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# Rehydration behaviours of high protein dairy powders: The influence of agglomeration on wettability, dispersibility and solubility

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# ABSTRACT

Five common high protein dairy powders and their agglomerates produced by fluidised bed granulation were evaluated and compared for their rehydration characteristics in this study. Wettability of powders was measured by immersion wetting time, capillary rise wetting and contact angles methods, while dispersion and solubilisation processes were quantified by the change of particle size and the sediment height after centrifugation. The results showed that these high protein dairy powders generally had poor wettability, especially for whey protein isolate and the caseinates, which formed an impermeable layer separating the water surface and powders just after they contacted the water. However, the casein-micellar dominant powders exhibited prolonged dispersion due to strong interactions inside the increased wettability. However, agglomeration only caused the external structural modification and thus is difficult to accelerate the dispersion process of micellar structure inhibits the release of materials into surrounding liquid phase, which is mainly responsible for the extended rehydration time.

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# 1. Introduction

The production of milk protein is growing rapidly worldwide due to its advantageous nutritional and functional properties. As milk protein consists of casein and whey protein, different milk protein materials can be produced using different manufacturing processes (Oftedal, 2013). For example, milk protein is obtained from skimmed milk by ultrafiltration to remove lactose and minerals; subsequently micellar casein can be achieved by microfiltration to further separate whey protein; therefore, whey protein is also produced from the permeate (Chandan, 2011; Kilara, 2011; O'Mahony & Fox, 2013). In addition, some non-micellar caseinate, e.g. sodium caseinate and calcium caseinate, can be produced from acid casein by adding alkali solution (sodium hydroxide or calcium hydroxide respectively) (Farrell, Brown, & Malin, 2013; Pitkowski, Nicolai, & Durand, 2009). These milk protein materials are widely used in dairy products and infant formula, or used as emulsifiers and stabilisers in food and beverages (Chandan, 2011; Moughal, milk protein, liquid materials are usually spray-dried into the powdered forms for the ease of handling, storage and transportation (Ann Augustin & Clarke, 2011; Selomulya et al., 2013). In that case, the various milk protein powders are necessarily required to be rapidly and completely rehydrated again before use, as complete rehydration is a prerequisite for expressing the functionality of the dried ingredients. The literature has already reported that micellar casein powders were difficult to disperse in water and whey protein powders also have very poor wettability (Gaiani et al., 2006; Gaiani, Schuck, Scher, Desobry, & Banon, 2007; Schuck et al., 2007). Comprehensive assessment is still needed for the rehydration characteristics of these common milk protein powders. Consequently, it is of interest to investigate their rehydration ability and understand their rehydration mechanism.

Munro, & Singh, 2000). However, no matter what the type of

It is commonly believed that the rehydration process mainly consists of three sequential stages, which are wetting, dispersing and solubilisation. Wetting is the first step where the particles contact liquid while dispersing and solubilisation are the critical phases where primary particles start to release materials from the particle surface into the liquid (Forny, Marabi, & Palzer, 2011; Ji, Fitzpatrick, Cronin, Crean, & Miao, 2016; Richard et al., 2013).







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Once any of these three processes is limited, the time for the whole rehydration is prolonged. Casein-dominated powders are believed to be poorly-dispersible due to the strong interactions among the micellar structures. Hence, they usually take a longer time to totally dissolve in water (Baldwin & Truong, 2007; Havea, 2006; Schokker et al., 2011). However, whey protein powders demonstrate poor wetting behaviour where the material floats on the surface of the solution, which is considered to be the rate-limiting factor for whey protein rehydration (Gaiani, Scher, Schuck, Desobry, & Banon, 2009). Therefore, it is necessary to characterise the individual behaviours of milk protein powders during wetting, dispersion and solubilisation processes as different milk protein powders exhibit completely different wettability, dispersibility and solubility (Schuck, Jeantet, & Dolivet, 2012).

Agglomeration is a particle size enlargement process that creates granulates by adding a binder and forming bridges to link primary particles together. The process is used to change the structural and physical properties by increasing the size of the particles and the voids between particles, and also by decreasing the bulk density of powders (Rajniak et al., 2007). Hence, the modified structure is believed to influence the rehydration characteristics of powders. For example, the wetting phase is affected by large particles with large pores, which allow water to penetrate into particles more easily (Hogekamp & Schubert, 2003). Dispersibility is also related to the particle size and the density of powders (Goalard, Samimi, Galet, Dodds, & Ghadiri, 2006). Some studies reported that the agglomeration process played a beneficial role in the wetting behaviour of milk protein isolate powders but no significant improvement for dispersion (Gaiani et al., 2007; Ji, Cronin, Fitzpatrick, Fenelon, & Miao, 2015). Therefore, it is of interest to find out if the agglomerated powders can positively affect the rehydration behaviours for the cases of other protein powders. The rehydration process of milk protein can be described generally in the following mechanism: wetting of the powders; detachment of powders into primary particles; release of materials from particles into the aqueous phase and simultancously continuous erosion of the surface layer until the collapse of particles and their complete dissolution (Mimouni, Deeth, Whittaker, Gidley, & Bhandari, 2009). The agglomerated powders may also have an additional step which is the dissolution of the solid bridges linking the particles with the resulting granules dispersing into primary particles (Forny et al., 2011). To the best of our knowledge, few reports have investigated this mechanism for the rehydration kinetics of milk protein and compared with its agglomerated form.

The objective of this study is to investigate the effect of agglomeration on the rehydration properties of high protein dairy powders (protein content >80%). Milk protein isolates (MPI), whey protein isolates (WPI), micellar casein (MC), sodium caseinate (SC), and calcium caseinate (CC) are used as the model systems. The results will be used to exhibit the rehydration characteristics and also to better explain the rehydration mechanism of milk protein powders and their agglomerates.

### 2. Materials and methods

#### 2.1. Materials

The composition of the milk protein powders used in this study is showed in Table 1. MPI and SC were supplied by Kerry Ingredients (County Kerry, Ireland). WPI was supplied by Davisco Food International (Le Sueur, MN, USA). CC was produced by Teagasc (County Cork, Ireland). Skim milk (Kerry Ingredients, County Kerry, Ireland) was used to produce MC by a pressure driven process with 100 kDa molecular weight membranes and then the obtained retentate was vacuum evaporated to increase the solid content to approximately 38%. The concentration process was performed at 65 °C. Finally, the MC powders were obtained by a spray drying process, where the inlet and outlet temperatures were 180 °C and 85 °C, and the drying air-flow rate was 750 m<sup>3</sup> h<sup>-1</sup>. Before the measurements, all the powders were dried in a vacuum oven (Jeiotech, Seoul, Korea) at 45 °C overnight to obtain the final moisture content of about 1.5% and then kept in the desiccators.

#### 2.2. Agglomeration process

The agglomeration process of all these milk protein powders was carried out by a top-spray fluid bed granulator (VFC-Lab Micro flo-coater, Vector Corporation, Lowa, USA). 50 g of each model powder was fed into the product vessel. As different milk powders have different fluidisation behaviours in the fluidised bed, the appropriate upward flowing air streams from 30 L  $min^{-1}$  to 250 L min<sup>-1</sup> were adjusted for the fluidisation of each powder (MPI: 200 L min<sup>-1</sup>; WPI: 70 L min<sup>-1</sup>; MC: 250 L min<sup>-1</sup>; SC:  $30 \text{ Lmin}^{-1}$ ; CC:  $40 \text{ Lmin}^{-1}$ ). Meanwhile, the adjustable amount of 15% lactose solution binders, based on the different granulation behaviours of these milk protein powders, were injected by a peristaltic pump (1 mL min<sup>-1</sup>). (25 g liquid was used for MPI, MC and CC granulation process, while WPI and SC needed 20 g and 10 g binders respectively.) The air pressure on the nozzle was 1 Bar. When the lactose binders had been used up, the agglomerates were dried by air for another 15 min at 50 °C. After that, all agglomerated powders continued to be dried in the vacuum oven together with the standard powders to ensure similar moisture content.

#### 2.3. Wettability measurements

Wetting process can be described as: firstly, the interface of solid and gas is replaced by the interface of solid and water; secondly, inward diffusion of the liquid through the capillary structures of the porous powder particle (Yuan & Lee, 2013). Three methods were used to quantify the wettability of powders. Wetting time by immersional wetting procedures can be used as an initial screening and distinguish between powders with general good or poor wettability. Modified Washburn method by capillary rise wetting was used to describe the water diffusion capacity of these powders, while contact angle in spreading wetting procedure is a widely used index to evaluate the wettability by water droplet overcoming interfacial tensions between the solid and gaseous phase.

#### 2.3.1. Wetting time

This traditional method evaluates the wettability by measuring the time required to achieve complete wetting. A set quantity of powder is gently discharged onto the surface of water and allowed to immerse spontaneously without agitation. Powder wetted in less than 60 s is usually considered easy to wet while powder which takes longer than 120 s is considered non-wettable. Thus, in this study, 6 g of each sample was dropped into a 400 ml beaker containing 100 ml of distilled water at 20 °C (GEA Niro, 2005). The beakers were chosen as the same size with a diameter of 70 mm and a surface area of approx. 38.48 cm<sup>2</sup>. Wetting time was recorded by a timer and all the measurements were repeated three times. Images of WPI particles were also captured by an optical microscope (Olympus BX51M) just after the particles contacted with water on glass slides. Images taken at different magnifications were used to show the formation of external layers outside the particles surface, which restrained the water from further wetting of the particles.

# 2.3.2. Modified Washburn method

The wettability of powders can also be measured by the

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