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Impact of fluidized bed granulation on structure and functional properties of the agglomerates based on the durum wheat semolina

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Abstract - The granulation step determines the production yield and the final characteristics of the agglomerated couscous grains of durum wheat. The objective of the present work was to explore the capability of the fluidised bed technology to produce agglomerates of durum wheat semolina. The impacts of different processing conditions have been investigated on the structure and functional properties of the agglomerates. The size, shape, water content, compactness, and mechanical strength of the granules were measured. The fluidized bed agglomeration process has been found to produce agglomerates of durum wheat with different attributes compared to those produced by granulation using the low shear mixers. The results were discussed in regard to the hydro-textural approach, in order to get a better understanding of the mechanisms and relationships between process, structure, and properties. Two major agglomeration mechanisms contribute to the growth of the wet agglomerates: a fractal-structuring process followed by a phenomenon of densification. By studying the evolution of the compactness, diameter and water content, it was demonstrated that inter granular arrangements led to an expansion followed by a densification of the wet agglomerates. A relationship was proposed to describe the growth using a fluidized bed of the wet agglomerates of durum wheat semolina.

Keywords - Fluidized bed ; agglomeration ; couscous grains structure ; hydrotextural diagramm

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1. Introduction

Wet agglomeration is based on the coupling of two phenomena: mixing the powder and incorporating a binder. The structuration of associated sticky particles takes place under shear stress (Iveson, Litster, Hapgood, & Ennis, 2001). Agglomeration of powders using a fluidized bed has been largely investigated either in batch or in open mode (Boerefijn & Hounslow, 2005; Nienow, 1995; Philippsen, Vilela, & Zen, 2015; Rambali, Baert, & Massart, 2001; Rieck, Hoffmann, Bück, Peglow, & Tsotsas, 2015; Turchiuli et al., 2005; Zhai et al., 2009). This technology can be used to agglomerate powders by spraying a binder solution directly onto the fluidised powder bed (Saleh & Guigon, 2009; Turchiuli, Eloualia, El Mansouri, & Dumoulin, 2005). The growth mechanisms of the agglomerates depend on the ability of process parameters to activate the surface reactivity of particles and promote collisions. The liquid binder is usually distributed by means of an atomising nozzle, above or inside the powder bed. A vertical air stream agitates the fluidized particle's bed and contributes to the heat and moisture transfers. Several simultaneous and/or competitive mechanisms occur (wetting/drying, growth/rupture, swelling/shrinkage, consolidation, chemical reactions…) and contribute to the agglomeration (Leuenberger, Puchkov, Krausbauer, & Betz, 2009). Spray nozzle orientation and design, binder flow rate, flows and distribution of the fluidization gas inside the processing chamber, relative humidity, and temperature are the multiple process parameters involved (Jacob, 2007; Pathare, Baş, Fitzpatrick, Cronin, & Byrne, 2012). As the binding liquid is sprayed as small droplets, the agglomeration mechanisms are initiated by the coating of the native particles. The formation of initial nuclei is followed by the coalescence mechanisms (Banjac, Stakic, & Voronjec, 1998; Barkouti, Turchiuli, Carcel, & Dumoulin, 2013; Turchiuli, Smail, & Dumoulin, 2013). The pneumatic agitation of the powder bed produces collisions between the wetted particles that could coalesce by the formation and the persistence of liquid bridges. The growth of granules is also affected by the rupture mechanisms of the structures due to shear stress between the wall and/or breakage induced by the intensity of the collisions. The distinction between these coupled phenomena in terms of rate and spatial localisation in the reactor represents a challenge to improve agglomeration (Hemati, Cherif, Saleh, & Pont, 2003; Smith & Nienow, 1983). The shape and texture of the agglomerates are essentially depending on the mechanical environment generated in the reactor. Due to the low shear stresses developed by the air flow, the granules formed using a fluidised bed are generally more porous and have

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more irregular shape than those produced by using rotating drums and mechanical shear mixer (Jacob, 2007; Ji, Fitzpatrick, Cronin, Fenelon, & Miao, 2017; Morin & Briens, 2014).

The food powders can be agglomerated to improve their properties (e.g. size distribution, shape, flowability, instant properties, colour, *etc.*) (Barkouti et al., 2013; Dacanal & Menegalli, 2010; Palzer, 2011). Fluid bed agglomeration has been used to improve the instant properties and the flowability of dairy powders, instant coffee, cocoa beverages, or culinary powders (Palzer, 2011; Turchiuli et al., 2013). The binder solution is simply water when processing amorphous water-soluble particles, but viscous liquids are used to agglomerate crystalline water-soluble particles (Cuq, Mandato, Jeantet, Saleh, & Ruiz, 2013). The agglomeration is a key operation in the elaboration of couscous grains of durum wheat semolina (Barkouti, Rondet, Delalonde, & Ruiz, 2012; Saad, Barkouti, Rondet, Ruiz, & Cuq, 2011). Industrial couscous grains are made by successive operations: wet agglomeration, rolling, cooking, drying, and screening. In the industrial context, the durum wheat semolina is granulated using mechanical low shear mixers, to imitate the traditional homemade processing (Abecassis, Cuq, Boggini, & Namoune, 2012). Wet granulation stage is one of the critical stage that determines the production yield, the final size, shape and functionalities of the couscous grains (Barkouti, Delalonde, Rondet, & Ruiz, 2014; Hafsa et al., 2015; Hébrard, 2002; Oulahna, Hebrard, Cuq, Abecassis, & Fages, 2012; Saad et al., 2011). Only few technical and scientific works are available to rationalize the management of the process, which restricts the potential of rupture innovation by only optimization and incremental solutions.

The objective of the present paper is to explore the capability of the fluidised bed technology to produce agglomerates of durum wheat semolina. The impact of different processing conditions on the structure characteristics and functional properties of the agglomerates have been investigated by experiments. Results are discussed in regard to the hydrotextural approach (Thierry Ruiz, Rondet, Delalonde, & Desfours, 2011), in order to get a better representation and understanding of the mechanisms and relationships between process, structure, and properties.

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2. Material and Methods

2.1. Raw materials

Two different durum wheat semolina were used as raw material for the agglomeration experiments: standard industrial semolina (Panzani group, France) and coarse semolina (UMR IATE, France). Semolina were stored in hermetic containers at 4°C until experiments were carried out. Semolina were characterized using standardized methods (Table 1). The water content was determined according to the approved method 44-15A (AACC, 2000), by weighing after oven drying at 105°C for 24 h. The characteristics values of particle diameters were measured by a laser particle size analyser (Coulter TMLS 230, Malvern, England) at room temperature. The semolina true dry density (ρ_s^*) was measured by using a nitrogen pycnometer (ULTRAPYC 1200e, Quatachrom) after oven drying at 105°C for 24 h. The semolina apparent density (ρ) was measured by a hydrostatic balance (RB 360, WC Heraeus GmbH, Hanau, Germany). From these values, the compactness of the semolina particles at each water content was calculated as: $\phi_P = \frac{\rho}{(1+w).\rho_s^*}$. The total nitrogen content (TN) of semolina was determined by the Kjeldahl method, and the crude protein content was calculated according to TN-5.7 based on the AFNOR method V 03-050 (AFNOR, 1970). All experimental measurements were carried out in triplicate.

2.2. Agglomeration process

The wet agglomeration process was conducted by using a glass fluidised bed granulator (Mini-Airpro, ProcepT, Belgium) (Fig. 1), whereby the lower portion is conical in shape and the upper portion is cylindrical (height = 0.73 m). Water was used as the liquid binder. Water was sprayed using a nozzle in a downward direction (*i.e.* in the counter current to the air flow). The temperature of the inlet airflow was kept constant during experiments (25°C) and controlled using a temperature probe (Fig. 1). A constant weight of semolina (300 g) was first introduced inside the unit and was fluidized by ascendant airflow at constant rate (0.70 m^3/min) for 2 min to equilibrate the temperature. The flow rate of water was kept constant using a peristaltic pump (8 mL/min), and controlled by the continuous measurement of the weight of the water tank. The time for water addition (3, 6, 9, 12.5, or 16.3 min) was defined in regards with the amount of added water (24, 48, 72, 100, or 130 g) that was incorporated inside the reactor, in order to reach specific amount of added water (0.08, 0.16, 0.24, 0.33, or

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0.43 g of added water / g of semolina), and specific values of apparent process water contents (0.24, 0.33, 0.42, 0.53, or 0.65 g of water / g dry matter). Due to the simultaneous drying mechanisms occurring during processing, the values of the apparent process water content were significantly higher than the true water content measured in the bed at the end of the process (Fig. 2). An almost constant value of the drying rate (11.0 ±0.9 g of water / min) was determined whatever the process conditions. Immediately after the end of the water addition stage, the wet agglomerates were collected. Trials were conducted in triplicate.

2.3. Cooking and drying process

Immediately after the agglomeration stage, the collected wet agglomerates were sieved over a column of 2 metallic sieves of decreasing mesh (1.4 and 0.85 mm) for 2 min at ambient temperature. The sieve column was softly shaken manually to limit the possible breakage of the wet agglomerates. The range of mesh size was determined in regards with the capacities of the experimental equipment and with the values classically used for the industrial production of the couscous grains. The wet agglomerates that were collected over the 0.85 mm sieve, were immediately spread as a thin layer (about 3 mm) over a stainless steel plate inside a steam cooker (Kenwood rice cooker RC310), and steamed for 10 min at 100°C using saturated steam at atmospheric pressure. The steamed agglomerates were immediately collected, spread as a thin layer (about 3 mm) over stainless steel mesh and then drying in an oven (GallenKamp Plus II oven 150L) for 25 min at 50°C and 15% relative humidity. The dried agglomerates were collected and stored inside hermetic plastic cups until characterization.

2.4. Characterization of the agglomerates

Water content - The water content (w) of agglomerates (dry base) was determined on 3-5 g samples, by a drying method in an oven at 105°C for 24 h (AACC, Method 44-15A): $w = m_w/m_s$, m_w is the mass of water (g) and m_s is the mass of dry matter (g) in the sample. Mean values were determined from triplicate.

Compactness - The compactness of the agglomerates (ϕ) is calculated using Eq. (1).

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$$\phi = \frac{V_s}{V} = \frac{m_s}{V.\rho_s^*} \quad (1)$$

Where V_s is the solid volume of the agglomerate (cm^3 of solid matter); V is the total volume of the agglomerate (cm^3); m_s is the solid mass (g of dry matter), ρ_s^* is the solid real density of durum wheat semolina. The apparent volume (V_a) of the agglomerates was measured by using a Camsizer (Retsch, Germany) digital image analyser. The solid mass (m_s) was measured with a digital balance with sensitivity of 0.001 g. In the case of unsaturated structures, the compactness is a function of the water content and the saturation degree ($S=V_w/V_{void}$) and for strictly wet media ($0 < S \leq 1$) Eq. (2) can be written as follow:

$$\phi(w,S) = \frac{1}{1+d_s^*\frac{w}{S}} \quad (2)$$

Size distribution of the wet agglomerates - The wet agglomeration process generates agglomerates with dispersed sizes, which were measured directly after the agglomeration stage. Size distribution was determined by sieving all the collected agglomerates over a column of 7 metallic sieves of decreasing mesh size (3.35, 2.8, 2.36, 1.40, 0.85, 0.71, and 0.425 mm). The sieve column was manually shaken for only 1 min, to limit the possible particle breakage. The size distribution was obtained by weighing the mass of agglomerates remaining on each sieve. The weight distribution according to size criteria was expressed as the percent of total weight. Even if the particle size distribution curves were not fully unimodal, the apparent median diameter (d_{50}) and apparent span: $(d_{90}-d_{10})/d_{50}$, of the size distribution were calculated as global apparent descriptors. The agglomerates, that were collected on the sieves of 0.425, 0.71, 0.85, 1.40, or 2.36 mm mesh size, were characterized by their size, water content, and compactness.

Median diameter of the dried agglomerates - Samples of agglomerates (about 10 g) were used to determine the median diameter on each sieve, using the Camsizer (Retsch, Germany) digital image analyser. Particle size distributions were characterized by the value of the median diameter. Measurements were conducted in triplicate.

Agglomerates shape – The sphericity of the dried agglomerates was determined by using the Camsizer (Retsch, Germany) digital image analyser. Samples of agglomerates (about 10 g) were used. Sphericity is a aspect ratio defined by the measurement of the length/width

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relationship. The values range between 0 (perfect circle) and 1 (needle shaped object). Measurements were conducted in triplicate.

Agglomerates microstructure - The microstructure of the dried agglomerates was observed by scanning electron microscopy (Inspect F by FEI operating at 5kV). A sample of agglomerates was dispersed on a conducting carbon tape prior to analysis. Representative pictures were selected for each sample.

Agglomerates strength - The strength of the dried agglomerates was measured using a texture analyser (TA-HD Plus, Stable Micro Systems, UK). The agglomerates strength is defined as the force needed to compress one agglomerate over a distance of 400 µm. A 5 kg load cell was used with a compressive probe operating at 100 $\mu m.s^{-1}$. Texture experiments were conducted by characterizing the agglomerates one by one. It was thus not possible to characterize a large number of agglomerates. A restricted number of agglomerates (about 10) having some homogeneity in their size, to be statistically representative of the sample were tested. Mean values were determined from 10 measurements.

2.5. Statistical analysis

The statistical significance of results was assessed using single factor analysis of variance (ANOVA). Multiple comparisons were performed by calculating the least significant difference using Microsoft Excel 2010, at a 5% significance level.

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3. Results

3.1. Characterisation of the wet agglomerates after the agglomeration stage

The wet agglomeration process by using the fluidized bed was first conducted with the standard semolina and apparent process water content of 0.53 g/g dry matter. As expected, the wet agglomerates collected after the fluidized bed processing were characterized by high values of measured water content (0.37 ±0.04 g/g dry matter) and low values of compactness (0.41 ±0.04), compared to the native semolina particles (0.148 g/g dry matter and 0.85, respectively). The size distribution of the wet agglomerates (Fig. 3) demonstrated a relatively large range in the diameter values (from 500 to more than 4000 µm), with median diameter (d_{50}) close to 650 µm (±3 µm). The value of the span (2.27) was relatively low, indicating that the majority of the grains were centred on the median diameter. The mass fraction of the wet agglomerates with diameter ranging between 1000 and 2000 µm was about 40%. This value is almost similar than the mass yield usually observed during the industrial processing of the couscous grains using low shear mechanical mixers (Abecassis et al., 2012).

The physicochemical characteristics of the wet agglomerates were measured for each class of agglomerates as a function to their diameter (Fig. 4). The water content of the wet agglomerates was positively correlated with their diameter (Fig. 4a). Similar positive correlations have already been observed for the wet agglomerates of durum wheat semolina processed by the mechanical mixing under low shear conditions (Barkouti et al., 2012; Bellocq, Ruiz, Delaplace, Duri, & Cuq, 2017).

The increase in the median diameter of the wet agglomerates is also associated to changes in compactness (Fig. 4b). The compactness of the wet agglomerates decreases (from 0.43 to 0.36) with an increase in the median diameter until 1600 µm. Above 1600 µm, the compactness tends to increase as the median diameter increases.

The results also demonstrated a positive relationship between the values of sphericity and the diameter of the wet agglomerates (Fig. 4c). The large agglomerates were more spherical than the small ones. It should be noticed that the wet agglomerates with diameters between 1000 and 2000 µm were characterized by high values of sphericity (0.82 - 0.87) and low values of compactness (0.36 - 0.40), when compared to the wet agglomerates obtained by using a low shear mechanical mixer (0.67 and 0.61, respectively) (Bellocq et al., 2017).

3.2. Impact of the process parameters of the fluidized bed

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3.2.1. Impact of the amount of water

The impact of the water content of the bed was studied by applying different processing times for water spraying. The physicochemical characteristics of the wet agglomerates were measured for each class of wet agglomerates according to their diameter, and plotted for the different measured true water content from 0.19 to 0.48 g/g dry matter (Fig. 5-7). Increasing the water content did not significantly change the shape of the curve of the size distribution, but generated higher amounts of the fractions of the large agglomerates (Fig. 5). An increase in the water content improved the capability of the fluidised bed to produce higher amounts of the wet agglomerates of larger diameter. The mass fraction of agglomerates between 1000 and 2000 µm reached a value of 66% when processed at 0.48 g / g dry matter.

An increase in the water content induced an almost linear increase in the median values (d_{50} from 349 µm to 1032 µm) of the diameter (Fig. 6). The values of the diameter span were constant (from 1.49 to 1.10) with true water content from 0.24 to 0.42 g/g dry matter. It then increased with values of water content higher than 0.37 g/g dry matter.

The relationships between the diameter and measured water contents for the different wet agglomerates did not change (Fig. 7a), whatever the amount of added water. There were positive relationships between diameter and water content of the wet agglomerates. The large agglomerates were more hydrated than the small ones.

The impact of an increase of the amount of added water on the measured water content of the wet agglomerates depended on their diameter (Fig. 7a). Increasing the amount of water largely increased the water content of the agglomerates. We could suppose that the fluidized bed homogeneously distributed the added water over the small grains and the grains of intermediate size.

An increase in the amount of added water decreased the compactness of the wet agglomerates, irrespectively of their diameter. Nevertheless, increasing the amount of added water did not significantly change the dependence observed previously, between the compactness and the diameter of the wet agglomerates (Fig. 7b). The compactness decreased with an increase in the median diameter until a median diameter close to 1600 µm. The compactness then slightly increase with an increase in diameter above 1600 µm. Similar phenomena were observed whatever the amount of added water added during processing.

Whatever the amount of added water, the large agglomerates were more spherical than the small ones (Fig. 7c). An increase in the amount of water did not have a significantly impact

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on the sphericity of the wet agglomerates. The wet agglomerates were characterized by the same values of sphericity, depending on their diameter.

3.2.2. Impact of the particle size of the native semolina

We evaluated the influence of the diameter of the native raw material, by comparing the agglomeration behaviour of the standard semolina (d_{50} = 280 µm) and coarse semolina (d_{50} = 619 µm) (Fig 8-9). It was possible to produce wet agglomerates by the fluidised bed using the coarse semolina. Using coarse semolina favoured the production of larger wet agglomerates (Fig 8), with higher value of median diameter (d_{50} = 1273 µm) and similar span (2.11), when compared to those previously determined using the standard semolina (d_{50} = 650 µm and span = 2.27). It is recognized that the wet agglomeration process increases the diameter of the structures of approximately a decade, regarding to the size of the native particles (T. Ruiz, 2012).

Using the coarse semolina gave wet agglomerates with lower values of water content and compactness (Fig 9a-9b). By incorporating the same amount of water in the fluidized bed (100 g), we could expect the same measured water content in the bed and for the wet agglomerates at the end of the wet agglomeration, whatever the type of semolina. However, the measured water content of the collected powder is close to 0.30 ±0.04 g/g dry matter, which is broadly lower than the value measured using the standard semolina (0.37 ±0.04 g/g dry matter). The lower values of compactness of the wet agglomerated of coarse semolina could lead to higher water evaporation mechanisms during fluidized bed processing and to lower values of water content.

Using the coarse semolina significantly affected the values of sphericity (Fig 9c). A slight decrease in the values of sphericity was observed from the small to the large wet agglomerates, while a positive relationship was demonstrated for the standard semolina. The large agglomerates were less spherical than the small ones. It could be more difficult to form a spherical grain with fewer and larger particles.

3.3. Characterisation of the agglomerates after cooking and drying

After sieving, the wet agglomerates that were collected between the sieves 850 and 1400 µm were processed by two successive stages: steam cooking and final drying. The

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characterization of the agglomerates was conducted after the cooking stages and after the final drying stage (Fig. 10 and Table 2).

The steam cooking stage and the drying stage did not change the shape of the distribution curves of the diameters of the agglomerates of semolina (Fig. 10). The size distribution of the wet agglomerates was almost fixed by the wet granulation stage (Table 2) and only slightly affected by the steam cooking stage and by the drying stage. The median diameter of the wet agglomerates (d_{50} = 910 µm) was slightly increased by the subsequent steam-cooking stage (d_{50} = 1000 µm) due to partial water absorption and swelling mechanisms. The final drying stage slightly reduced the median diameter of the agglomerates through possible shrinking mechanisms (d_{50} = 950 µm). The results also demonstrated that the span of the distribution of the diameters of the wet agglomerates (0.47) was slightly increased by the cooking stage (0.53) and by the drying stage (0.64). It could be associated with some breakage or collapse mechanisms generated by these two stages, that increase the range of the diameters. The present results indicated that distribution of the diameters of the agglomerates of durum wheat was mainly controlled by the agglomeration process stage. This stage greatly controlled the total yield of dried agglomerates production of correct diameter, according to the classic couscous manufacturing.

The steam-cooking stage induces swelling caused by hydration. This phenomenon reduced the compactness (0.30), which corresponds to the increase of the agglomerates volume exactly due to the increasing of the water content (0.45 g/g dry matter) (Table 2). The cooking stage promotes mechanisms of expansion due to water absorption and swelling mechanisms, which take place at the scale of the wheat components. These heat and mass transfers impact some physico-chemical changes under high temperature, such as starch gelatinization. The swelling of the agglomerates seemed to be more important than the progressive filling of their internal pore by the water molecules.

The drying stage induced an increase in the compactness (0.49) and a decrease in the water content (0.14) of the agglomerates (Table 2). The dewatering generates slight mechanisms of shrinking, which induce a decrease of the internal porosity and diameter of the agglomerates. The drying stage contributes to the texturation of the grains by the densification of the structure.

We observed that swelling and shrinking mechanisms generated by the successive steam cooking and drying stages did not significantly affect the sphericity of the agglomerates (Table 2). Water penetration and water extraction were in isotropic condition of the transfers in this case. The sphericity ranged from 0.687 after the wet agglomeration stage and 0.662

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after the drying stage. The wet agglomeration stage mainly determined the sphericity of the agglomerates.

The microstructure of the dried agglomerates were observed by scanning electron microscopy and compared to those of industrial couscous grains (Fig. 11). The dried agglomerates of durum wheat semolina processed by the fluidised bed are characterized by irregular shape, with very rough surfaces. The native semolina particles were clearly visible and distinguishable in the dried agglomerates: they appeared as semi discrete particles glued to each other. The dried agglomerates were not dense objects as internal porosity between adjacent particles was observed demonstrating that solid fusion between the semolina particles had not occurred. On the other hand, the classical couscous grains were characterized by spherical shape with regular surfaces. The native semolina particles were poorly visible in the grains, as they were partly glued to each other. The grains were almost dense objects, as only little internal porosity can be observed demonstrating that solid fusion between the semolina particles had largely occurred.

The mechanical properties of the agglomerates were measured by the mean force required to compress them (Table 3). As expected, the wet agglomerates were characterized by lower values of mechanical strength. The cooking and drying stage improved the mechanical resistance of the agglomerates by increasing the mechanical strength (from 1.23 to 4.25 N). However, the force required compressing the dried agglomerates produced by the fluidized bed processing remained significantly lower than those measured for the classical couscous grains (23 N).

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4. Discussion

Granulation process by using the fluidized bed technology generates agglomerates of durum wheat semolina with specific functional attributes. The present results describe the potentialities of the fluidized bed to agglomerate wheat powders and investigate the process - structure - properties relationships. The final properties of the dried agglomerates can be discussed in regards with the classical couscous grains that are granulated by using low shear mixers.

4.1 Agglomeration mechanisms

Whatever the investigated process conditions using the standard semolina, the growth of the wet agglomerates was characterized by one positive correlation between water contents and diameters (Fig. 7a), and by a specific diameter (d_{50} = 1600 µm) that separated two correlations between compactness and diameter: a negative slope at $d_{50} \leq 1600$ µm and a positive slope at $d_{50} \geq 1600$ µm (Fig. 4b and 7b).

For the $d_{50} \leq 1600$ µm, a power law (Eq. 3) can be used to describe the relationship between the compactness and the median diameter:

$$\phi = A_i d_i^{(D_f - 3)} \quad (3)$$

where A_i is a prefactor characterizing the amplitude of the phenomenon and D_f is the mass fractal dimension linked to the growth mechanism of the grain. The existence of a power law relationship between the diameter and the compactness was demonstrated by Rondet et al. (2010) during the wet agglomeration of various powders (kaolin, microcrystalline cellulose, and calcium phosphate) using low shear mixers. Our results could be interpolated with a good agreement by the power law (R^2 from 0.68 to 0.98) (Table 4). For $d_{50} \leq 1600$ µm, the calculated values of the D_f factor (2.81 - 2.90) were not affected by the changes in the water content. We only observed a decrease in the A_i values (from 0.48 to 0.32) when increasing the water content. The prefactor is linked to the nuclei characteristics (compactness and median diameter). Explanation of this variation needs to characterise the nuclei, elementary association of semolina, which lead to the agglomerate structure by association, unrealised in this study. By this analyse, mechanism for the wet agglomeration using the fluidized bed in the cited process conditions can be considered as fractal morphogenesis for the smaller

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structures ($d_{50} \leq 1600$ µm). Saad et al. (2011) also found a fractal-structuring model to describe the agglomeration of durum wheat semolina using a low shear mixer with a value of $D_f = 2.89$ and $A_i = 0.56$.

For $d_{50} \geq 1600$ µm, the value of the compactness slightly increased with the diameter of the agglomerates (Fig. 4b and 7b). This increase in compactness could be associated to the values of D_f close to 3. We could then suppose that some phenomena of densification of the grains occurred for the larger structures ($d_{50} \geq 1600$ µm) due to collisions with the wall and/or between the agglomerates themselves in the fluidized bed velocity field.

Considering the coarse semolina, the calculated value of $D_f = 2.56$ was lower than this obtained with the standard semolina (Fig. 9b). The lower value of D_f could indicate that the agglomeration process induces less mechanisms of densification on the investigated range of water contents.

Two types of agglomeration mechanisms can be considered. The first type is the association of the native semolina particles that generates nuclei at the beginning of the wetting/mixing process. Nuclei are small and compact. Nuclei association leads progressively to form larger structures, called agglomerates. Agglomerates growth is characterized by an increase in their size and a decrease in their compactness. The power law model highlights the interdependency between compactness and size until 1600 µm. The fractal growth model allowed describing the increase in the median diameter along with the decrease in compactness. A second type of mechanisms of densification of the grains occurred for the larger agglomerates due to collisions with the wall and/or between the agglomerates themselves. Several structures could be distinguished in the fluidized bed, according to their sizes, compactness and water contents: native semolina, nuclei, small agglomerates (<1600 µm) and large agglomerates (>1600 µm).

4.2 Hydrotextural analysis

The values of the physico-chemical characteristics of the wet agglomerates can also be discussed according to the hydrotextural analysis (Ruiz, Delalonde, Bataille, Baylac, & Dupuy de Crescenzo, 2005). The wet agglomerates were considered as the superposition of three dispersed phases: solid particles, liquid and gaseous phases. Three non-reduntant variables provide a description of the system in term of mass and volume. The easiest chosen variables are generally the apparent volume (V), the mass of water (M_w) and the mass of solid

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(M_s). These variables were used to calculate the water content, the compactness and the saturation degree.

The hydrotextural diagram is limited in its upper part by the saturation curve, which represents the maximum water content that a grain of a given constant compactness can contain (Fig. 12). The saturation curve is calculated using Eq. (4):

$$\phi_{sat} = \frac{1}{1+d_s^* w_{sat}} \quad (4)$$

Whatever the process conditions, the values of water content and compactness measured for the wet agglomerates were located under the saturation curve (Fig. 12). The wet agglomerates were porous structures at the scale of semolina particles (intraparticle porosity) and between particles (interparticle porosity). The range of variation of the compactness is essentially due to the particle re-arrangment. During the add of water, the filling of the interparticle voids did not occur by a simple remove of the air trapped. Capillary forces induced a re-arrangment between the particles that was observed by a variation of the compactness (Fig. 12) and the saturation degree.

The saturation degree increased (from 0.20 to 0.44) with an increase in the water content: experimental values became closer to the saturation curve (Fig. 12). It was observed that the growth could be fitted by a sigmoidal curve, comparable to this purposed by Ruiz et al. (2005) :

$$S(w) = 1 - \frac{1}{1+e^{\frac{w-w_m}{d}}} \quad (5)$$

where w_m is the water content at S=50% and d is the reverse slope of the central quasi-linear section of the sigmoidal curve. The relationship between water content and compactness (Fig. 12) means that inter granular arrangements led to an expansion followed by a densification of the wet agglomerates. The first stage of expansion was induced with an addition of water: the compactness decreased significantly and the saturation degree slightly increased. During the second stage, the structures continued to grow up but with densification mechanisms due to the capillarity influence. During this stage, the compactness slightly changed but the saturation degree highly increased, which correspond to a high filling of the inter granular voids. By associating Eq. (2) and Eq. (5), it was possible to describe the variation of the

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compactness by the water content, the real densities of the solid and liquid phases and the two parameters (w_m and d):

$$\phi(w) = \frac{1}{1+d_s^* w(1-e^{-\frac{w-w_m}{d}})} \quad (6)$$

This relationship (Eq. 6) reduces to one (water content) the number of state variables necessary and sufficient to describe the structure of the wet agglomerates of durum wheat semolina produced by using the fluidized bed. In all case, our results could be interpolated with a good agreement (R^2 = 0.93) by the relationship using Eq. (6), w_m = 0.822 and d = 0.513. Our experimental results did not investigate the properties of the wet agglomerates beyond this stage. Consolidation mechanisms could follow the stage of densification (Ruiz et al. 2005). The values of water content and compactness of the agglomerates measured after the agglomeration, cooking, and drying stages were presented on the hydro-textural diagram (Fig. 13). The cooked and dried agglomerates were not dense grains as they remain under the saturation curve. It can be observed that the phenomenon of retraction during the drying stage increased the compactness of the grains. The dried grains of wheat semolina produced using the fluidized bed technology were significantly different from the classical couscous grains, which were more compact and almost saturated.

4.3 Comparison with the couscous grains

The dried grains of durum wheat semolina produced by the fluidized bed were more brittle and less compact than the classical couscous grains. Using the fluidized bed technology, the water addition stage was critical as it promotes the cohesion between the particles and the physicochemical reactivity of the wheat components. The simultaneous agglomeration and drying mechanisms restricted the contribution of the physicochemical reactivity of the wheat components at the surface of particles, which were briefly in contact with water. Adhesion between the particles could mainly comes from physical forces, as capilarity forces, and liquid bridges involving superficial mechanisms. This may explain the lower compactness in the grain prepared by fluidized bed processing. It can be supposed that partial starch gelatinisation mechanisms only occurs at the interface between the particles. The resulting strength to compress the granules was significantly lower than those measured for the classical couscous grains. The classical couscous grains were more compact and consolidated

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by complete starch gelatinisation during the cooking stage. The fluidized bed technology produced agglomerates of durum wheat semolina with specific functional attributes, which were significantly different from those associated with the couscous grains.

4.4 Process efficiency

The process efficiency is related to the mass fraction of wet agglomerates with diameter between 1000 and 2000 µm collected after the granulation stage. It is admitted that the wet agglomeration stage controls for the process efficiency of the manufacture of the industrial couscous grains (Abecassis et al., 2012). The classical low shear mixers generate low process yield, with only 30 to 40% of grains with correct diameter and high amounts of out of specification wet grains that do not have the correct diameter. In the present study, the fluidized bed technology allowed reaching high values of yield of agglomeration, close to 66%. The fluidized bed granulation process generated low dispersion in the diameter of the wet agglomerates, with a low proportion of the too large grains (only 10%), compared to those obtained by a low shear mixer (20-25%). Fluidized bed technology could be a novel way to produce high amounts of agglomerates of durum wheat semolina.

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5. Conclusion

The agglomeration process using a fluidized bed has been found to produce agglomerates of durum wheat with different attributes compared to those produced by granulation using low shear mixers. The dried agglomerated grains obtained using the fluidized bed were less compact than the traditional couscous grains manufactured using low shear mixers. Fluidized bed granulation allowed producing higher amounts of agglomerates with diameter between 1000 and 2000 µm. Whatever the process conditions, the relationship between the compactness and the median diameter can be described using a power law model. It allowed demonstrating that agglomeration is driven by a fractal formation process for the wets agglomerates with $d_{50} \leq 1600$ µm, and by densification mechanisms for the large agglomerates ($d_{50} \geq 1600$ µm). This result indicates that the mechanisms which occur in the fluidized bed present analogy between low shear mixer agglomeration. By using the hydrotextural diagram, we proposed a specific model to describe the interparticle arrangements, which associated expansion and densification of the wet agglomerates. The relationship between the saturation degree and the compactness of the wet agglomerates was reduced to only one state variables to describe the agglomeration mechanisms of durum wheat semolina using a fluidized bed. This result is interesting because by using Eq. (3) and Eq. (6), we could predict the variation of the median diameter of agglomerates at different water contents. Investigation of the nucleation phase needs to be conduct to describe the agglomeration mechanism with more details. As in the case of agglomeration in high shear mixer, nucleation regimes could be identified in relation to the hydrodynamic conditions of the fluidized bed process.

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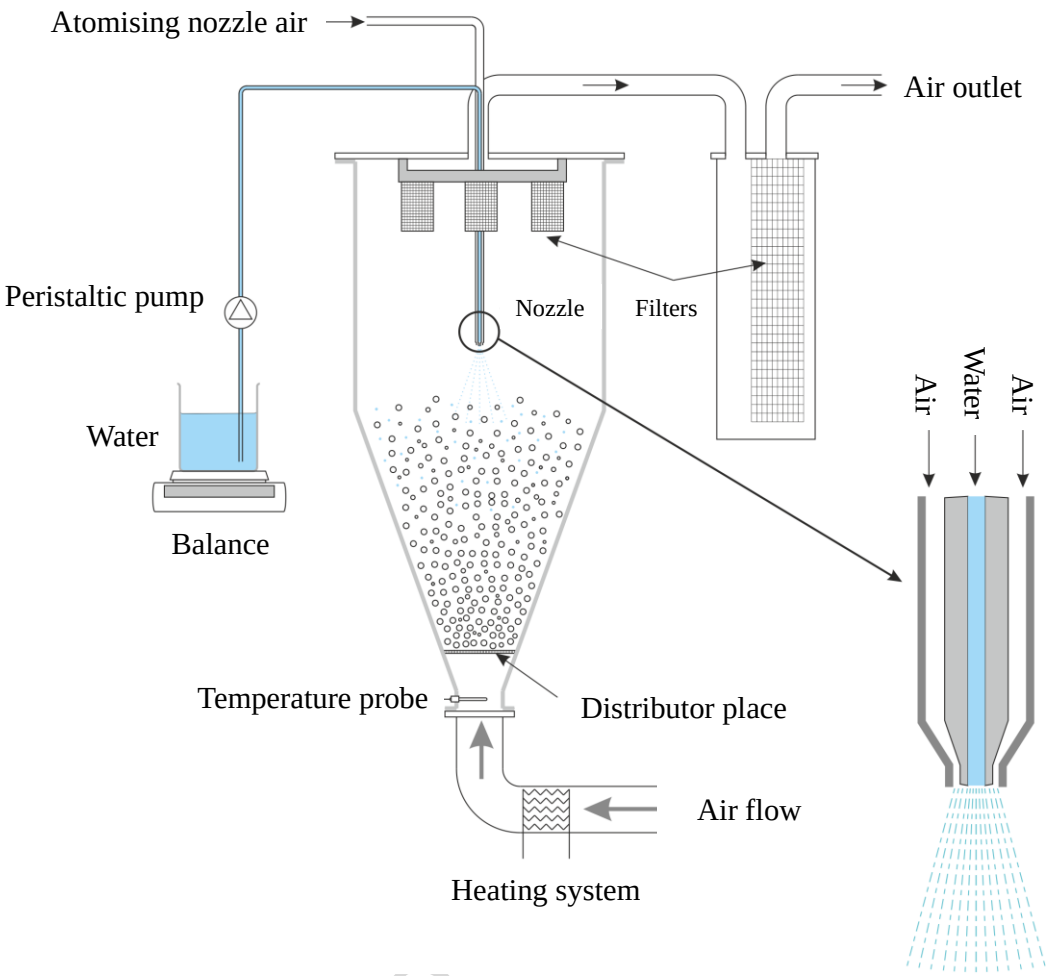


Fig. 1. Schematic description of the fluidized bed equipment.

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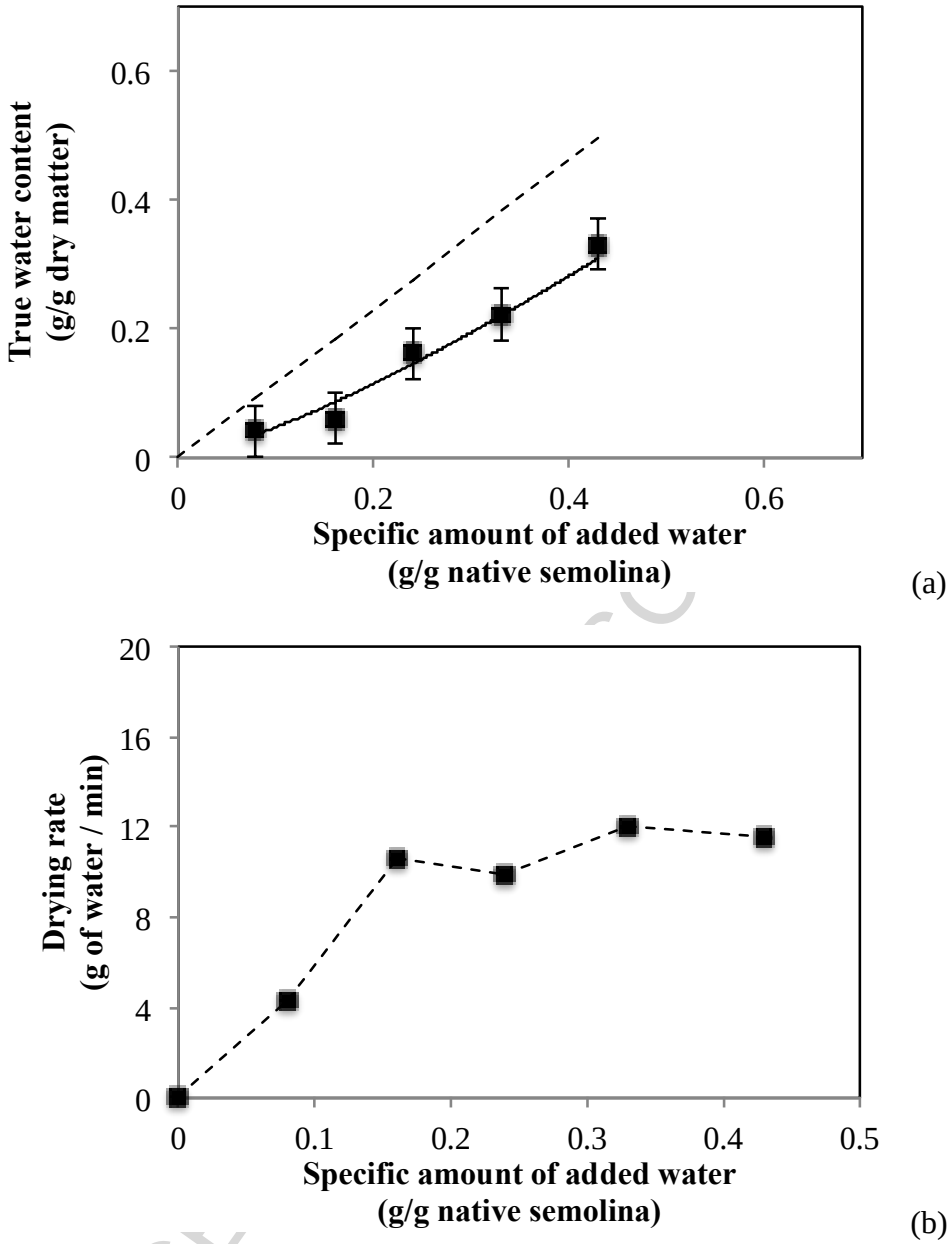


Fig. 2. Experimental curves of the true water contents (a) and the drying rates (b) as a function of the specific amount of added water in the fluidized bed (the dotted line in figure (a) corresponds to the apparent process water content).

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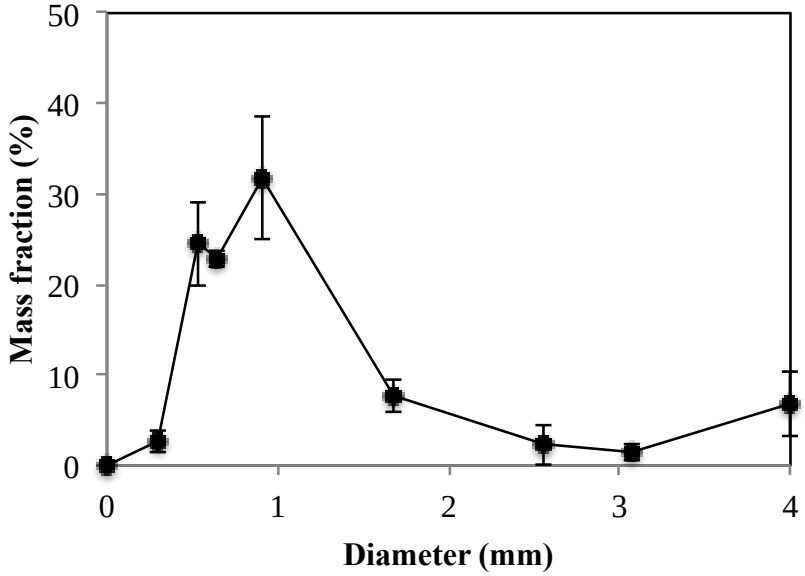


Fig. 3. Distribution curve of the diameters of the wet agglomerates processed by the fluidized bed (experiments using standard semolina and true water content of 0.37 g/g dry matter).

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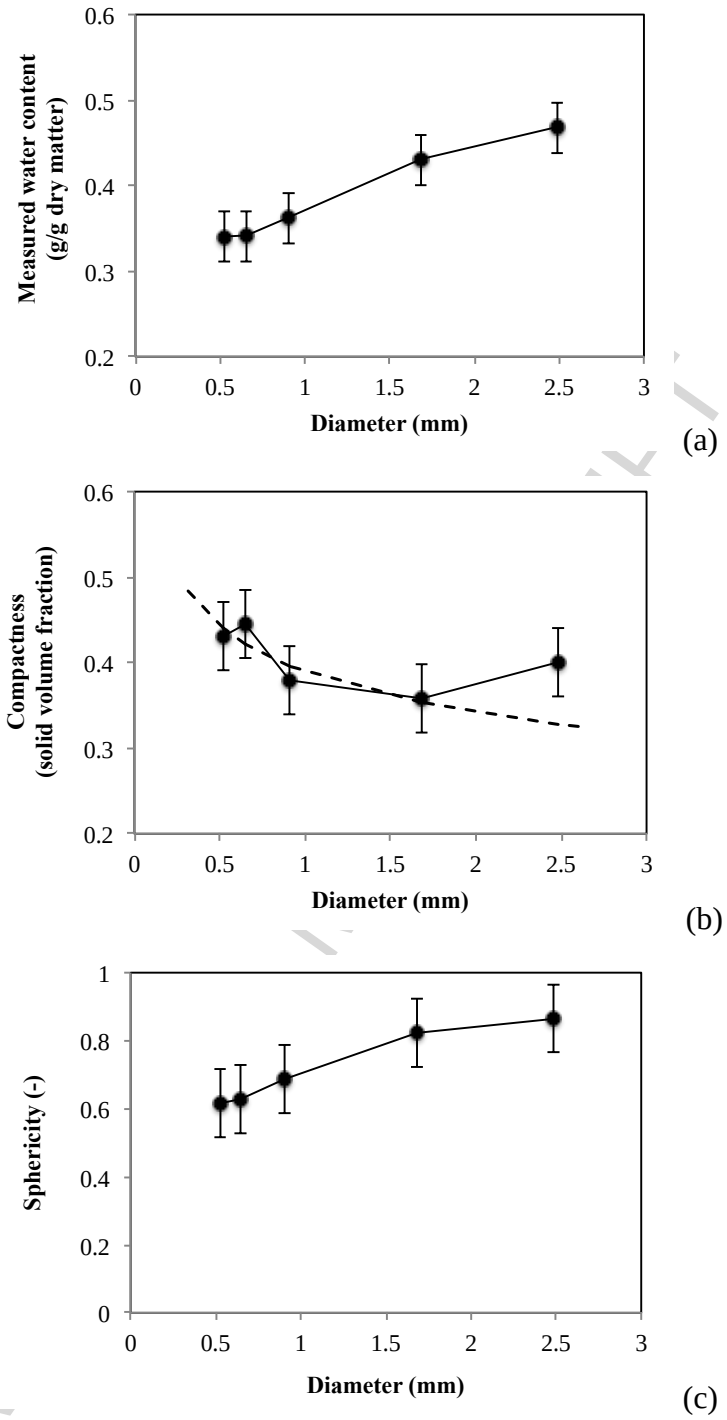


Fig. 4. Distribution curves of the measured values of water content (a), compactness (b) and sphericity (c), as a function of the diameter of the wet agglomerates processed by the fluidized bed (experiments using standard semolina and true water content of 0.37 g/g dry matter). (The dotted line in figure (b) is the fractal model using Eq. (3))

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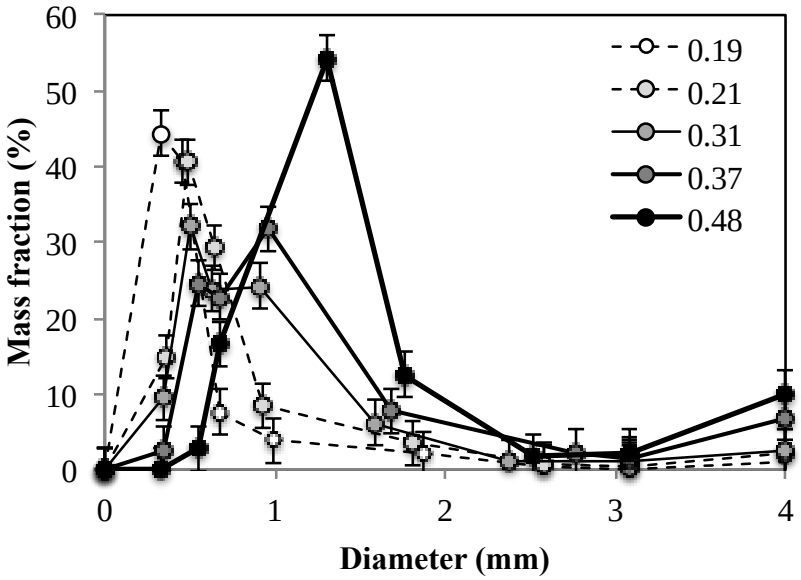


Fig. 5. Impact of the true water content on the distribution curves of the diameters of wet agglomerates processed by the fluidized bed (experiments using standard semolina).

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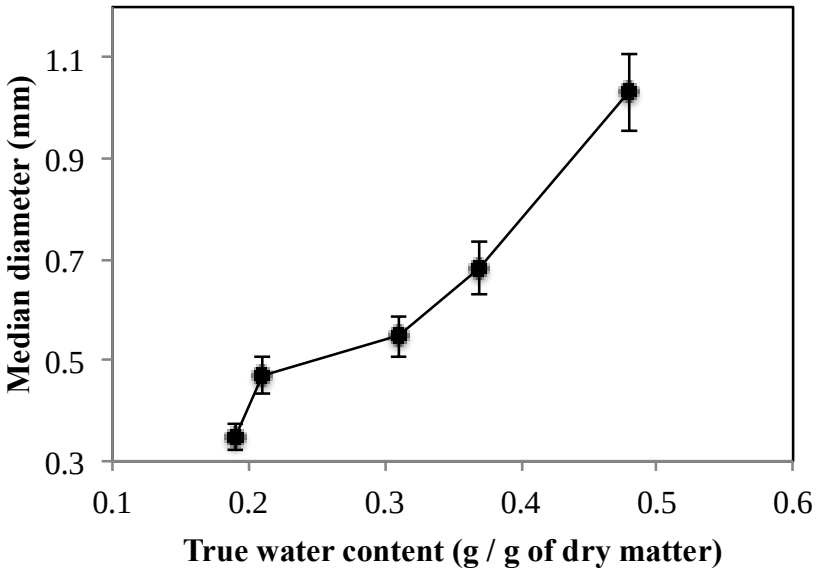


Fig. 6. Impact of the true water content on the median diameters (d_{50}) of wet agglomerates processed by the fluidized bed.

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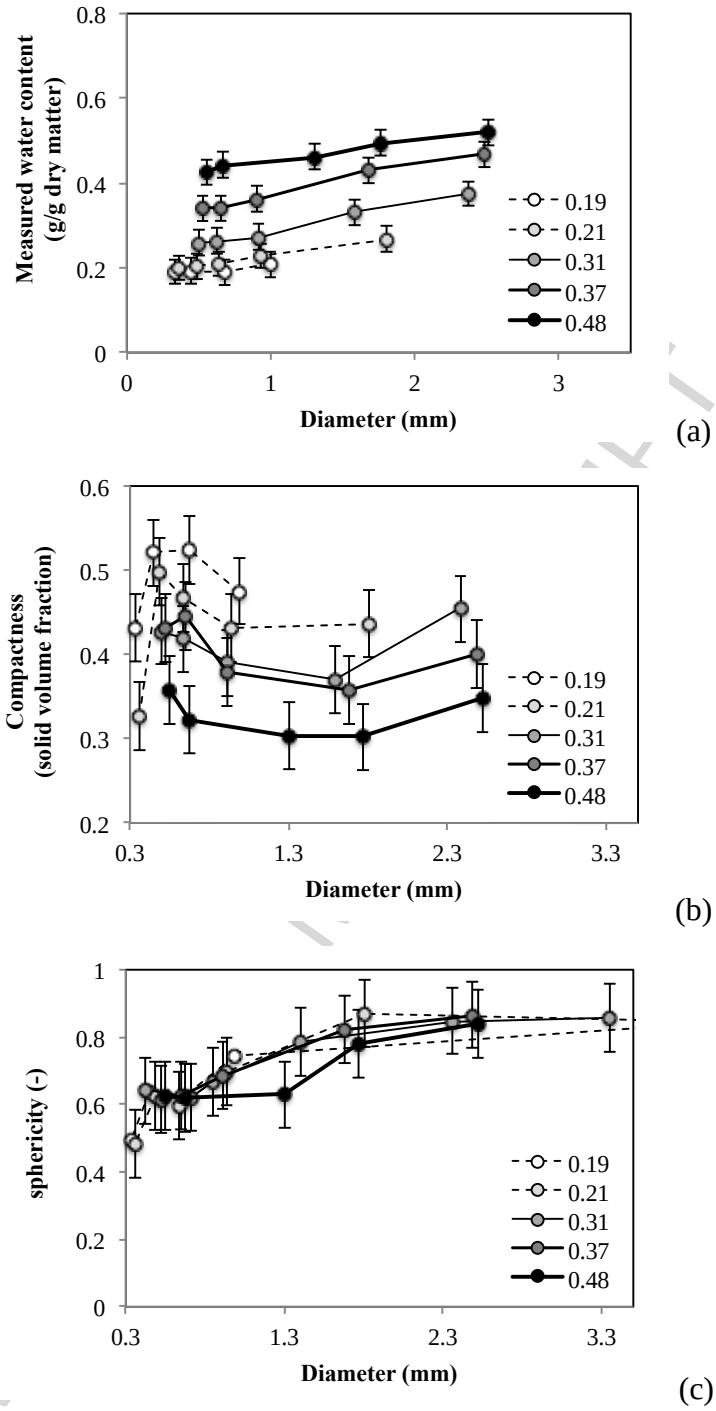


Fig. 7. Distribution curves of the measured values of the water content (a), compactness (b), and sphericity (c), as a function of the diameter of wet agglomerates processed by the fluidized bed at different true water contents (from 0.19 to 0.48 g / g of dry matter).

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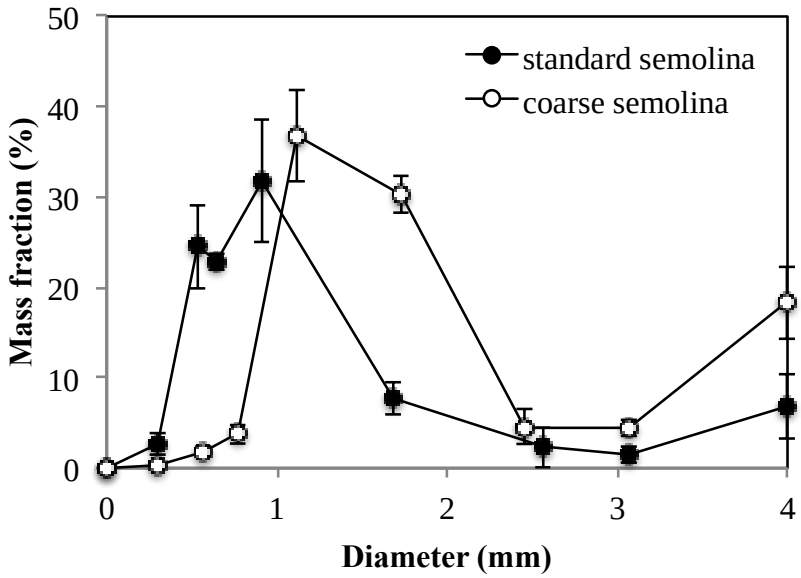
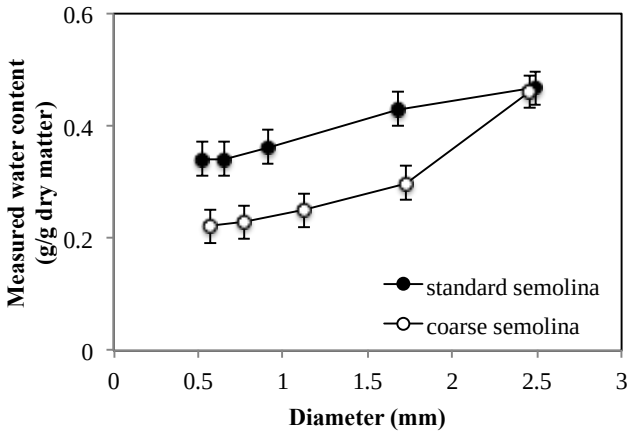


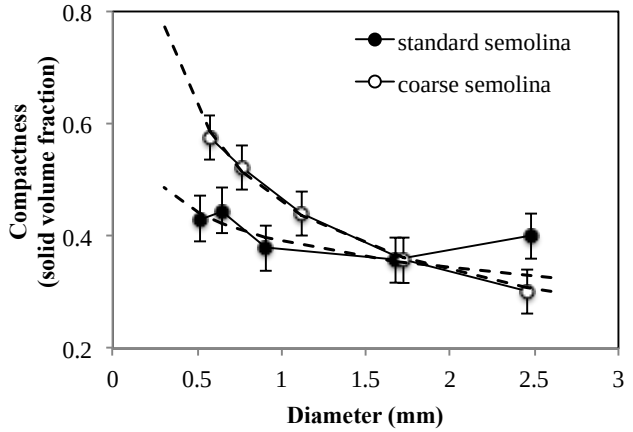
Fig. 8. Impact of the diameter of the semolina on the distribution curves of the diameters of wet agglomerates processed by the fluidized bed.

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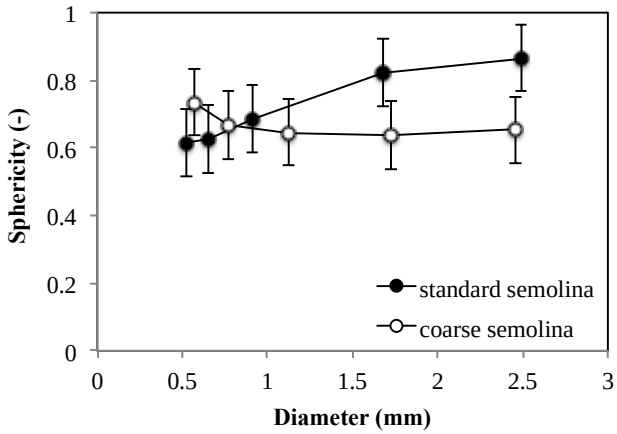
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(a)



(b)



(c)

Fig. 9. Impact of the diameter of the particles of semolina on the values of water content (a), compactness (b), and sphericity (c) as a function of the diameter of wet agglomerates processed by the fluidized bed. (The dotted lines figure (b) is the power law model using Eq. (3)).

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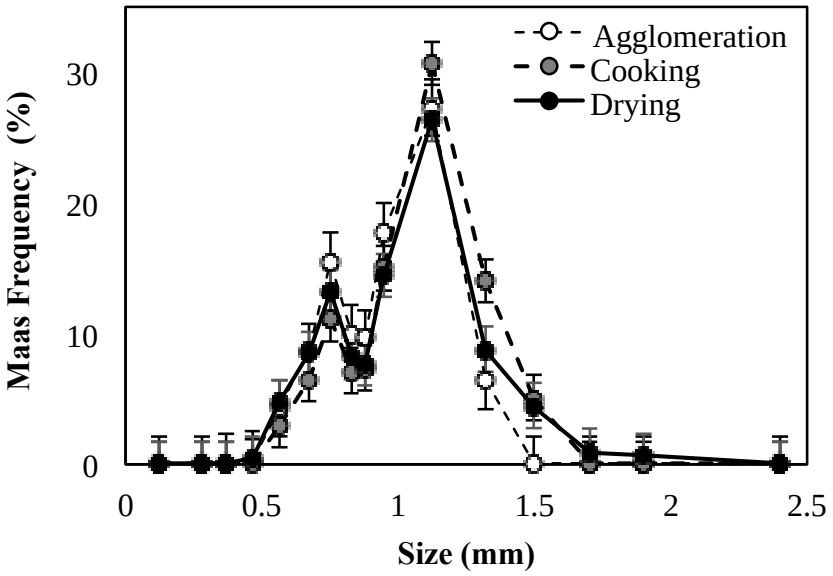
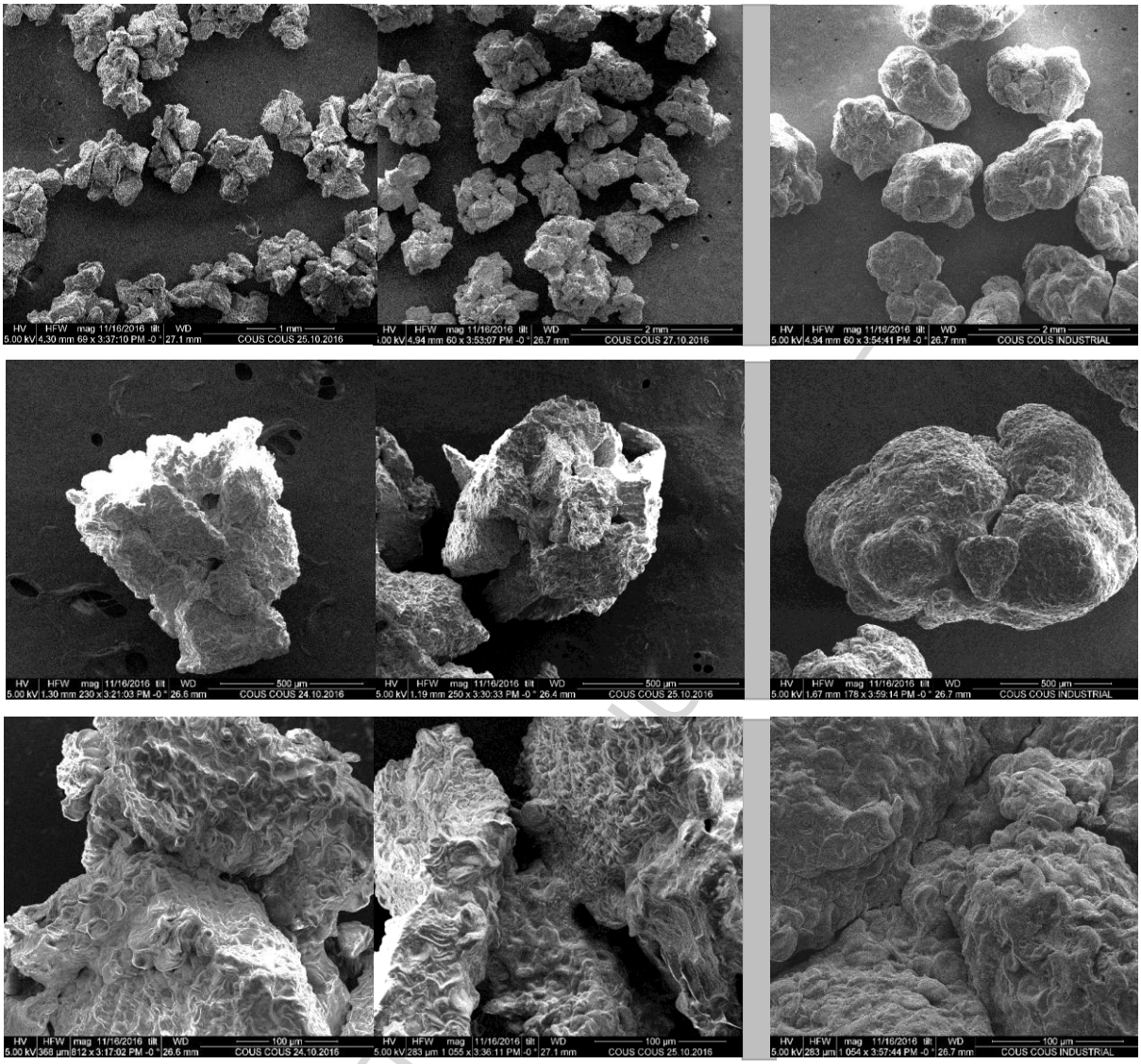


Fig. 10. Distribution curves of the diameters of the agglomerates after the agglomeration, cooking and drying stages (experiments using standard semolina and true water content of 0.37 g/g dry matter).

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(a) (b)

Fig. 11. Microstructure of the agglomerates based on of durum wheat semolina prepared with the fluidized bed (a) and commercial couscous grains (b) at different magnifications.

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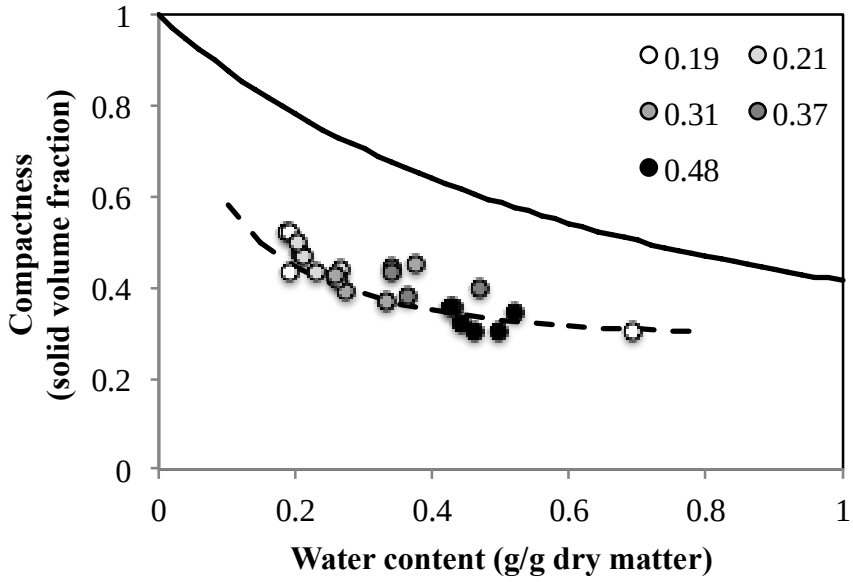


Fig. 12. Hydrotextural description (compactness vs. water content) of the wet agglomerates of durum wheat semolina according to the true water content. The black line represents the saturation curve. The dotted line represents the model curve (Eq. 6)

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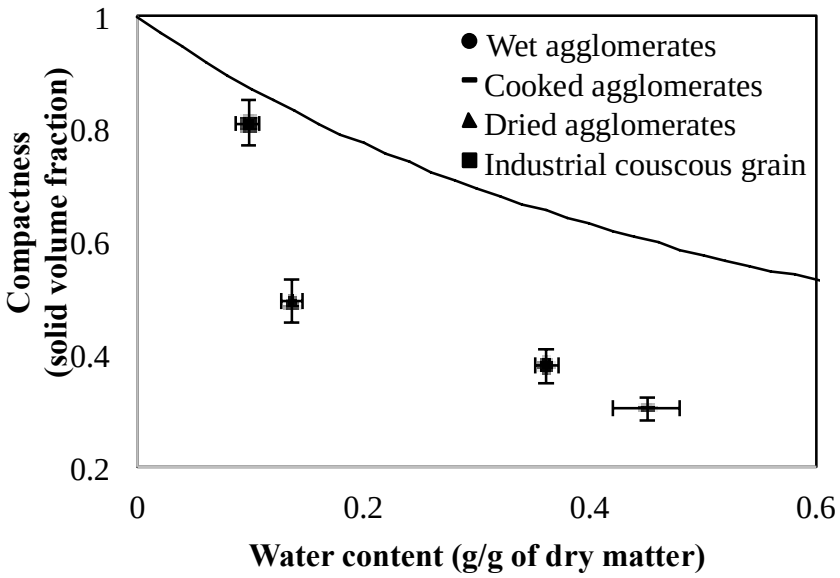


Fig. 13. Hydro-textural description (compactness vs. water content) of the agglomerates of durum wheat semolina after the agglomeration, cooking, or drying stage. Comparison with an industrial couscous grains.

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Table 1

Physicochemical characteristics of the two-selected durum wheat semolina.

	Standard semolina	Coarse semolina
Water content (g/g dry semolina)	14.8 (±0.5)	15.0 (±0.5)
Protein content (g/g dry matter	12.4 (± 0.1)	11. 6 (± 0.1)
Particle diameter - d_{10} (µm)	66 (±1)	434 (±1)
Particle diameter - d_{50} (µm)	283 (±1)	619 (±1)
Particle diameter - d_{90} (µm)	542 (±4)	888 (±1)
True density	1.415 (±0.005)	1.453 (±0.003)
Apparent density	1.372 (±0.007)	1.414 (±0.006)
Compactness	0.845 (±0.009)	0.846 (±0.009)

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Table 2

Physicochemical characteristics of the agglomerates after the cooking stage and after the drying stage.

	d_{50} (mm)	Span (-)	Water content (g / g dry matter)	Compactness (-)	Sphericity (-)
Wet	0.91 *(±0.05)* [a]	0.47 *(±0.02)* [a]	0.36 *(±0.02)* [c]	0.38 *(±0.02)* [b]	0.687 *(±0.02)* [c]
Cooked	1.00 *(±0.01)* [b]	0.53 *(±0.02)* [b]	0.45 *(±0.06)* [d]	0.30 *(±0.03)* [a]	0.572 *(±0.05)* [a]
Dried	0.95 *(±0.02)* [a]	0.64 *(±0.02)* [c]	0.14 *(±0.01)* [b]	0.49 *(±0.04)* [c]	0.662 *(±0.01)* [b]
Industrial	1.60 *(±0.01)* [c]	0.56 *(±0,.1)* [b]	0.10 *(±0.01)* [a]	0.81 *(±0.04)* [d]	0.844 *(±0.01)* [d]

Values are means (± standard deviation).

Values in column with the same letter were not significantly different ($P<0.05$).

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Table 3

Comparison of the mechanical strength of the wet agglomerates and of the dried agglomerates of durum wheat semolina prepared with the fluidized bed. Comparison with commercial couscous grains.

	Strength (N)
Wet agglomerates	1.23 *(±0.89)* [a]
Dried agglomerates	4.25 *(±1.19)* [b]
Classical couscous grains	22.99 *(±5.60)* [c]

Values are means (± standard deviation).

Values in column with the same letter were not significantly different (P<0.05).

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Table 4

Model parameters, coefficients of correlation and absolute deviations for all the experiments, using the power law Eq. (3).

Semolina	w	A_i	D_f	R^2	Absolute deviation
Standard	0.19	0.48	2.87	0.69	0.25
	0.21	0.45	2.90	0.68	0.12
	0.31	0.39	2.87	0.98	0.12
	0.37	0.39	2.81	0.83	0.13
	0.48	0.32	2.87	0.81	0.10
Coarse	0.30	0.46	2.56	0.99	0.03

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Highlights

Fluidised bed technology is able to agglomerate particles of durum wheat semolina.

The final dried agglomerates are less compact than the traditional couscous grains.

A fractal model could be used to describe the growth mechanisms of the agglomeration.

The hydrotextural approach shows successive expansion and densification mechanisms.

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