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Effects of fluid bed agglomeration on the structure modification and reconstitution behaviour of milk protein isolate powders

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ABSTRACT

The effects of agglomeration on the density, morphology and subsequent reconstitution behaviours of milk powders were investigated. In this study, milk protein isolate, as a model system, was agglomerated in a fluid bed granulator with three different binders: water, lactose and sucrose solution (*15% w/v*). Morphology was quantified by circularity, convexity and elongation. Wettability was measured by the modified Washburn method. Powders solubilisation was quantified by dynamic particle size measurement and the kinetics of dissolved solids concentration in solution. The results showed that granules with water as the binder produced significantly lowest circularity and convexity and highest elongation. An increase in the size of the agglomerated MPI corresponded with an increase in the wettability but a decrease in the ability of solubilisation in water. Granules agglomerated with hydrophilic sugars were found to contribute differently which improved the wettability significantly but no improvement for the kinetics of dissolution.

1. Introduction

Fluidized bed agglomeration, as one of the most used granulation methods, is a process by which granulated particles are obtained by spraying a binder as solution, suspension, or melt onto a fluidized powder bed (Rajniak et al., 2007a; Turchiuli et al., 2013; Weber et al., 2007). In the food industry, powders are usually required to have good handling properties (dust-free, good flowability and easy to rehydrate). Milk protein isolate powder (MPI), as a widely used dairy ingredient, not only has fine particles and dense structures but is also difficult to rehydrate (Gaiani et al., 2007). The process of fluid bed agglomeration is applied to optimise the physical and structural properties of dairy powers, such as, particle size, densities, porosity, shape (Szulc and Lenart, 2013) and subsequently the reconstitution behaviour (Knight, 2001). Because there is strong interaction between the fluid bed process and the properties of obtained powders (Knight, 2001), controlling the process conditions is a potential way to improve the quality of MPI powders.

The use of binders in fluid bed agglomeration is one of the most frequent methods to modify the structure of agglomerates. They form solid bridges adhering primary particles and create new structures with pores and voids. Therefore, they affect the rate of particles size enlargement, densities and morphology of the granules (Keningley et al., 1997; Mills et al., 2000). However, different binders have different bonding efficiency due to varied chemical compositions, mechanical properties, concentration, viscosity and inter-particular interactions between the particles and the binders. For example, sucrose or glucose usually produces hard and brittle bridges while gelatin and acacia gum provide slow disintegration and high hardness to the agglomerates (Barbosa-Cánovas and Juliano, 2005). In food systems, aqueous solutions of lactose and sucrose are the most used binders in fluid bed agglomeration. Thus, the relationship between types of binders and agglomerates structural properties needs to be clearly identified. Although some pharmaceutical research related to agglomerate behaviour in fluidized beds has been published (Abberger et al., 2002; Chua et al., 2011; Parveen et al., 2013; Rajniak et al., 2007b; Seo et al., 2002), less attention has been given to milk powder systems, especially high protein-based powders which also present great potential for improvement to the quality of dairy powder structures.

The reconstitution ability of milk protein powders is an essential attribute as most of them are dissolved before use. It is commonly believed that the whole reconstitution process mainly consists of three sequential stages which are wetting, dispersing and dissolving phases (Forny et al., 2011; Gaiani et al., 2007; Hogekamp and Schubert, 2003; Richard et al., 2012). Each stage is affected by the

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different physical properties. Generally, wettability depends on large particles with large pores in between and also small contact angles (Hogekamp and Schubert, 2003); dispersibility is influenced by particle size, porosity and density (Goalard et al., 2006); dissolution is favoured by the presence of small hydrophilic molecules on the surface (Lillford and Fryer, 1998). Using sugar solutions as binders in agglomeration modifies the powders' structure and surface composition and influences the interaction with water. Consequently, the performances of different binders on the reconstitution ability should be investigated. Furthermore, as the reconstitution process is dynamic and powders with high protein content have relatively poor wettability and dispersibility (Gaiani et al., 2007; Richard et al., 2012), it is impossible to quantify the total process according to traditional standard methods. Moreover, different reconstitution stages often overlap (Fang et al., 2007) and it is a challenge to observe each stage independently, particularly dispersing and dissolving stages. Hence new methods were developed to better quantify the reconstitution ability of powders in this study. For example, the modified Washburn method, as a dynamic wettability measurement, was found to describe the ability of the powder particles to overcome the surface tension between themselves and the liquid (Depalo and Santomaso, 2013). Meanwhile, particle size measurement by static light scattering was used to monitor the solubilisation (both dispersing and dissolving) of powders during rehydration process (Richard et al., 2012). Final solubility was expressed as the kinetics of dissolved solids concentration (w/w%) in water.

The overall aim of this study was to acquire a better understanding of the influence of fluid bed agglomeration of milk protein isolate (MPI) powder, using three different binders and two agglomerate size fractions, on improving the reconstitution ability of MPI. Within this study, the influence of the three binders on the physical properties of the agglomerates, including powder densities, porosity and morphology, was also investigated.

2. Materials and methods

2.1. Materials

Milk protein isolate (MPI) was supplied by Kerry Ingredients (County Kerry, Ireland). The composition of MPI is 86% protein, 1.5% fat, 6% ash and <1% carbohydrate. The binding liquids used in the fluid bed agglomeration were: distilled water; aqueous solutions of lactose, 15% w/v (Arla Food Ingredients, Viby J, Aarhus, Demark) and aqueous solutions of sucrose, 15% w/v (Sigma Aldrich, USA).

2.2. Fluid bed agglomeration

A top-spray fluidised bed granulator (VFC-Lab Micro flo-coater, Vector Corporation, Lowa, USA) was used to carry out the agglomeration of MPI. 200 g MPI powder was fed into the bottom of product vessel and fluidised by upward flowing air stream. The temperature of injected air was 50 °C and the air flow rate was 200 L min^{-1}. 100 g binding liquid was injected from the top of vessel by a peristaltic pump (1 mL min^{-1}) and sprayed into small droplets by a two-fluid spray nozzle. The air pressure on the nozzle was 1 bar. When the binding solution had been used up, the product was dried for 15 min at 50 °C and the air flow was reduced to 100 L min^{-1}.

2.3. Powder characterisation

Three different samples were obtained based on three different binders in the fluid bed granulation (A_1: Water binding, A_2: Lactose solution binding, and A_3: Sucrose solution binding). The 106 μm, 180 μm and 300 μm level sieves (Endecotts, London, UK) were used to obtain two different agglomerate size fractions (P_1: 106 μm ~ 180 μm and P_2:180 μm ~ 300 μm). Including unagglomerated MPI standard powder (MSP), a total of 7 powders (MSP, A_1P_1, A_1P_2, A_2P_1, A_2P_2, A_3P_1, and A_3P_2) were investigated in this study. The obtained samples were dried overnight in the vacuum oven (Jeiotech, Seoul, Korea) at 60 °C temperature and then kept in the desiccator to cool down into room temperature.

2.4. Density and porosity

Loose and tapped bulk densities were measured using a graduated cylinder and a tapped machine (Funke Gerber, Berlin, Germany). The volume occupied by a given mass of powder (50 g) after 100 taps was measured three times to calculate the tapped bulk density. Gas Pycnometer (AccuPyc II 1340, Micromeritics Instrument Corporation, Georgia, USA) was used to measure the apparent density of samples, which were placed in the sample cell and purging with a flow of helium to degas the cell by ten pressurisation cycles. Furthermore, porosity (ε) was calculated using tapped bulk density ρ_T and apparent density ρ_A. The relation shows as Eq. (1):

$$\varepsilon = (\rho_A - \rho_T)/\rho_T \times 100 \quad (1)$$

2.5. Particle shape

The shape properties were described by Malvern Morphology G3 (Malvern Instruments Ltd, Worcestershire, UK). 15 mm^3 volume samples were placed in the dispersion unit to be dispersed into single layer on the glass plate so that they can be observed by the microscope. Generally, three parameters were used to quantify and indicate the shape properties of powders (Fig. 1). Firstly, the circularity value between 0 and 1 was defined as the ratio of the perimeter of the surface equivalent disc (P_e) to the real perimeter of the particle silhouette (P_r). The bigger value means the more alike to the equivalent circle. Secondly, the convexity describes the compactness of a particle. The maximum theoretical convexity is 1 that means the surface of particle is very smooth. Finally, elongation stands for aspect ratio of particles and a needle shape has a high value which is close to 1. These indicators were usually expressed as average value for bulk population distribution. Each sample was measured five times to obtain the average value.

2.6. Reconstitution properties

2.6.1. Wettability

Dynamic wettability measurement was based on the Washburn method (Washburn, 1921) which applied the capillary force to wet the powder (Fig. 2). In this study, 2 g samples were added into a glass tube without bottom (powder holder), and covered by filter paper and a piece of gauze at the bottom of the tube to prevent the powders falling down. Then the tube was fixed just above the distilled water (24 °C) surface. After 10 min, an analytical balance was used to measure the additional mass of wetted powder. Each sample was measured five times.

2.6.2. Solubilisation

Due to it is difficult to observe the dispersing and dissolving phases independently, solubilisation ability of the powders, including dispersibility and dissolution, was expressed as the reduction rate of particle size in dispersant based on granule erosion and break-up (Chen and Lloyd, 1994; Goalard et al., 2006; Richard et al., 2012). An optical fibre sensor (Fig. 3) (Galet et al., 2004; Larsen et al., 2003), which collected the light backscattering of

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