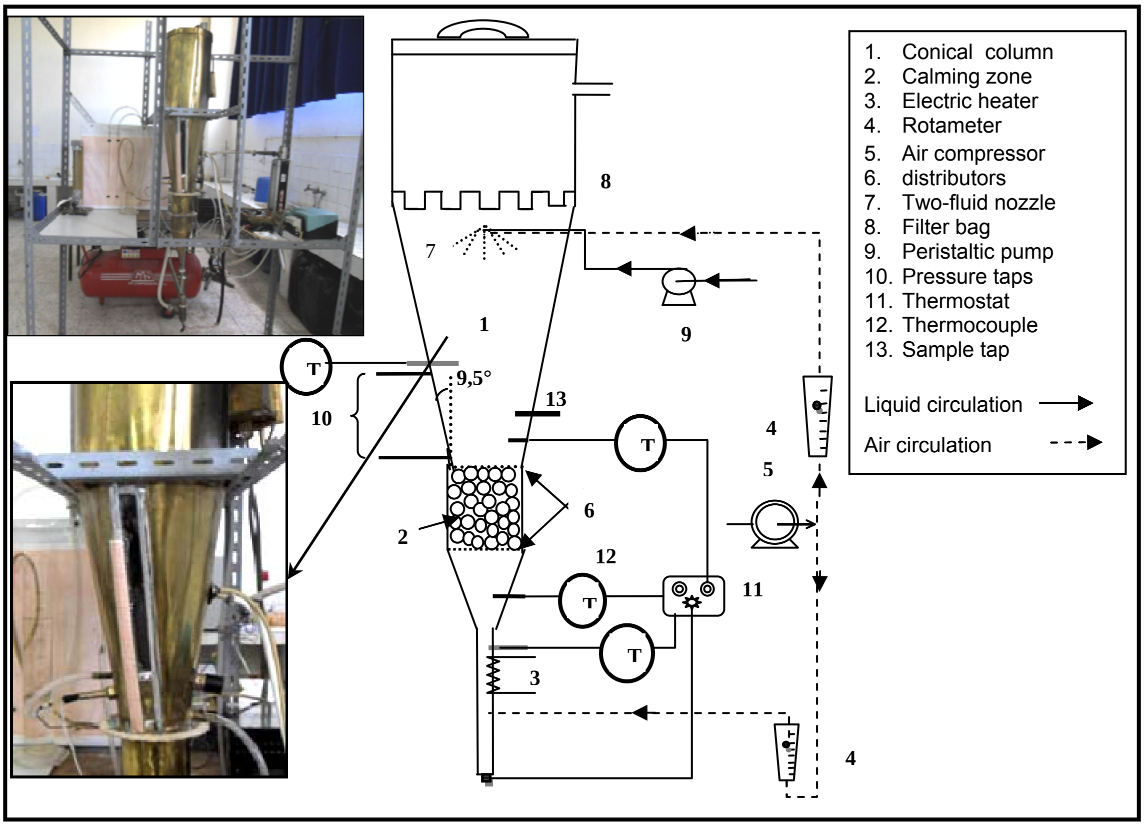

Figure 2: Photos and schematic diagram of experimental set-up

3. Results and discussion

3.1 Hydrodynamic study

Prior to granulation experiments, preliminary hydrodynamic experiments were carried out to confirm the operating stability and to determine hydrodynamic parameters such as minimum fluidization velocity and maximum pressure drop for the three cereals which are group-C, A and B particles in Geldart classification. For these three kinds of particles, Figure 3 presents the pressure drop across the bed versus the superficial air velocity for three static bed heights. According to the curve shape, three regions corresponding to different bed modes are distinguished. In Region I, fixed-bed mode, the pressure drop increases with superficial air velocity approaching the maximal value at minimum fluidization velocity. In region II, partially fluidized-bed mode, the pressure drop diminishes with higher air velocity and then stays nearly constant in Region III corresponding at fully fluidized-bed mode.

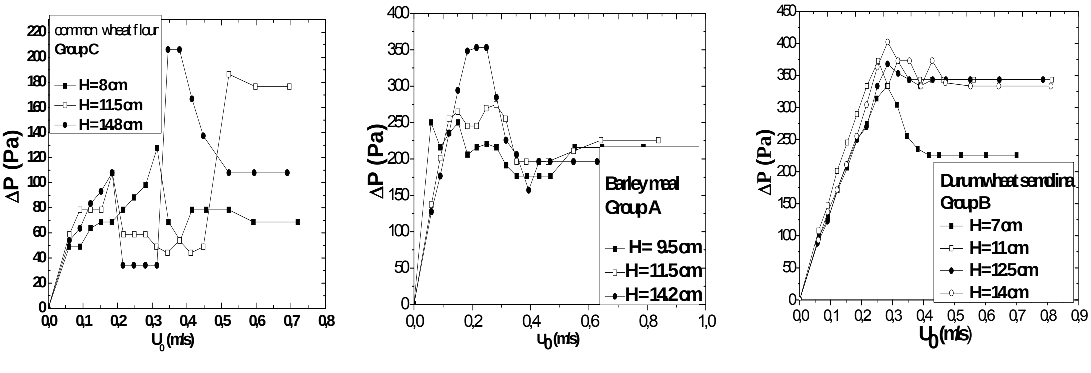

Figure 3: Evolution of pressure drop versus superficial air velocity for various bed heights

It appears that group particles in the Geldart classification had a considerable effect on the pressure drop evolution like on measurements quality. With durum wheat semolina and barley flour, groups B and A respectively, fluidization was easy without many disturbances. On the other hand with the common flour of wheat, group C and which is a cohesive powder with a tendency to agglomerate, fluidization was difficult and significant fluctuations in pressure drop measurements were recorded.
The minimal fluidization velocity as well as the maximum pressure drop was deduced graphically from the plot of pressure drop versus air velocity for each case.

Table 2: Comparison of experimental data with those predicted by Peng and Fan correlations

Powder	H_0 (cm)	U_{mfcal} (m/s)	U_{mfexp} (m/s)	Deviation (%)	ΔP_{maxcal} (Pa)	ΔP_{maxexp} (Pa)	Deviation (%)
Durum wheat semolina	7.0	0.163	0.179	8.86	190.03	330.16	42.43
	11.0	0.201	0.232	13.29	308.78	369.12	16.34
	12.5	0.209	0.266	21.30	354.20	362.92	2.40
	14.0	0.217	0.230	5.66	400.74	397.92	0.71
Barley flour	9.5	0.115	0.132	12.83	249.5	131.141	47.43
	11.5	0.118	0.10	18.40	273.37	159.27	41.73
	14.2	0.128	0.119	3.37	352.13	197.67	43.86

The obtained results showed a satisfactory accord with the predicted values suggested by Peng and Fan correlations as it is shown in Table 2.

3.2 Wet granulation

Granulation trials were carried out in batch only with durum wheat semolina and barley flour particles for their easy aptitude to be fluidized.These experiments were performed in three periods: bed heating, pulverization/ Drying and Cooling/ final dry. The adopted operating conditions are presented in Table 3.

Table 3: Operating conditions for tests granulation

Trial	Load (g)	Binder solution	Q_L (mL/min)	P (bar)	T (°C)
		Durum wheat semolina			
1	150	Water	20.68	2	55
2	150	Water	20.68	1	55
3	150	Water	29.68	2	55
4	350	Water	20.68	2	55
5	150	Water	20.68	2	50
6	150	NaCl saturated aqueous solution	20.68	2	55
7	150	Starch saturated aqueous solution	20.68	2	55
		Barley flour			
8	150	water	20.68	2	55
9	150	NaCl saturated aquous solution	20.68	2	55

Influence of operating conditions on particles growth

In order to examine the influence of operating parameters on particle growth, granulation experiments were carried out as indicated in Table 3.
Particles growth was observed for each granulation test. According to results in Figure 4, median diameter d_{50} of aggregates increased quickly during spraying step to reach a maximum value then it decreased slowly during drying period.
The increase in liquid flow rate increased considerably particle size. For durum wheat semolina powder, the median particle diameter (d_{50}) increased from 1,278.73 to 3,151.70 µm when liquid feed rate increased from 20.68 to 29.68 mL/min. This is due to the fact that more liquid binder is available for particle agglomeration. On the other hand, when particle load was increased from 150 to 350 g the median particle diameter decreased from 1,278.73 to 1,169.66 µm. In this case smaller agglomerates were obtained because of more particles had to be agglomerated with the same amount of liquid binder.
For two wheat powders used, binder solution characteristics influenced size agglomerates. The NaCl saturated water solution being denser than water led to an increase in size agglomerates from 1,278.73 to 1,425.40 µm.

When the inlet air temperature was augmented from 50 to 55 °C, the median particle diameter decreased from 1,658.58 to 1,278.73 µm. This size reduction can be attributed to fast drying (Jimènez, et al. 2006).

In addition, it was observed that during drying step, median agglomerates diameter decreases. For durum wheat semolina the size reduction was from 12 to 50 % and 30 to 60 % for barley flour .This tendency is not only due to the consolidation of solidified bridges but also to the fact that agglomerates are in the same time subjected to mechanical constraints due to the collisions with other agglomerates and column walls. These phenomena cause the attrition and/or the rupture of fragile agglomerates, modifying the agglomerates size and size distribution according to their mechanical resistance.

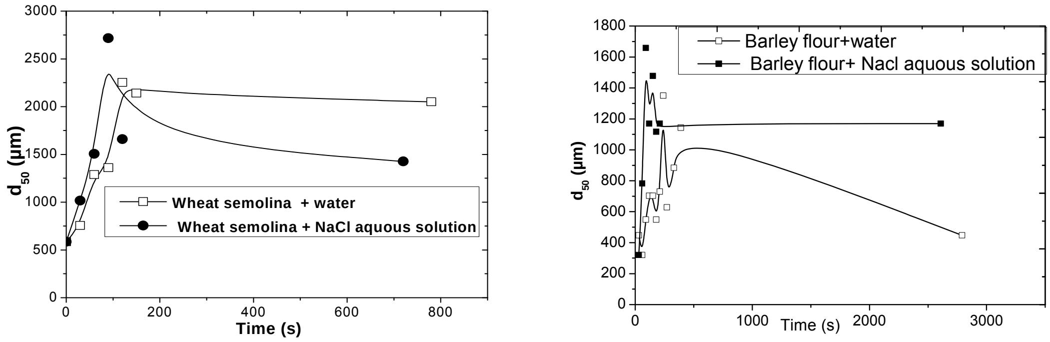

Figure 4: Median particles diameter evolution for durum wheat semolina and barley flour powders

Characteristics of final agglomerates

The sieve analysis performed on dry grains permitted to compare the mass distribution particle size before and after granulation. It can be seen from the data presented in Figure 5 that the particle size distribution varied significantly. The initial load was distributed after granulation on a large size interval. For durum wheat semolina, the diameter extended on an interval from 500 to 8000 µm and for the barley flour on an interval from 100 to 8,000 µm. It appears also that 2 to 15 % of the initial particles were not granulated or they were reproduced by rupture of aggregates.

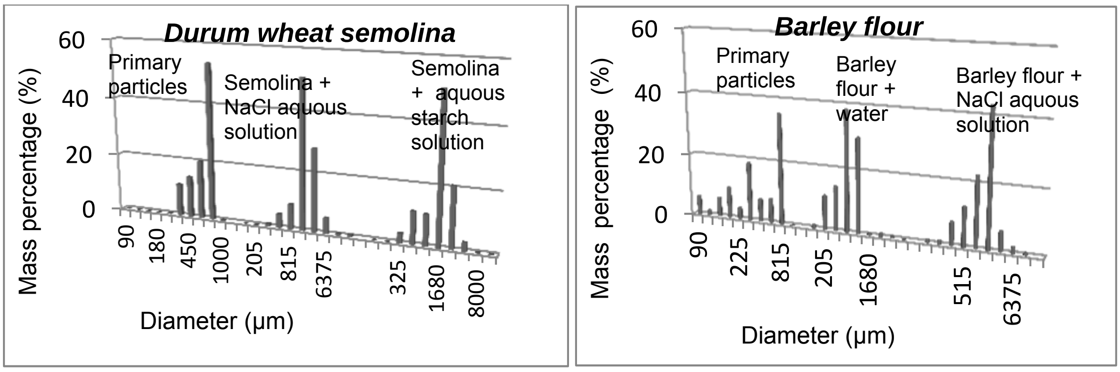

Figure 5: Mass distribution particle size before and after granulation

In order to determine flow and compressibility aptitude of obtained grains Hausner ratio and Carr's index values were calculated using taped and aired densities. For durum wheat semolina grains, these indices were respectively lower than 1.20 and 0.16 confirming a good flowability and compressibility; however for barley flour grains Hausner ratio and Carr's index were respectively lower than 1.40 and 0.35 proving an average flowability.

In addition, the mechanical resistance of the agglomerates, acquired during spraying and consolidated during final drying step, is an important property for their stability during handling, transport and final use.

This property was expressed as the percentage of agglomerates weight which initial size was reduced after being subjected to intense vibrations on a sieve during a given time.

Figure 6 shows that the friability increases slightly and then tends to stabilize. This slow increase confirms that the agglomerates crumble not easily so they are relatively rigid. Friability of durum wheat semolina agglomerates was between 1.3 to 11.6 % whereas that of barley flour agglomerates was between 14.2 and 16.5 %.

Residual humidity measurements (Table 4) showed that obtained agglomerates, which were subjected to a drying step, present residual humidity lower than those of initial powders.

Table 4: Agglomerates residual humidity

	Durum wheat semolina 14.81							Barley flour 15.10	
Trial	1	2	3	4	5	6	7	8	9
Residual humidity (%)	14.28	12.99	8.10	16.27	12.99	12.99	11.11	7.5	13.63

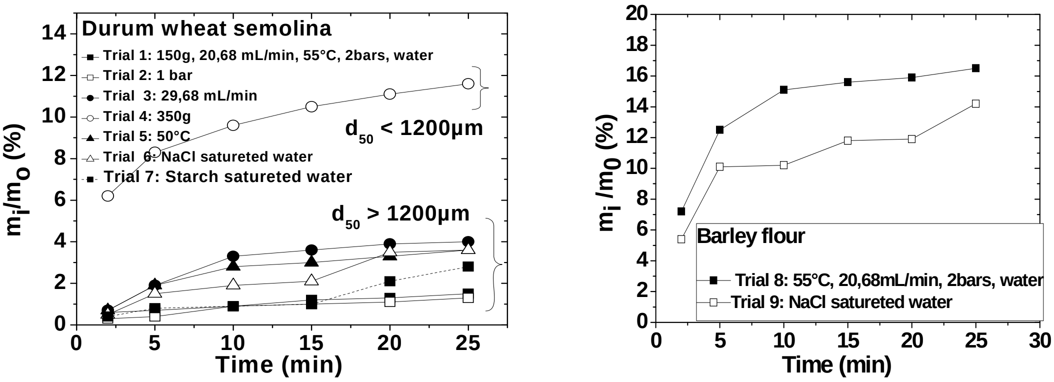

Figure 6: Agglomerates friability

4. Conclusion

The operating parameters studied had a significant influence on the agglomerate quality.

The granulation with NaCl satureted water produced grains more rigid than those formed with water. The NaCl and starch aqueous solutions led to obtain agglomerates of larger size and with better mechanical resistance.

The drying step involved a reduction of 12 to 50 % of the agglomerate median diameter for durum wheat semolina and 30 to 60% for barley flour.

The agglomerates obtained with group- B particles in Geldart classification show the best characteristics of flowability and resistance.

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