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Impact of the agglomeration process on structure and functional properties of the agglomerates based on the durum wheat semolina

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ABSTRACT

The agglomeration process controls the structure characteristics and end-use properties of the agglomerates. Understanding the mechanisms involved in the structuration of the food agglomerates still represents a real scientific stake. The objective of the present study is to improve the knowledge of the process–structure–properties relationships for the agglomeration of the durum wheat semolina. Pneumatic and mechanical agglomeration technologies were selected to generate different hydro–thermo–mechanical stresses during process. The agglomerates were characterized by their structures (electron microscopy, compactness, X-ray micro-tomography, starch gelatinization) and functional properties (colors, flowability, swelling, water solubility index). The results demonstrate specific effects of shear rate, process duration, and temperature on the physicochemical mechanisms occurring during processing. A functional mechanistic model was proposed to improve the description of the agglomeration mechanisms by considering the reversible and irreversible changes. This offers innovative potential to design new agglomeration processes by considering the physicochemical reactivity of the food powders.

1. Introduction

Very present in everyday life (*e.g.* salt, pepper, spices, sugar, flour, coffee, etc.), food powders are also in the service of the industrial production of food as they are easy to preserve, transport, store, and process. The natural origin of the raw materials brings complexity in the behavior of the food powder. Agglomeration is a unit operation, which is implemented to improve the powders functionalities relating to flow properties, dust generation, mixing capacity, solubility, etc. During agglomeration, native particles are gathered to form larger assemblies, called agglomerates. Different agglomeration technologies are used according to the aimed functional properties. Knowledge about the agglomeration of food powders still remains partial, although significant scientific works have been conducted over the last 15 years (Cuq et al., 2013; Iveson et al., 2001; Litster and Ennis, 2004; Palzer, 2011). The description of the physicochemical mechanisms, which are involved during the agglomeration of the food powders, still represents a real scientific stake and will allow a better understanding of the process–structure–property relationships.

Agglomeration mechanisms result from the spatial arrangement of the native particles and binder components, and their interactions and links. A part of the agglomeration mechanisms find its origin in physical phenomena related to the establishment of capillary forces between native particles. For food powders, the physicochemical reactivity of native particles under hydro–thermo–mechanical stresses adds significant contribution of the viscous forces. When subjected to hydration and/or temperature increases, the food molecules are able to undergo glass transition and/or irreversible physicochemical changes. For instance, the partial melting mechanisms of the native particles could contribute strengthening the link between particles and consolidating the internal structure of the agglomerates (Cuq et al., 2013).

Wet controlled growth agglomeration, which refers to agglomeration processes during which a liquid binder is pulverized over an agitated powder bed, is largely considered for food applications (Iveson et al., 2001; Litster and Ennis, 2004; Palzer, 2011). Wet agglomeration process is based on the coupling of three unit operations. Liquid addition first causes adhesion forces to develop between the particles. Mixing the powder bulk disperses the liquid over the particles, promoting growth of the agglomerates by enhancing the particles motions, controlling the kinetic energy and the contact time between the particles. An end drying operation is required to stabilize the agglomerates. The agglomeration

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equipments are usually classified according to the mode of motion of the particles and to the sequentiality of the unit operations. The agglomerators based on pneumatic mixing use air stream to simultaneously agitate the particles and dry the agglomerates. The agglomerators based on mechanical mixing use impellers like blades to agitate the particles under low or high shear conditions. The end drying stage is subsequently conducted after the mechanical mixing stage.

In the field of the agglomeration of food powders, numerous works have allowed to deepen the knowledge in relation to the description of the relationships between process, structure, and the functional properties. These approaches still remain related to specific fields of applications. For example, the description of the agglomeration of dairy powders leans mainly on the use of pneumatic mixing processes, by atomization or fluid bed technologies (Barkouti et al., 2013; Palzer, 2011). On the other hand, the works concerning the agglomeration of the couscous grains based on the wheat powders have only investigated the processes of mechanical mixing technologies (Barkouti et al., 2014; Oulahna et al., 2012; Saad et al., 2011).

The contribution of the physicochemical reactivity of the food powders through irreversible or reversible mechanisms is greatly dependent on the hydro–thermo–mechanical conditions that are applied by the agglomeration process (Cuq et al., 2013). The objective of the present study is to bring understanding of the process – structure – properties relationships, by investigating the impact of different pneumatic or mechanical technologies, on the structure characteristics and functional properties of agglomerates based on durum wheat semolina. The agglomerates were characterized through their structural and functional properties. The process–structure relationships are discussed in regard to the process conditions that generate them.

2. Materials and methods

2.1. Raw material

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

2.2. Agglomeration processes

Five different processes were selected to investigate the impact of process conditions (*i.e.* shear, times, water content, and temperature), by promoting the mechanisms (wetting, mixing, cooking, and drying) as successive long-times stages (processes 1–3) or as simultaneous short-times stages (processes 4–5) (Fig. 1). The process 1 involved three successive mechanisms. The wetting– mixing stage under low mechanical shear rate using a vertical mixer, the consolidation stage by steam cooking, and the final slow drying stage. The process 2 is similar to the process 1, except the lack of the steam cooking stage. The process 3 is similar to the process 2, except the mixing stage, which was done by using a horizontal low shear mixer. The process 4 promoted simultaneous mechanisms. The wetting–mixing–drying stage is conducted using a fluidized bed under low pneumatic shear rate. The process 5 is similar to the process 4, except the use of a spray dryer as agglomeration equipment. For each process, the experimental conditions were defined from preliminary experiments. Only one set of conditions for each process was tested in the present work.

The process 1 was conducted as three successive stages. The wet mixing stage was conducted by using a vertical low shear mixer (SPI 10 Labo V03, VMI, France), equipped with a rotating 10 L bowl and a spiral blade (Fig. 2A). A sample of 1.5 kg of semolina was introduced in the bowl and first mixed for 2 min at constant mixer arm (80 rpm) and mixer bowl (9 rpm) speeds, to equilibrate the temperature at 20 °C. Water was directly sprayed over the semolina under mixing, at constant flow rate (0.31 mL s^{-1}), by using a flat spray nozzle (TPU650017, Spraying System Emani, France) connected to the water supply network. The water droplet diameters were estimated to 170 μm (Mandato et al., 2012). The calculated value of the dimensionless spray flux (0.2) corresponds to the nucleation regime, according to Hapgood et al. (2003). Water addition was conducted to reach a final water content of 35–40 g water/100 g dry matter. After the water addition step, the mixture was stirred for 2 min to homogenize. The agglomerated mass was then rolled over a column of 2 metallic sieves of decreasing mesh (2 and 0.9 mm) for 2 min at ambient temperature. The moist agglomerates were collected over the 0.9 mm sieve and spread as a thin (3 mm) layer over a stainless steel plate and steamed for 15 min at 100 °C under saturated steam flow (20 kg h^{-1}) at atmospheric pressure, inside a 20 L steam cooker (Ravant Chaudronnerie, France). The cooked agglomerates were dried as thin (5 mm) layer, over stainless steel mesh (25 × 25 cm), by using a pilot scale dryer (Afrem, Lyon, France), for 60 min at 70 °C and 80% relative humidity. The dried agglomerates were collected and stored inside hermetic plastic cups until characterization.

The process 2 was similar to the process 1, except for the steaming stage, which was not carried out. Immediately after mixing, the moist agglomerates were directly dried for 90 min at 50 °C and 50% relative humidity.

The process 3 was similar to the process 2, except the mixing stage, which was conducted using a horizontal low shear mixer (Sercom, France) (Fig. 2B). A sample of 0.8 kg of semolina was introduced in the mixing tank (30 cm length, 11.5 cm width, 16.5 cm height). The horizontal shaft axis was positioned at 6.7 cm from the bottom of the tank, with 14 metal rotating paddle blades (4 cm length, 2 cm width, 7.5 cm gap between 2 blades). Mixing was conducted at 62 rpm.

The process 4 was conducted using fluidized bed technology (Fig. 2C). A sample of 2 kg of semolina was agglomerated by using fluidized bed equipment at lab scale (ProCell 5, Glatt, Germany). The fluidization was conducted with hot air at 80 °C and air flow rate of 1.67 × 10^{-2} m^3 s^{-1}. Tap water was bottom sprayed on the moving semolina particles, using a two-fluid nozzle (970/0 – S4, Schlick, Germany) with of a spraying rate of 1.56 mL s^{-1}. The product was then collected and spread over a column of metallic sieves of decreasing mesh (2 and 1 mm). The sieving stage was conducted mechanically for 10 min at ambient temperature. The dried agglomerates were collected over the 1 mm sieve and stored inside hermetic plastic cups until characterization.

The process 5 was conducted using spray drying technology (Fig. 2D). Experiments were performed by using a modified spray dryer at pilot scale (MSD 20, GEA-PE, France) under a flow of hot air of 19.7 m s^{-1} global flow rate, with an inlet temperature of