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E. Rondet, Bernard Cuq, Denis Cassan, Thierry Ruiz

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Agglomeration of wheat powders by a novel reverse wet agglomeration process

Rondet E. [(1)], Cuq B. [(2)], Cassan D. [(2)] and Ruiz T. [(2)]

[1] UMR 95 QUALISUD, Université de Montpellier,
Faculté de Pharmacie, Laboratoire de Physique Moléculaire et Structurale, 15 avenue Charles Flahault, 34093 Montpellier cedex 5, France

[2] UMR 1208 IATE, Montpellier SupAgro, Université de Montpellier, INRA, CIRAD
2 place Viala, 34000 Montpellier, France
eric.rondet@univ-montp1.fr

Abstract - The wet agglomeration process is implemented to improve the powders functionalities. The objective of the present work is to investigate a novel reverse wet agglomeration process for reactive powders. This novel process is conducted in two main successive phases during mechanical mixing. The first phase consists of mixing a specific amount of powder and water to produce a continuous saturated paste, called dough, by controlling the physicochemical reactivity of the biochemical components of the powder. The second stage consists of generating the agglomerates by adding the powder inside the continuous dough under mixing conditions. This agglomeration process is mainly promoted by fragmentation mechanism. We have evaluated the impact of the process conditions on the electric consumption, agglomeration yield and characteristics of the agglomerates. Experiments were conducted using durum wheat semolina as raw materials. The results were compared to those obtained by using the classical wet agglomeration process, at same final water content of the agglomerates.

Keywords - Reverse agglomeration, fragmentation mechanisms, electric consumption, wheat powders, compactness, microstructure.

1. INTRODUCTION

Agglomeration is a unit operation, which is implemented to improve the powders functionalities relating to flow properties, dust generation, mixing capacity, solubility, *etc.*, by increasing the size of structures. During agglomeration, native particles are gathered to form larger assemblies, called the agglomerates. Wet controlled growth agglomeration refers to agglomeration processes during which a liquid binder is sprayed over an agitated powder bed (Ennis et al., 1991; Iveson et al., 2001). Knowledge about the agglomeration process of food powders still remains partial, although significant scientific works have been conducted over the last 15 years (Litster and Ennis, 2004; Saleh and Guigon, 2009; Palzer, 2011; Cuq et al., 2013). The description of the mechanisms, which are involved during the agglomeration of the food powders, still represents a real scientific stake.

The wet agglomeration of powders requires the addition of water to generate capillary bridges which adds cohesion between particles. Agglomeration mechanisms result from the spatial arrangement of the native particles with the binder components, promoting attractive interactions and links. The contribution of liquid bridges between native particles largely overtakes the physical forces and the van der Waals interactions (Hapgood et al., 2003). For food powders, besides the contribution of physical phenomena, the mechanisms also depend on the physicochemical reactivity of the molecules, which strengthens the adhesion forces between particles, by adding a significant contribution of the viscous forces (Cuq et al., 2013). When the food powders are subjected to the addition of water, and/or to mechanical stresses, the molecules are able to undergo the glass transition and/or irreversible physicochemical changes (*e.g.* solubilisation, denaturation, diffusion, *etc.*). The process conditions, including mechanical stress, temperature, water addition, and process time, greatly determine the effective contribution of the physicochemical reactivity of the molecules (Cuq et al., 2013). Based on their chemical composition, food molecules have the capacity to establish, between themselves and with the surrounding molecules, interactions of different energies. Low energy interactions include hydrogen bonds, hydrophobic interactions, and ionic interactions, while covalent bonds such as disulfide bonds represent high energy interactions. These interactions contribute to defining their capacity to react when subjected to water addition and/or heat treatments. For examples, the wheat proteins can establish continuous macromolecular network when subjected to water addition and mixing, and they can establish irreversible intermolecular covalent bonds, under heat treatments, that contribute to irreversible reticulation mechanisms (Abecassis et al., 2012).

Wet agglomeration is generally described as a combination of three processes at different rates: wetting-nucleation, consolidation-growth, and attrition-breakage (Iveson et al., 2001). The apparent rate of the agglomeration depends on the specific contribution of each processes. The growth mechanisms depend on the opposite contributions of cohesion and rupture mechanisms. The cohesion forces generate interactions between particles, while the rupture forces and the local shearing effects lead to breakage. The mechanisms of attrition and breakage help to improve homogeneity and strength of the granules by promoting consolidation, in addition to the obvious growth retardation (Reynolds et al., 2005).

Recent works have investigated the interest to consider the mechanisms of breakage and fragmentation as a major way to produce agglomerates. Rondet (2008) was the first to develop a reverse agglomeration process applied on three different powders (microcrystalline cellulose, calcium phosphate, and kaolin) classically used for pharmaceutical applications. The first stage of the process is the preparation of a wet saturated paste by mixing the native powder with high amounts of water. The second phase consists of adding the native powder inside the paste during mixing, in order to promote the fragmentation mechanisms and generate the agglomerates with controlled water content. The authors demonstrated the reversibility of the paste transition, between the discrete granular state and the continuous paste state. The paste transition was identified at a specific range of water contents, close to the plastic limits of the powders. The agglomerates are more spherical and compact than those produced by the classical wet agglomeration process.

In 2010, a reverse wet granulation process was patented for pharmaceutical uses (Li et al., 2010). Granules are formed by incorporating a mixture of dry powders (*i.e.* the excipients) into the drug-polymer slurry (*i.e.* the active pharmaceutical ingredient), and by drying the products. The granules formed comprise a core containing the active pharmaceutical ingredient having poor aqueous solubility.

Recently, Wade et al. (2013, 2014, 2015) developed a novel reverse-phase wet granulation process for pharmaceutical applications, based on the immersion of the dry powder mix, into the binder liquid. From a slurry state, the addition of the powder mass enables mechanical distribution of binder liquid to take place, until the desired particle size is obtained, through controlled breakage. The main mechanisms are supposed to be breakage of the large saturated agglomerates. The binder liquid is dispersed throughout the powder by mechanical distribution. This novel approach is supposed to have the potential advantages of eliminating the traditional variations associated to the stage of granule nucleation, and of reducing the

potential risk of uncontrolled growth of the granules. They evaluated the effects of: liquid saturation, binder liquid viscosity, and impeller speed on the physical and end-uses properties of the granules.

The objective of the present paper is to investigate the novel reverse wet agglomeration process by fragmentation of a pasty state, in the specific case of reactive powders, able to form a continuous dough stabilized by a molecular protein network. Durum wheat semolina was used as reactive powder for the present study. The novel process is conducted as two main successive phases, during continuous mechanical mixing. The first stage consists of mixing a specific amount of powder and water to produce a continuous saturated paste, called the dough, by controlling the physicochemical reactivity of the biochemical components of the powder. This paste could not be considered as a granular suspension because the original granular state of the native particles is lost. While the cohesion of the granular paste is almost reversible in the previous works (Rondet, 2008), the presence of reactive proteins of the wheat (i.e. gluten) in the particles of durum wheat semolina led the formation of a dough stabilized by a biochemical molecular network and the irreversible loss of the native granular state. The second stage consists of ~~in~~ generating the agglomerates, by adding the dry powder inside the continuous dough, during the mixing. The fragmentation mechanisms induce dough disruption and agglomerates production.

We have evaluated the impact of the process conditions on the electric consumption, agglomeration yield and characteristics of the agglomerates. Experiments have been conducted by using durum wheat semolina as raw materials. Durum wheat semolina is a reactive powder able to form a continuous protein network, when it is wetted and mechanically sheared (Pollini et al., 2012). The physicochemical reactivity was controlled by the water content of the dough. Two conditions have been tested: low reactivity at low water content of the dough (above the plastic state) and high reactivity at high water content of the dough (above the pseudo-liquid state). The results are compared to those obtained by using the classical wet agglomeration process, at the same final water contents of the agglomerates.

2. MATERIAL AND METHODS

2.1. Raw materials

Durum wheat semolina of industrial quality (Panzani group, France) was used as raw material for the agglomeration experiments. Semolina was stored in hermetic containers at 4°C until experiments were carried out.

Semolina was first characterized using standardized methods. The water content of semolina (16.4 ±0.5 g water/100 g dry semolina) was determined according to the approved method 44-15A (AACC, 2000), by weighing after oven drying (RB 360, WC Heraeus GmbH, Hanau, Germany) at 105°C for 24 h. The total nitrogen content (TN) of semolina was determined by the Kjeldahl method, and the crude protein content (12.4 g protein/100 g dry matter) was calculated according to TN x 5.7 based on the AFNOR method V 03-050 (AFNOR, 1970). Median value of particle diameter of semolina (d_{50} = 287 ±8 µm) was measured by using laser granulometry (Coulter TMLS 230, Malvern, England) at room temperature. The diameter span ((d_{90}-d_{10})/d_{50}) was 1.56. The semolina true density (1.478 ±0.005 g.cm^{-3}) was measured by helium pycnometry.

Liquid limit and plastic limit, which correspond to Atterberg tests used in soil science (Holtz and Kovacs, 1981) are carried out on semolina in accordance with the French P 94-051 and P 94-052-1 methods (AFNOR, 1993, 1995). For granular media, the plastic limit (w_P) is the water content which gives the transition between the solid and the plastic state. The liquid limit (w_L) is the water content which gives the transition between the plastic and the pseudo-liquid state. The plastic and liquid limit values are measured at respectively 59% and 76% for the durum wheat semolina. The plasticity index (w_L-w_P) is equal to 17% and allows assimilating the durum wheat semolina at a plastic organic soil like plastic silt (Holtz and Kovacs, 1981).

2.2. Agglomeration processes

The present work specifically investigates the effect of two different agglomeration paths (the direct classical wet agglomeration process and the reverse agglomeration process), by using a low shear planetary mixer (Fig. 1).

The direct classic wet agglomeration process is conducted by spraying water directly on the native powder inside the mixer bowl, during the mechanical mixing (Fig. 1). Water spraying is conducted until reaching the defined water content (i.e. the process water content), of the wet agglomerates (30, 40, or 50%).

The reverse agglomeration process is conducted with two successive steps. A part of the native semolina powder is first mixed in the mixer bowl. The first step consists of generating the continuous wet dough in the mixer bowl, by adding the defined amounts of

water on the native semolina, during the mechanical mixing. Water addition is conducted until reaching the defined water contents, to produce the plastic dough (at 75% water content) or the pseudo-liquid dough (at 110% water content) (Fig. 1). The second step corresponds to the formation of the powders by fragmentation mechanisms, lead by the addition of the native powder. The native powder was poured on the free surface of the dough and incorporated inside by the mechanical mixing. The amount of the added powder is defined to reach the final water content of the wet agglomerates (30, 40, or 50%).

We tested 9 different experimental conditions, which are associated to the 3 process paths and to the 3 final water contents of the agglomerates. The reproducibility of the process paths was checked by realizing some experiments in triplicate, at least one for each different process path. Experiments were done by using a batch planetary mixer (Kenwood Major 1200), equipped with a fixed bowl of 5 liters and a K blade that rotates around a vertical axis. All the experiments were conducted with the constant final load (1.5 kg) of the wet agglomerates and the constant rotation speed of the blade (70 rpm), to promote the conditions of low shear and constant total time of mixing (12 min). The electric consumption of the mixer was continuously measured by using a wattmeter (Wattcom®). Before the start of the experiments, we have conducted a stage of no-load mixing, at 70 rpm during a long enough duration (i.e. 30 min) to reach steady state conditions of the electric consumption of the mixer (Fig. 2). The initial load of the native powder (Table 1) was rapidly poured inside the mixer bowl and mixed for 2 min. During the wetting stage (Fig. 2), performed during the mixing, water was directly sprayed on the free surface of the powder bed at constant flow rate (5.25 $mL.s^{-1}$) by using a flat spray nozzle (TPU650017, Spraying System Emani, France) connected to the water supply network. The diameter of the water droplets were estimated to 50-100 μm (Mandato et al., 2012). In these conditions, the dimensionless spray flux was estimated to be around 0.2. This value is slightly higher than the threshold value (0.1) that distinguishes the nucleation regime by the drop controlled, in the nucleation regime map as defined by Hapgood et al. (2003).

For the direct process, the spraying time ~~(30-63 sec)~~ was defined to incorporate the necessary mass of water (Table 1) to reach the water content of the final agglomerates. After the spraying phase, the wet agglomerates were mixed until a total time of process of 12 min (Fig. 2).

For the reverse processes, the first stage of the formation of the continuous dough is conducted during a constant time of 2 min. During this stage, the spraying time was defined to incorporate the necessary mass of water (Table 1) to reach the defined water content of the

continuous dough (Fig. 2). During the following fragmentation stage, the addition of the powder was conducted at constant feeding rate (8.7 $g.sec^{-1}$) by using a weight feeder (K-SFS-24, K-TRON, New Jersey, USA), which was directly placed above the mixer bowl. The time for the addition of the powder was defined to incorporate the necessary mass of powder (Table 1) to reach the water content of the wet agglomerates. After the powder addition phase, the wet agglomerates were mixed until a total process time of 12 min.

The wet agglomerates were characterized according to their size distribution immediately after the processing. 100 g of product was sampled inside the mixer and poured over a column of 11 sieves of decreasing mesh size (10, 5.6, 2, 1.25, 0.9, 0.8, 0.71, 0.63, 0.5, 0.4, and 0.315 mm). The sieve column was moved by gentle manual stirring for 1 min to separate the agglomerate fractions. The repartition of the fractions on each sieve was measured by weighing. The size distribution was expressed as the percent of total weight. Even if the particle size distribution curves were not 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, that could under or over-represent the trend.

2.2. Consolidation and stabilization of the agglomerates

After the agglomeration stage, the wet agglomerates were consolidated by the steaming stage and they were stabilized by the drying stage. The stages of steaming and drying were only conducted on the wet agglomerates that were sampled between the sieves of 1.25 and 2 mm mesh size. We made this choice, because it corresponds to one of the fields of application expected for our works: the manufacturing of the agglomerated couscous grains of durum wheat (Abecassis et al., 2012). Obviously, the knowledge of the properties of this fraction does not allow describing the properties of the larger or smaller fractions.

Immediately after sieving, the agglomerates were spread as a thin layer (about 3 mm) over a stainless steel plate, inside a steam cooker of 20 L (Ravant Chaudronnerie, France). The stage of steaming was conducted for 15 min at 100°C, under saturated steam flow of 20 $kg.h^{-1}$, and at atmospheric pressure. The steamed agglomerates were immediately dried as a thin layer (about 3 mm) over stainless steel mesh, by using a pilot scale dryer (Afrem, Lyon, France). The drying was conducted for 60 min at 70°C and at 80% relative humidity. The dried agglomerates were collected and stored inside hermetic plastic cups until characterization.

2.3. Characterization of the (wet and dried) agglomerates

Water content - The water content was determined on 3-5 g samples, by a drying method in an oven (RB 360, WC Heraeus GmbH, Hanau, Germany) at 105°C for 24 h (AACC Method 44-15A). Mean values were determined from triplicate.

Compactness - The solid mass (m_{Solid}; g dry matter) was first calculated from the weight values of the agglomerates (m; g) and their water content (w; g water / g dry matter)..

$$m_{Solid} = \frac{m}{1+w} \quad (1)$$

The density of the agglomerates (ρ; g.cm^{-3}) was determined by using a hydrostatic balance (Precisa serie 321 LX 120A SCS, equipped with kit for determining density for solid and liquid) and using paraffin oil. The solid volume fraction of the agglomerates (ϕ; cm^3 solid matter / cm^3) was calculated using Eq. (2).

$$\phi = \frac{V_{Solid}}{V} = \frac{\rho}{\rho^*_{Solid}(1+w)} \quad (2)$$

Where V_{Solid} is the volume of the solid matter of the agglomerates (cm^3 solid matter) and ρ^*_{Solid} is the true density of material (1.478 ±0.005 g.cm^{-3}) measured by helium pycnometry. Mean values were determined from triplicate.

Shearing properties - Shearing properties of the wet agglomerates were measured using the FT_4 powder rheometer (Freeman Technology, Worcestershire, UK) according to Rondet et al. (2013). Measurements were carried out in a cylindrical glass cell (diameter = 50 mm; height = 50 mm) and the FT_4 probe (with a helix of a -5° angle and a diameter of 23.5 mm). The sample was subjected to a pre-shearing stage (0.3°.s^{-1} on 60° with a pressure of 15 kPa) carried out with the FT_4 powder rheometer shearing probe. Four shearing cycles were carried out for various normal stresses (1.75, 1.5, 1.25, and 1 kPa), at a speed of 0.3°.s^{-1}. Failure shear stress was recorded for each studied normal stress. Undrained shearing tests were analysed using Mohr's representation of the plane stress state with the normal stress (σ, kPa) and the shearing stress (τ, kPa). The shearing properties were described using the Mohr-Coulomb rupture criterion.

$$\tau = \mu\sigma + c \quad (3)$$

where μ is the friction coefficient (dimensionless) and c is the cohesion (kPa). The friction coefficient is the slope and cohesion is the intercept with the y-axis of the linear relationship between σ and τ. Experiments were carried out in triplicate.

Scanning electron microscopy - The microstructure of the dried agglomerates was observed by scanning electron microscopy (JSM-T2000, JEOL, Tokyo, Japan). A sample was mounted in epoxy resin and coated by gold to provide the conductivity and the emission of secondary electron. For each sample, pictures with different magnifications were taken.

Color – A sample of dried agglomerates was densely packed in a cylindrical glass cell (6 cm diameter and 2 cm thickness) and levelled off. Color coordinates (L* lightness, a* redness, and b* yellowness) were measured directly by using a reflectance colorimeter (Konica Minolta CR - 410, France) with the calibration to daily light (D65). Colours measurements were carried out in triplicate on three different samples. The arithmetic means of the coordinates were calculated together with total color difference.

$$\Delta E = \sqrt{\sum (x_i - x_0)^2} \quad (4)$$

Where *x* is L*, a* or b*, and *i* refers to the different products and *0* means durum wheat semolina as standard.

Swelling capacity – The swelling capacity of the dried agglomerates was measured according to Guezlane and Abecassis (1991). A sample (20 g ± 0.1 g) was placed inside a graduated 100 mL test tube (25 cm long and 3 cm diameter) containing 50 mL of distilled water. The test tube was sealed and turned upside down 10 times successively. Water at 20°C was carefully added to wash down the agglomerates stuck to the internal sidewall of the test tube, until the final volume of the mixture reached 100 mL. The test tube was then placed at room temperature for 30 min. The volume of solid hydrated "phase" was directly read on the tube. Measurements were realized in triplicate. The swelling capacity criterion at 20°C ($ml.g^{-1}$) corresponded to the ratio the final volume of the hydrated agglomerates (V; ml) over the initial mass of agglomerates (M; g).

$$S_{20°C} = 100 \frac{V}{M} \quad (5)$$

2.4. 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 2011, at a 5% significance level.

3. RESULTS

The comparison of the direct or reverse agglomeration processes was conducted by both monitoring the energy consumption during the processing and by measuring the characteristics of the wet agglomerates and of the dried agglomerates.

3.1. Electric consumption during process

The electric consumption of the mixer was measured during the successive stages of the agglomeration processes. During the first stage of no-load mixing, we observed a small peak of electric input power, followed by a progressive decrease of the electric power until it reach a constant value, in steady state conditions (data not presented). According to Rondet et al. (2012), the electric input power during the no-load mixing is the sum of the mechanical output power for rotating the blade and of the dissipation terms (heat dissipation, motor magnetic dissipation, and motor mechanical dissipation). The measured values of the electric input power were normalized by subtracting the plateau value measured at the end of the stage of no-load mixing (Rondet et al., 2012).

During the mixing of the initial load of the native powder (Fig. 3), the normalized electric input power values are low. These values (Table 2) are correlated (R^2 = 0.988) with the weight of the native powder in the mixer tank before the water addition (Table 1), except when the initial load is too low (less than 400 g load), compared to the mixer size.

During the agglomeration stage, the curves of the normalized electric input power are specific of the process. For the direct agglomeration process (Fig. 3A), the electric input power rapidly increases during the spraying of the water (stage 3), due to the increase in cohesion between the particles (Rondet et al., 2013). The electric input power reaches a maximum value at the end of the stage of water spraying, and then progressively decreases during the final stage of mixing, and reaches a final plateau value.

For the reverse agglomeration processes (Fig. 3B-C), we first observe an increase in the electric input power during the stage of water spraying (stage 3), due to the formation of the continuous dough. For the plastic dough conditions (Fig. 3B), the power curve is characterized by a high peak and large bandwidth due to the high consistency of the dough. For the pseudo-liquid dough conditions (Fig. 3C), the power curve is characterized by a slight increase and small bandwidth, due to the liquid behaviour and low consistency of the dough. When starting the final stage of powder addition, we first can observe a short transitory phase

with fluctuations of the electric input power, due to the lubricant effect of the added particles, which are not incorporated inside the dough structure. The addition of powder induces a rapid increase of the electric input power (Fig. 3B-C), due to the increase of the dry matter content of the dough: the continuous dough becomes more cohesive. Similar increases in the cohesion of wheat semolina dough with decreasing water contents were observed by Rondet et al. (2013). They identified a maximum dough cohesion value near 52% water content (i.e. the plastic limit), which is the limit water content, at the structure transition from the continuous dough to the granular pasta. Below 52%, the decrease of the water content decreases the values of the cohesion of the discrete granular cohesive medium. During the reverse agglomeration process (Fig. 3BC), we also observe that final powder addition decreases the electric power values, due to the transition from the continuous dough to the discrete granular cohesive medium. During the final period of mixing (Fig. 3BC), the electric input power continues to slightly decrease and reaches a final plateau value.

Whatever the processes, increases in the water content (from 30 to 50%) of the wet powders increase the values of the electric power measured at the end of the process (Table 2). This is due to the increase in the cohesion between the wet agglomerates (Rondet et al., 2013).

The measured values of the normalized electric input power were used to calculate the electric energy consumption during each stage of the process (Table 2). For the direct agglomeration process, the values of the total energy consumption are low (11-27 kJ), because the products still stay in a powdered granular state with low cohesion forces, which were determined at 4 to 11 kPa for the water contents ranging from 30% to 50% (Rondet et al., 2013). These forces are low when comparing to the wheat dough with continuous protein network, whatever the end water content of the wet agglomerates.

For the reverse agglomeration processes, the mixing of the continuous dough during the stages of water addition and of fragmentation requires higher level of mechanical energy (Table 2). Whatever the water content, the total energy consumption for the liquid dough conditions (13-38 kJ) is slightly higher than those measured for the direct process. The level of viscosity of the continuous pseudo-liquid dough does not require high levels of electric energy. On the other hand, the total energy consumption for plastic dough conditions (21-61 kJ) are largely higher than those measured for the direct process. The cohesive behaviour of the plastic dough requires high level of electric energy during the stage of mixing, whatever the end water content of the wet agglomerates.

3.2. Characteristics of the wet agglomerates

To compare the agglomeration processes, we have measured the distribution of the size properties and of the hydro-textural characteristics of the wet agglomerates (diameter, water content, and compactness).

3.2.1. Size distribution

The agglomeration processes generate agglomerates with dispersed sizes. Whatever the processes and the water contents, the end size distribution display multimodal shapes curves (Fig. 4-5). The size distributions of the wet agglomerates were here described by considering four classes of agglomerates according to their diameter: small ($d < 0.4$ mm), medium ($0.4 < d < 0.71$ mm), large ($0.71 < d < 1.25$ mm), and very large ($1.25 \text{ mm} < d$) agglomerates (Fig. 6). Even if the distribution curves of diameter did not display unimodal normal shape, we calculated the apparent median diameter (d_{50}) and apparent dispersion parameter (span) as global criteria (Fig. 7).

The results demonstrated a significant impact of the process on the slope of the size distribution curves (Fig. 4), whatever the water content. The reverse plastic process is characterized by the agglomerates with slightly lower values of the apparent median diameters (-90 µm) and of the apparent span, compared to the direct process (Fig. 7). On the other hand, the reverse liquid process is characterized by agglomerates with slightly higher values of the apparent median diameters (+115 µm), and lower values of the apparent span, compared to the direct process. These effects can be mainly observed at 30% and 50% content, albeit with a greater difference at 50% level. We cannot give explanation into why at 40% the D_{50} for all process show similar values.

Whatever the process type, the increase of the water content induces expected increases of the apparent median diameter (Fig. 7), with lower contents of the small agglomerates and higher contents of the large agglomerates (Fig. 5-6). The content of the medium agglomerates is the highest at 40% water content. Increasing the water content (from 30 to 50%) does not affect the apparent diameter span values for the reverse processes (Fig. 7).

Large differences in the size distribution curves (Fig. 4-5) and in the apparent span values (Fig. 7) of the wet agglomerates have been found for the low water contents (30 or 40%). For the experiments conducted at 50% water content, only slight differences in the particles size distribution curves (Fig. 4C) and the apparent span values (Fig. 7) were demonstrated between the direct process and the reverse processes. There is a low impact of the process on

the properties of the agglomerates, when the water content is close to the plastic limit and to the formation of continuous dough.

3.2.2. Water content distribution

As showed by Barkouti el al. (2014) for the direct agglomeration, the results demonstrate whatever the process a significant dispersion of the measured values of the water content between the four classes of the agglomerates, defined by their size (Fig. 8): the large agglomerates are more wet than the small ones, whatever the process water content (30, 40, or 50%).

Increasing the process water content (from 30 to 50%) significantly increases of the measured water content for the agglomerates of medium size (0.4 mm < d < 1.250 mm), but does not affect the water content of the small and of the large agglomerates.

For the reverse plastic process, we observe a lower dispersion of the measured water contents between the four classes of agglomerates (Fig. 8B), compared to the direct process (Fig. 8A). For the reverse liquid process, we observe the higher dispersion of the measured water contents between the four classes of agglomerates (Fig. 8C), close to those of the direct process.

3.2.3. Compactness distribution

Whatever the process, the results demonstrate a significant dispersion of the measured values of the compactness between the four classes of the agglomerates, defined by their size (Fig. 9-10): the large agglomerates are less compact than the small ones, whatever the process water content. Compactness of the agglomerates is linked to their water content, in the same way that Barkouti et al. (2014) have showed.

The agglomerates produced by the reverse plastic process are characterized by slightly higher and less dispersed values of the compactness, compared to the direct process. The agglomerates produced by the reverse liquid process are characterized by slightly higher and more dispersed values of the compactness, compared to the direct process. This difference is certainly due to the difference between the agglomeration mechanisms. A potential future work conducted at the particle scale could provide relevant data to describe the impact of the reverse agglomeration process on the compactness values.

Increasing the process water content (from 30 to 50%) decreases the compactness values of the wet agglomerates, but does not significantly affect the dispersion of the compactness values.

437

438 3.2.4. Rheological properties

439 The cohesion and the friction coefficient were only measured for the selected wet
440 agglomerates (*i.e.* with size between 1.25 and 2 mm). The reverse processes (plastic or liquid)
441 give the agglomerates with higher values of the friction coefficient and lower values of the
442 cohesion, when compared with the direct process (Fig. 11). Whatever the process, the
443 cohesion of the bed of the wet agglomerates increases with the process water content. We did
444 not observe significant effects of the process water content on the friction coefficient. Similar
445 increase in the cohesion of wheat semolina dough with increasing water contents was
446 observed and discussed by Rondet et al. (2013).

447

448 **3.4. Properties of the dried agglomerates**

449 The dried agglomerates were obtained after the stages of steaming and drying of the selected
450 wet agglomerates (*i.e.* between 1.25 and 2 mm).

451

452 3.4.1. Microstructural description

453 The topology of the surface of the dried agglomerates was described from scanning electron
454 microscopy (Fig. 12). The process significantly affects the superficial microstructure of the
455 agglomerates. The agglomerates produced by the direct process (Fig. 12A) are characterized
456 by more or less spherical shapes, with some irregularities on their surfaces. They are not
457 dense objects, since an apparent porosity opening on the surface is observed. At the low water
458 content, the surface of the agglomerates is made of small particles that seem to be glued to
459 each other but still remained discernible. The increase of the water content during process
460 gives the dried agglomerates with smooth areas on their surface. It is difficult to identify the
461 native particles, because they are partly melted and merged, due to the specific reactivity of
462 the hydrated wheat proteins which composes the semolina particles.
463 The agglomerates produced by the reverse processes (Fig. 12B-C) are characterized with
464 heterogeneous and shapes far from sphere. When processed at low water content (30%) by the
465 reverse processes, the agglomerates are characterized by lengthened shapes. The surface of
466 the agglomerates is made by an apparent continuous medium, with some visible small
467 particles glued, giving the aspect of granular roughness. The proportion of the small particles
468 is higher on surface of the agglomerates produced by the reverse plastic process, when
469 compared to those produced by the reverse liquid process.

When processed at intermediary water content (40%) by the reverse processes, the agglomerates are characterized by original flat shapes, comparable to glitters or flakes. The shape ratio (i.e. apparent thickness / diameter) is estimated at 1/5. The surface of these agglomerates is made by the association of particles, which seems to be glued on each other.

When processed at high water content (50%) by the reverse processes, the agglomerates are characterized by heterogeneous and not spherical shapes. The surface of the agglomerates is then made by smooth areas on which some particles are merged. The proportion of the merged particles is lower on the agglomerates produced by the reverse plastic process, than on those produced by the reverse liquid process.

3.4.2. Compactness

Whatever the process water content of the wet agglomerates, the compactness of the dried agglomerates produced by the reverse plastic process (0.841) or by the reverse liquid process (0.848) are slightly higher than those produced by the direct process (0.831) (Fig. 13). We do not identify significant effect of the process water content on the values of compactness for the dried agglomerates.

3.4.3. Colour properties

The colour characteristics of dried agglomerates produced by the reverse processes are significantly different to those produced by the direct process (Fig. 13). The colour difference with wheat semolina (ΔE) is close to 36.8 for the agglomerates produced by the direct process. The reverse plastic process produced agglomerates with lower values of colour difference (ΔE = 33.4), due to specific values of the brightness (L*). For the reverse liquid process, the colour difference (ΔE = 35.9) is close to those of the direct process.

3.4.4. Swelling properties

The process influences the swelling ability of the agglomerates (Fig. 13). The swelling capacity of the dried agglomerates describes their ability to interact with water molecules, which depends on the crystalline state of the starch granules, and on the ability of the porous agglomerates to swell and entrap water inside the internal voids (Cuq et al., 2013).

The swelling capacity of the dried agglomerates produced by the reverse plastic or liquid process (3.10 or 3.08 $mL.g^{-1}$) are slightly higher than those measured for the direct process (2.95 $mL.g^{-1}$). Whatever the process, the increases in the process water content of the wet agglomerates before the stage of steaming increase their swelling capacity after the drying.

Higher water contents of the wet agglomerates enhance the mechanisms of gelatinization of the starch granules during the steaming stage, the loss of the crystalline structures, and the formation of amorphous hygroscopic structures in the dried agglomerates.

4. DISCUSSION

The major aim of this work was to investigate the performance of an original new reverse agglomeration process (based on the fragmentation of a dough and the formation of agglomerates, by adding a dry powder), in comparison with the direct wet agglomeration process: water droplets on particles, or the novel reverse-phase wet granulation process: particles in water layer (Wade et al., 2014).

During the reverse agglomeration process, the first stage of the elaboration of the continuous dough is conducted to initiate **physicochemical mechanisms** based on the reactivity of the wheat components under high water contents and mechanical shear stresses. These mechanisms generate irreversible changes of the wheat components (solubilization of proteins and fibers, formation of protein network) inside the dough (Bloksma 1990, Eliasson 1990,). Two conditions of dough water contents were investigated to modulate the extent of the physicochemical mechanisms: the reserve plastic process at 75% water content, with the dough of high consistency and low reactivity; the reverse liquid process at 110% water content, with the dough of low consistency and high reactivity.

The present study allows investigating the apparent electric cost of the physicochemical mechanisms during the process, and more particularly during the period of mixing of the continuous dough and of the fragmentation stage. Compared to the direct process, the electric cost of the reverse process is almost similar when considering the liquid dough conditions (Table 2). The low consistency of the liquid dough does not require overconsumption of the electric power in order to be mixed. The high molecular reactivity under the liquid dough conditions does not generate electric overcosts during the agglomeration process. The electric costs of the mixing of the continuous dough of low consistency are close to those of the mixing of the granular paste in the classic agglomeration process. On the other hand, the electric cost is higher when considering the plastic dough. The high consistency of the plastic dough requires electric overconsumption during the stage of mixing.

The energy cost of the reverse agglomeration process is thus not proportional to the extent of physicochemical mechanisms involving the wheat components. The reverse agglomeration process can thus be managed to promote different irreversible physicochemical reactions without negative impacts on the electric energy costs.

The present work demonstrates that the **characteristics** of the continuous dough are important factors during the reverse agglomeration process, because they directly contribute to the physical mechanisms involved in the formation of the agglomerates. They impact more particularly the relative dispersion of the individual properties of the agglomerates.

(i) The low consistency of the liquid dough is favourable to the mixing efficiency of the mixer, in order to quickly generate homogeneous dough at the end of the initial stage of the addition of water. On the other hand when considering the plastic dough with the high consistency, more process time is required during the initial stage of mixing in order to obtain a homogeneous dough.

However, the present results do not demonstrate the lower dispersion of the individual properties of the agglomerates produced by the liquid reverse process, when compared with the reverse plastic dough process (Fig. 8-9). The capability of the mixer to homogenize the dough thus does not seem to affect the dispersion of the individual properties of the agglomerates.

(ii) The final stage of the addition of power during the reverse process is conducted in order to promote the **controlled breakage mechanisms** of the continuous dough. The breakage mechanisms are generated by adding the native particles under mechanical stresses to produce the wet agglomerates. The wet agglomerates are made by the association of small pieces of dough and of the dry native particles. It should be pointed out that the spans of size distributions obtained by using the reverse process are narrower than that obtained for direct agglomeration process. The breakage mechanisms during the reverse process are thus not less efficient in order to produce grains with homogeneous characteristics, compared to the growth mechanisms during the direct process. The reverse process makes it possible to increase the agglomeration yields of the process. Wade et al. (2014, 2015) also indicated that size distribution of the granules may be simpler to be controlled in the reverse-phase granulation process. The size of the granules progressed towards a unimodal distribution indicating that improved mechanical dispersion occurred.

The present work demonstrates that the ability of the continuous dough to be fragmented in small pieces is partly dependent on the characteristics of the dough. The low consistency of the liquid dough is favourable to the dispersed mechanisms of breakage: this allows the formation of pieces of dough, with high dispersion in the sizes and properties (Fig. 8-9). On the other hand, the high consistency of the plastic dough requires more energy for the breakage mechanisms: the high cohesion of the dough favours the effects of mixing and allows the formation of homogeneous pieces of dough, with low dispersion in the size and properties, such as water content or compactness.

Strong differences in the **microstructure** of the agglomerates were demonstrated when comparing the direct and reverse processes (Fig. 12). The direct process generated classical round to ellipsoid structures. The reverse processes generate particles with no spherical shapes, but with original shape close to the flakes. Our results, show different trends when compared to Rondet (2008) who found that the reverse agglomeration experiments produced "more spherical and compact (agglomerates) than those produced by the classical wet agglomeration process". These differences could be due to differences in the range of plastic and liquid behaviour of the continuous dough. In addition, their microstructure is heterogeneous due to the fact that native particles at the surface are entrapped in an apparent pasty matrix. This makes it possible to generate composite material with different components. The end use properties can thus significantly differ from those of the agglomerates obtained thanks to the direct process. It makes it possible to consider the prospect of new product development in which the added powder is different from that used to generate the dough.

The variations of the compactness and water content of the agglomerates during the process can be discussed by using the **hydro-textural diagram** (Ruiz et al., 2005) (Fig. 14). This diagram is limited in its upper part by the saturation curve, which represents the maximum water content that a medium of a given compactness can contain. Whatever the process, the increase in agglomerates size is concomitant with an increase in their water content and a decrease of their compactness. The results are similar to those obtained thanks to a classical agglomeration process, in the conditions of wetting and mixing which corresponding to the drop control regime of nucleation (Rondet et al., 2010). Nevertheless, the agglomerates generated in this study (and whatever the process chosen) are found to be totally saturated with water (Fig. 14). In the case of the reverse process, this result is not surprising insofar the

agglomerates are generated from initially saturated dough pieces. When the direct agglomeration process is considered, the saturation of agglomerates is dependent on the conditions of wetting and mixing, that impose a mechanical dispersion regime of nucleation. Wade et al. (2014) have demonstrated that the reverse-phase process is able to generate granules with low intragranular porosity when starting from dense suspension, due to the complete filling of intraparticulate pores by the binder liquid in the initial phases of the process. The addition of further powders reduces the liquid saturation of the granules, with the desired particle size being obtained through controlled breakage. Proposed advantages of the reverse-phase process over the conventional wet granulation approach include a reduced risk of uncontrolled granule growth. Wade et al. (2015) have indicated that the conventional process showed granule growth driven by viscous forces, whereas the reverse-phase process showed granule breakage driven by capillary forces.

5. CONCLUSION

The different agglomeration processes have been found to generate agglomerates with specific functional attributes, due to the physicochemical reactivity of the wheat semolina components. Dough characteristics and fragmentation mechanisms have been found to contribute to the determinism of the end use properties of the agglomerates. The multiplicity of mechanisms and interactions identified during the reverse agglomeration of the durum wheat semolina offers the opportunity to generate diversity of the agglomerates. The relationship between the process parameters and the end use properties of the resulting agglomerates still needs to be better understood, for instance by investigating the spatial distribution of the mechanisms at a local scale, inside the structure of the agglomerates. This work offers innovative potentials to design the reverse agglomeration process for food powders and generate news products.

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Figure 1: Schematic description of the three investigated agglomeration processes, direct process (A), reverse plastic process (B), or reverse liquid process (C), as a function of the process water content.

Figure 2: Schematic description of the successive stages during the three investigated agglomeration processes: direct process (A) or reverse processes (B).

Figure 3: Typical normalized electric input power curves monitored during the agglomeration of wet agglomerates at 30% water content. With the direct agglomeration process (A), the reverse plastic process (B), and the reverse liquid process (C).

Figure 4: Impact of the agglomeration process (direct process, reverse plastic process, or reverse liquid process), and of the process water content (at 30% (A), 40% (B), or 50% (C) water content), on the measured particle size distribution curves of the wet agglomerates.

Figure 5: Impact of the process water content (at 30, 40, or 50%) and of the agglomeration process (direct (A), reverse plastic (B), or reverse liquid (C) process) on the measured particle size distribution curves of the wet agglomerates.

Figure 6: Mass proportion for the 4 classes of the wet agglomerates, as a function of the process water content (at 30, 40, or 50%) and the process type: direct process (A), reverse plastic process (B), or reverse liquid process (C).

Figure 7: Impact of the process water content on the apparent median diameter (d_{50}) (A) and apparent span diameter dispersion (B) of the wet agglomerates produced by the different processes.

Figure 8: Water content values for the 4 different classes of the wet agglomerates, as a function of the process water content (at 30, 40, or 50%) and the process type: direct process (A), reverse plastic process (B), or reverse liquid process (C).

Figure 9: Compactness values for the 4 classes of the wet agglomerates, as a function of the process water content (at 30, 40, or 50%) and the process type: direct process (A), reverse plastic process (B), or reverse liquid process (C).

Figure 10: Impact of the process water content on the compactness of the selected wet agglomerates (1.25 < diameter < 2 mm), produced by the different processes.

Figure 11: Impact of the process water content on the rheological properties (cohesion and friction coefficient) of the selected wet agglomerates (1.25 < diameter < 2 mm) produced by the different processes.

Figure 12: Microstructure by SEM of the dried agglomerates produced by the different process (direct, reverse plastic, or reverse liquid process) at the different process water content (at 30, 40, or 50%).

Figure 13: Impact of the process water content on the properties (compactness, color characteristics and the swelling properties) of the dried agglomerates, produced by the different processes.

Figure 14: Hydro-textural description (compactness vs. measured water content) of the wet agglomerates and of the dried agglomerates, produced by the different processes (direct, reverse plastic, or reverse liquid processes).

Table 1: Description of the experimental conditions (initial load of powder, mass of added water, and mass of added powder) defined for the successive stages of the agglomeration processes at the different process water content.

	Process water content (% db)	Initial load of powder (g)	Mass of added water (g)	Mass of added powder (g)	Total final Load (g)
Direct	30	1347	153	--	1500
	40	1250	250	--	1500
	50	1167	333	--	1500
Reverse (75%)	30	307	154	1039	1500
	40	500	250	750	1500
	50	667	333	500	1500
Reverse (110%)	30	191	153	1156	1500
	40	314	252	934	1500
	50	417	333	750	1500

Table 2: Mechanical energy consumption (kJ) measured during the successive stages of the agglomeration processes at the different process water content.

	Process water content (% db)	Mechanical energy consumption (kJ)			
		Total Process	Initial load mixing	Water addition stage	Fragmen-tation stage
Direct	30	11.3 *(±0.6)* [a]	0.76 *(±0.07)* [e]	10.4 *(±0.9)* [de]	-
	40	12.9 *(±0.8)* [b]	0.60 *(±0.05)* [d]	12.3 *(±1.2)* [e]	-
	50	27.0 *(±1.6)* [d]	0.57 (± 0.06) [d]	26.4 *(±2.4)* [f]	-
Reverse (75%)	30	25.6 *(±1.5)* [d]	0.64 *(±0.06)* [de]	7.2 *(±0.8)* [c]	13.1 *(±1.4)* [a]
	40	34.3 *(±2.0)* [e]	0.11 *(±0.02)* [b]	9.1 *(±1.0)* [d]	24.9 *(±2.6)* [b]
	50	60.6 *(±3.5)* [f]	0.26 *(±0.03)* [c]	10.8 *(±1.0)* [de]	49.3 *(±5.3)* [d]
Reverse (110%)	30	13.2 *(±0.8)* [b]	0.05 *(±0.01)* [a]	1.4 *(±0.2)* [a]	11.8 *(±1.2)* [a]
	40	16.7 *(±1.0)* [c]	0.31 *(±0.03)* [c]	3.3 *(±0.4)* [b]	13.1 *(±1.4)* [a]
	50	37.7 *(±2.2)* [e]	0.07 *(±0.01)* [a]	10.2 *(±1.1)* [d]	36.6 *(±4.2)* [c]

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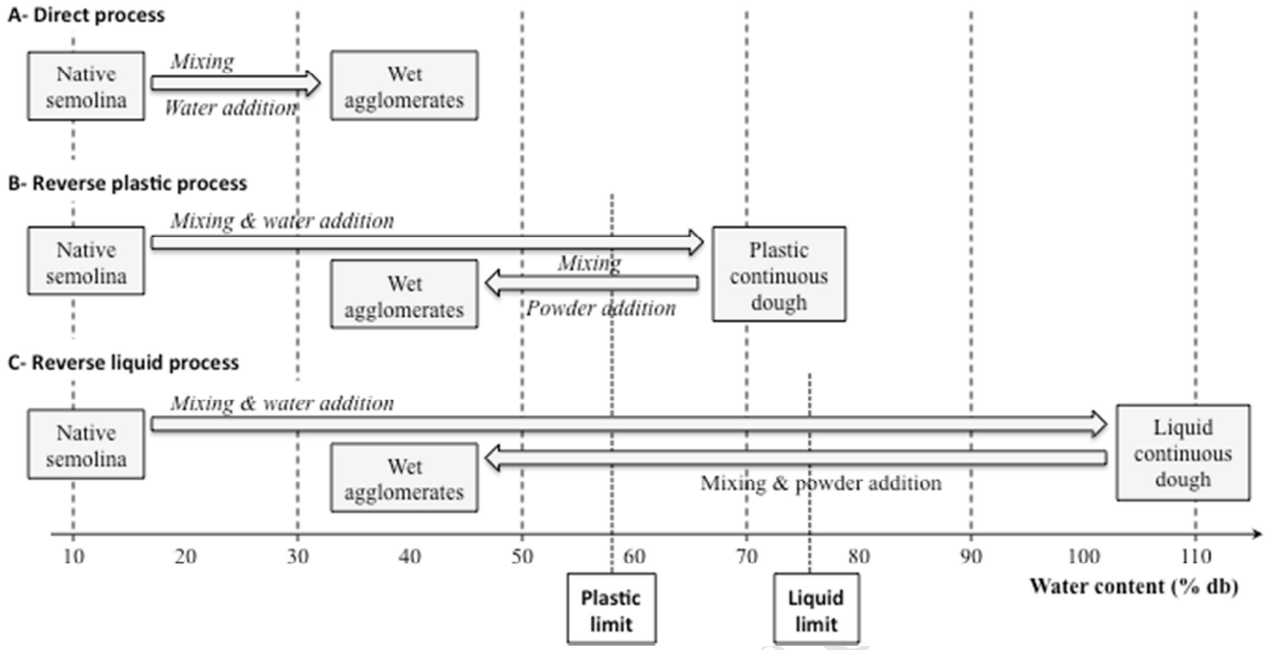

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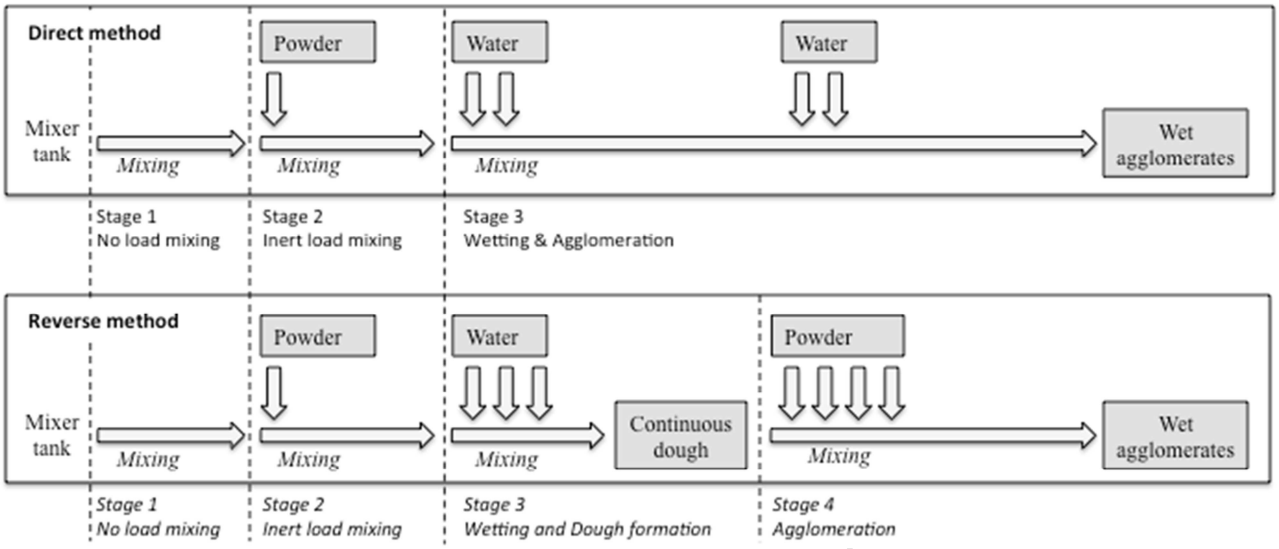
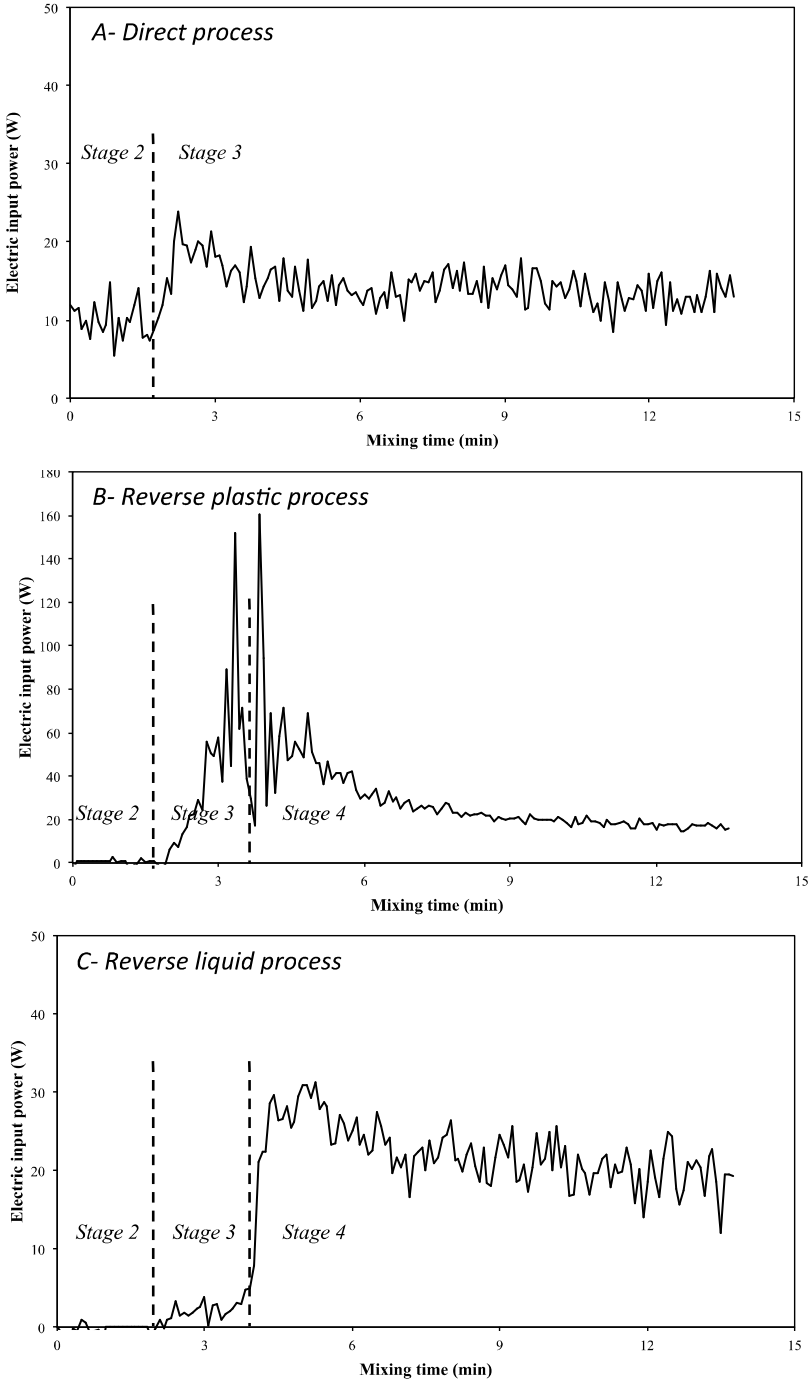


Figure 2: Schematic description of the successive stages during the three investigated agglomeration processes: direct process (A) or reverse processes (B).



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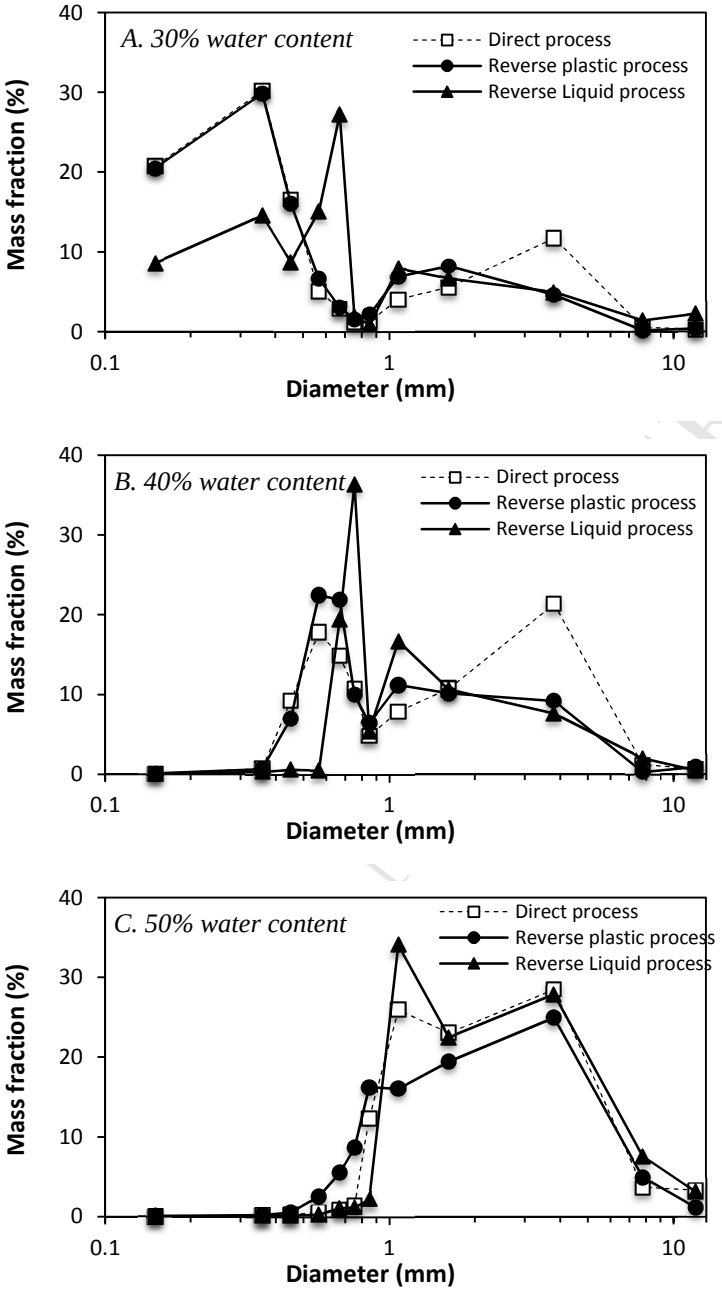

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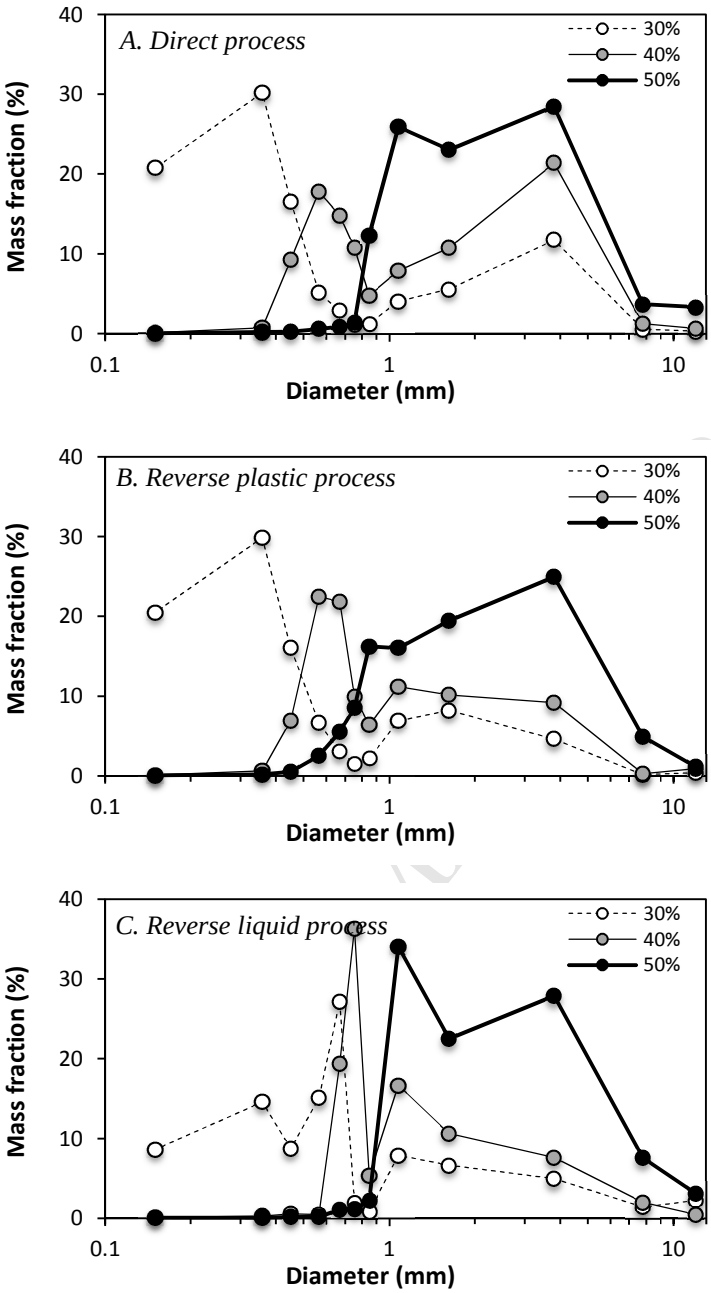

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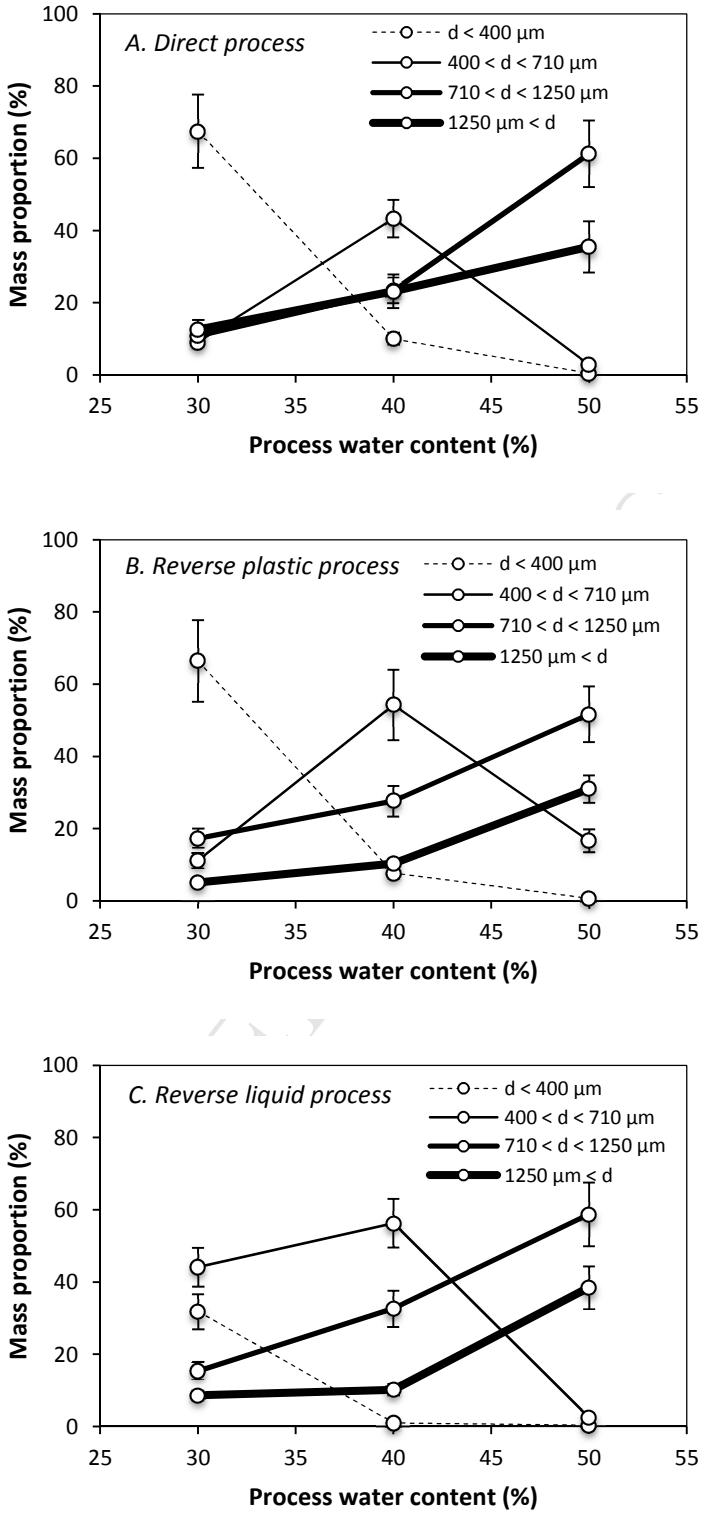

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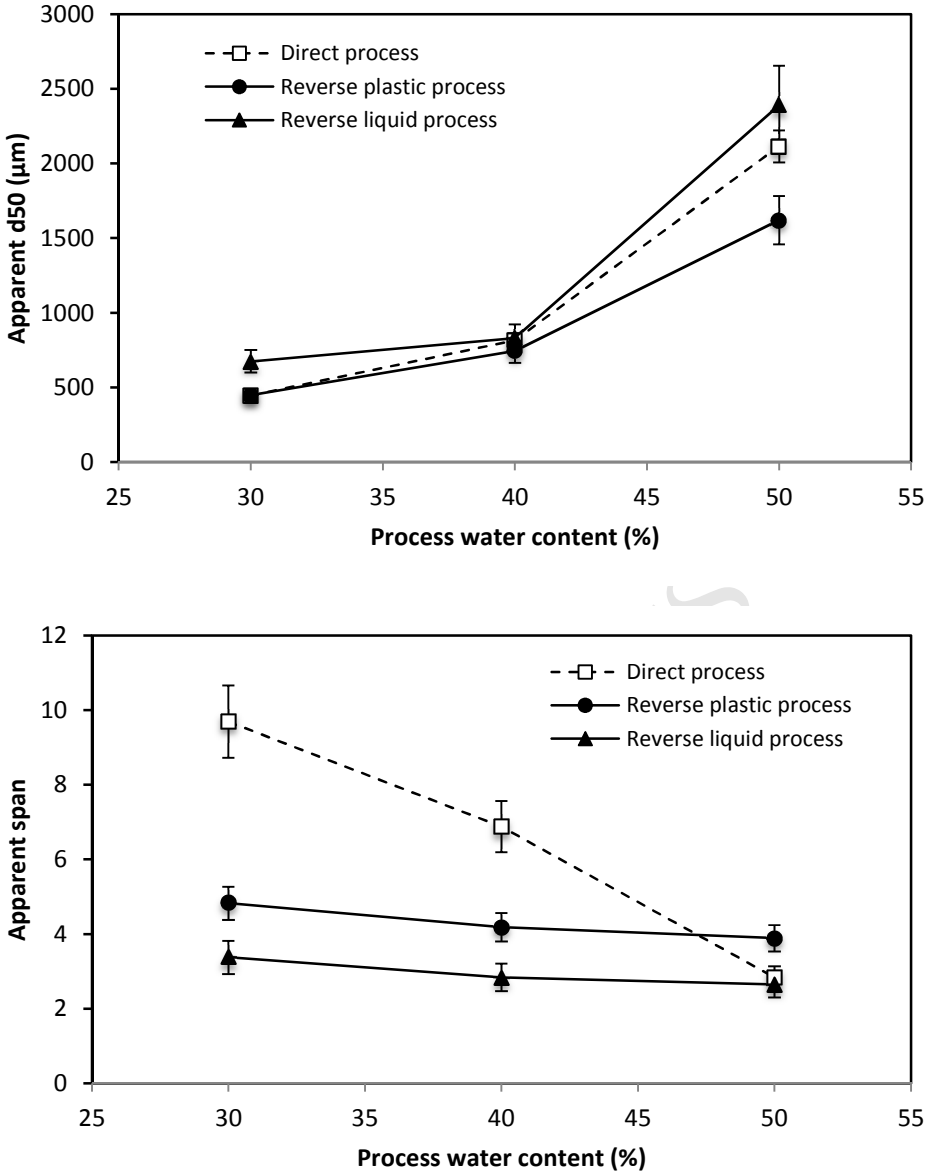

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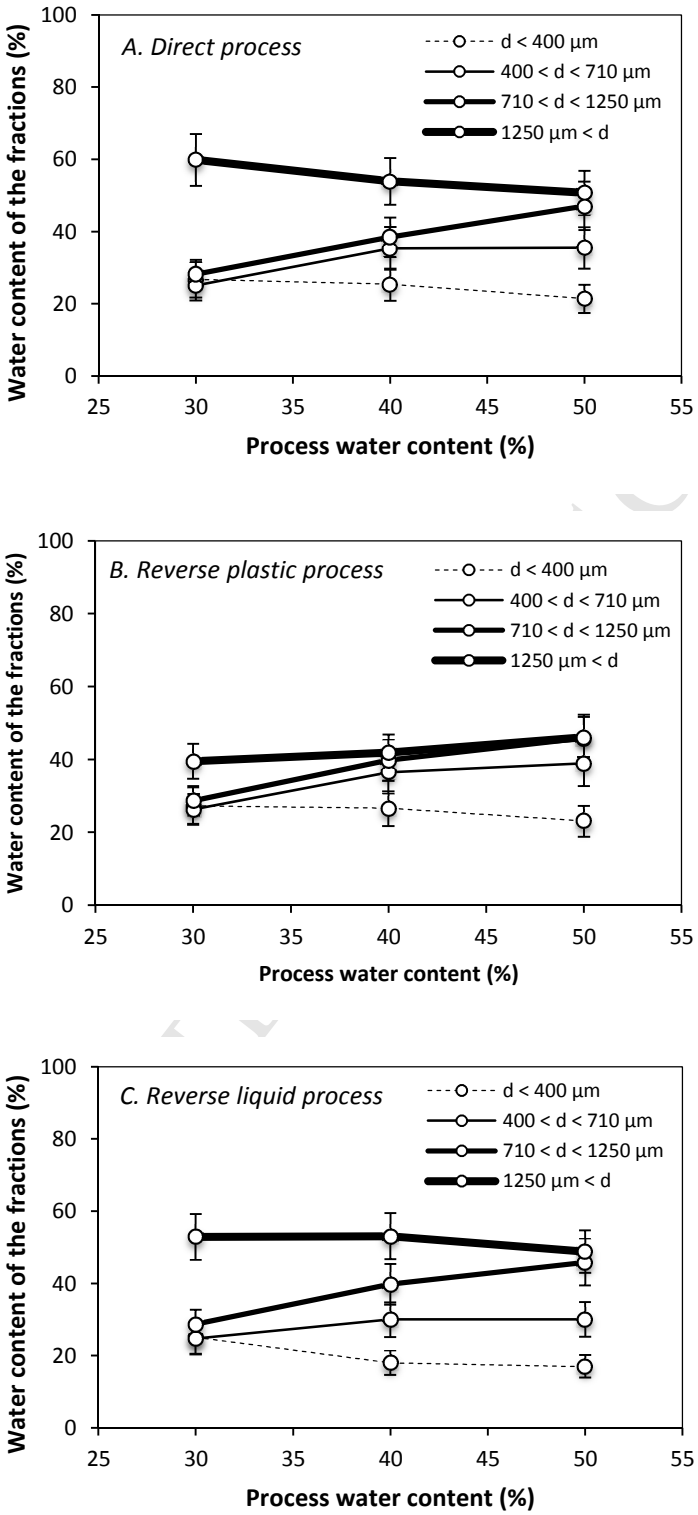

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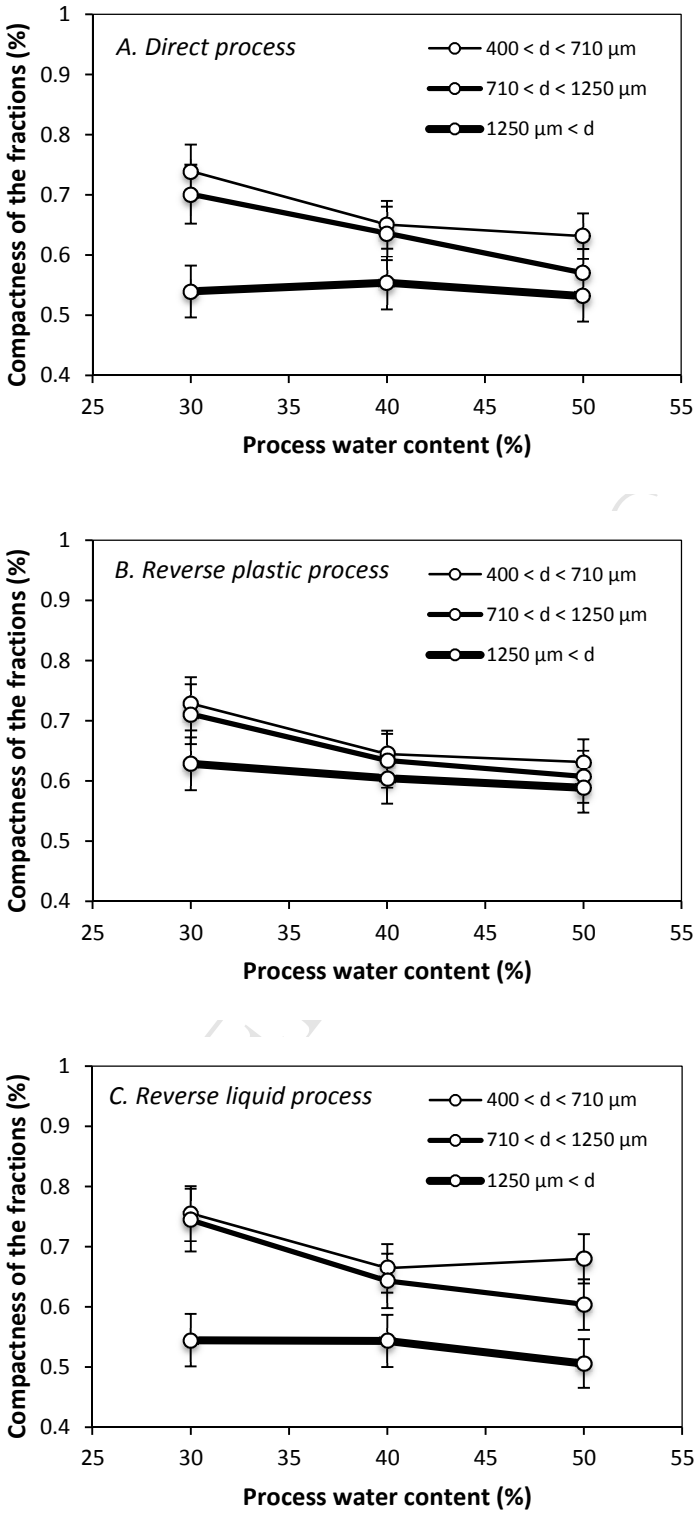

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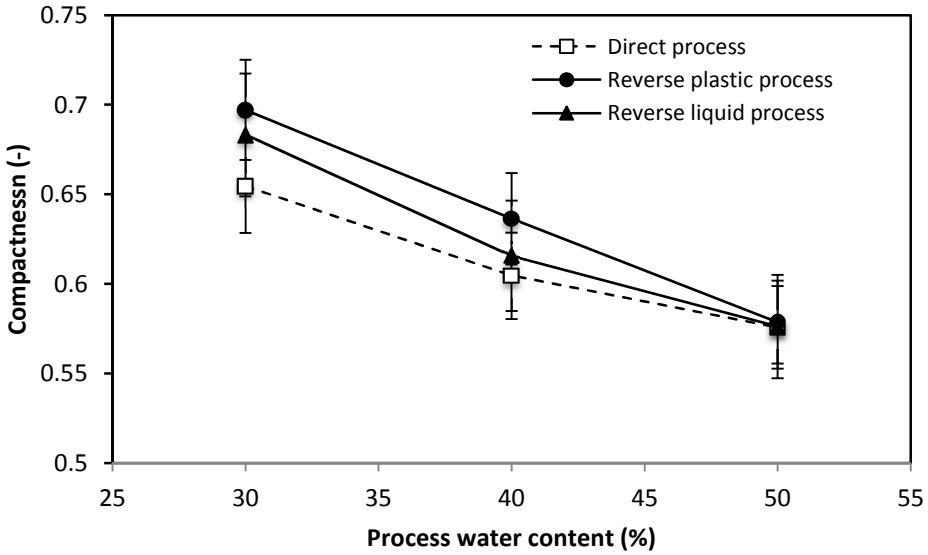
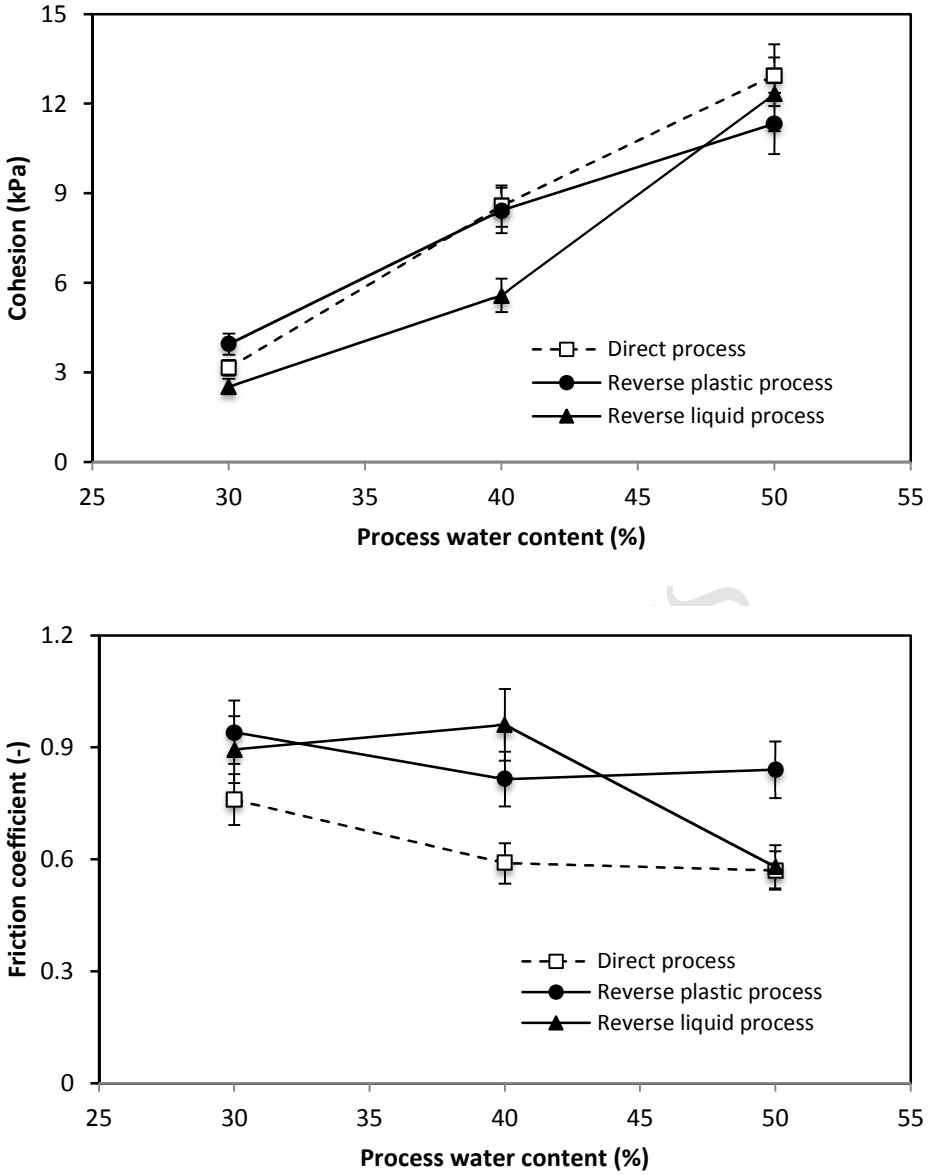


Figure 10: Impact of the process water content on the compactness of the selected wet agglomerates (1.25 < diameter < 2 mm), produced by the different processes.



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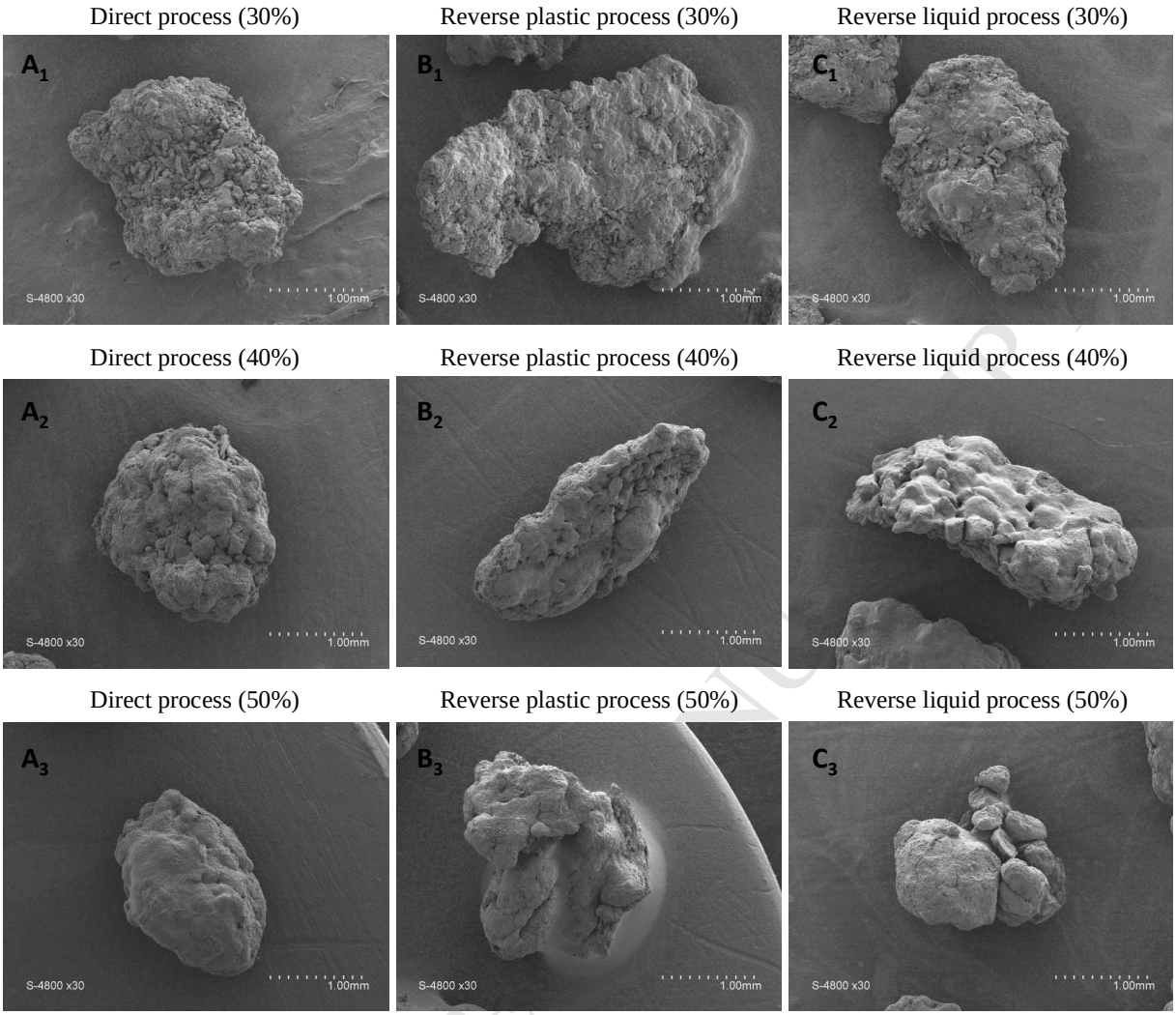

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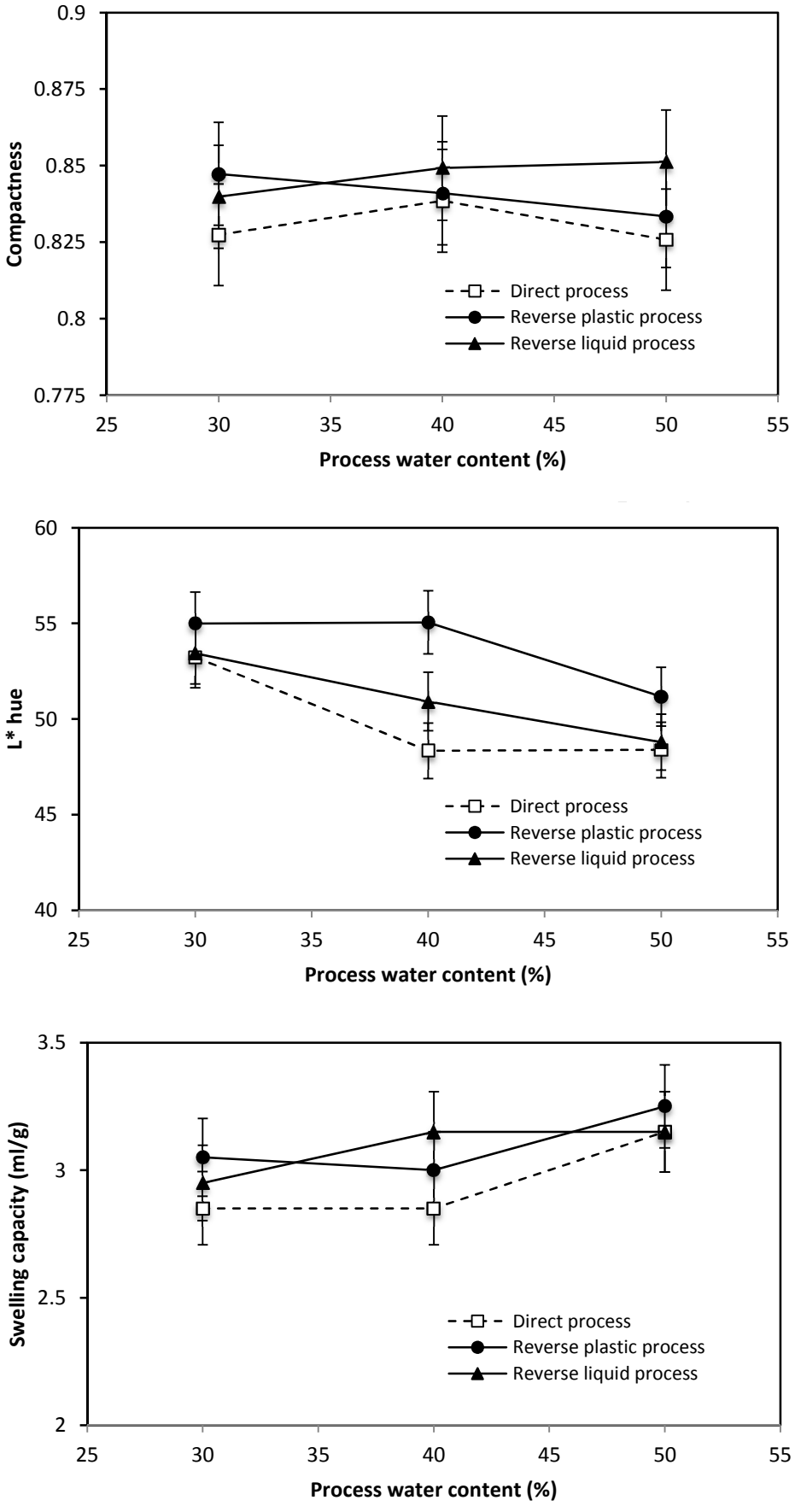

Figure 13: Impact of the process water content on the properties (compactness, color characteristics and the swelling properties) of the dried agglomerates, produced by the different processes.

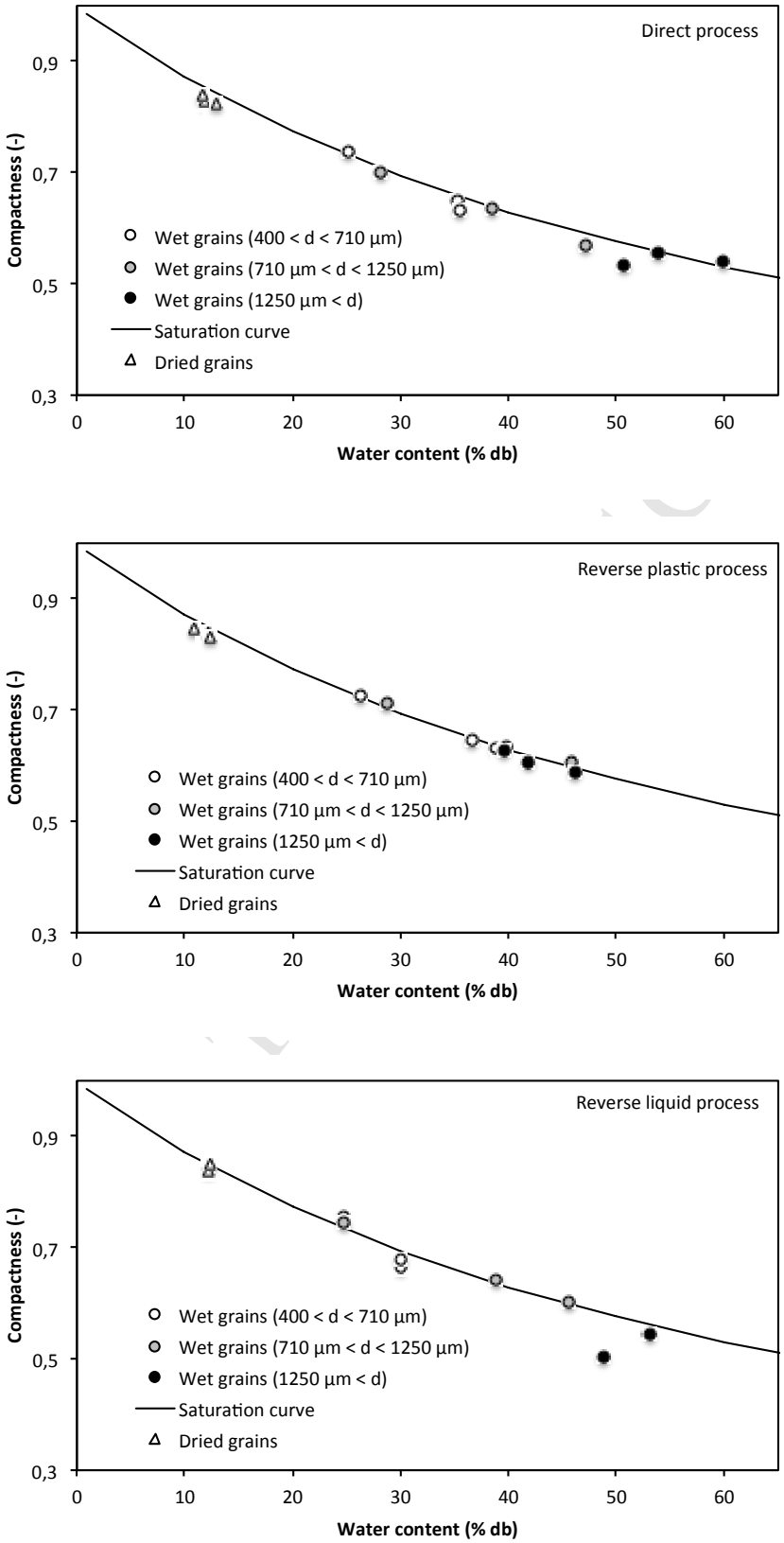

Figure 14: Hydro-textural description (compactness vs. measured water content) of the wet agglomerates and of the dried agglomerates, produced by the different processes (direct, reverse plastic, or reverse liquid processes).

Highlights

Innovative process to agglomerate wheat powder

Agglomerates were produced by the novel reverse wet agglomeration process.

The process conditions control the properties of the agglomerates.

The agglomeration process is mainly promoted by fragmentation mechanism.