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# The structural modification and rehydration behaviours of milk protein isolate powders: The effect of granule growth in the high shear granulation process

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Milk protein isolate is a widely developed functional ingredient

in the production of cheese, beverages, yoghurt and other food

products. As a powder, it plays an important role in industry, due to

its convenience for process, preservation and transportation (Ann

Augustin and Clarke, 2011). Milk protein isolate powders are usu-

ally produced from skimmed milk by ultrafiltration to remove

lactose and minerals and then dehydrated by spray drying (Chandan, 2011). The obtained products after spray drying are

normally fine particles with dense structures, thus they may bring

many problems, e.g. heterogeneity of the native structures (size,

shape and porosity) (Cuq et al., 2013; Knight, 2001), and difficult-

to-rehydrate (poor wettability and dispersibility) (Gaiani et al.,

2007; Selomulya et al., 2013). These issues potentially restrict the

applications of milk protein powders, as powders are required to

have good handling properties, as well as quick and complete

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## 1. Introduction

# ABSTRACT

The effects of granule growth in high shear granulation on the structures and rehydration abilities of milk protein powders were investigated. In this study, milk protein isolate, as a model powder, was agglomerated in a high shear granulator. The formed granules with different sizes were used to compare the densities, granule shapes and subsequently the wettability, dispersibility and solubility. It is found that the small nuclei showed the most compacted structures. Then the primary agglomerates coalesced to create irregular secondary structures with lower density and higher porosity until the final agglomerates formed. The densely packed structures allowed the granules to be more easily wetted by water. The large granules showed quicker release of materials into water until reaching a critical size, where more mechanical energy is potentially required for further granule break down. All the agglomerated MPI granules solubilised much more slowly than the standard MPI powder.

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rehydration behaviours to express their functionality. Granulation is a particle size enlargement process to form monodisperse granules and to optimise structural and physical properties (Cuq et al., 2013; Salman et al., 2007). Subsequently, the structures modified by the granulation process are also believed to strongly influence the rehydration properties (Ji et al., 2015; Knight, 2001).

Many different granulation processes using a variety of equipment have been widely developed in the applications of food and pharmaceutical materials (Barkouti et al., 2013; Litster and Ennis, 2013; Palzer, 2011; Rajniak et al., 2007). These processes result in different granule structures, due to the main differences in the mechanisms of particle growth and intensity of solidification (Barrera-Medrano et al., 2007). For example, fluidised bed wet granulation is used to atomize binding liquids into small droplets on the free-flowing solids without agitation (Turchiuli et al., 2013). Thus, the created granules usually show porous "raspberry" structures, which include large inter-particle void volume and internal pore volume, as the binding droplets play the role of bridges to coalesce the primary particles (Jacob, 2007; Ji et al., 2015). Another common used wet agglomeration equipment is the high shear granulator, which uses an impeller to vigorously agitate the

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powders during the addition of binding liquid, to produce the densely packed granules (Reynolds et al., 2006). These granules have not only comparatively higher density, but also a spherical shape and smooth surface, due to the effect of consolidation by agitation (Cuq et al., 2013). Although some studies have showed the advantages of agglomerated milk protein powders that were produced from a fluidised bed granulator (Ji et al., 2015), it is also necessary to find out if the high shear granulation process displays different beneficial roles on the milk protein powder due to its unique and special effects on the structural modification.

High shear granulation is a complex process and commonly consists of different groups of rate processes: 1) wetting and nucleation; 2) coalescence and consolidation; 3) attrition and breakage (Iveson et al., 2001; Mort, 2007). These competing physical phenomena occur in the granulator and control the granule size, shape and porosity, as well as many other important physical properties (Cuq et al., 2013). Wetting and nucleation is the first stage of granulation that distributes the binding liquid through the powders and then forms the nuclei aggregates, which are loosely packed (Litster and Ennis, 2013). When two or several particles collide during the liquid addition, they may stick together to create the new secondary agglomerates, and thus modify the structures. By the condition of high shear agitation, granules are consolidated through collisions with the granulator or other particles due to the force of agitation (Ennis, 2010). Plastic deformation may occur and that will squeeze out entrapped air and increase the internal pore saturation so as to reduce the granule size and porosity, which significantly influences the final structures of formed granules (Barrera-Medrano et al., 2007). Consequently, it is necessary to investigate how the different granule growth processes affect the structural formation of milk protein powders during the high shear granulation. However, as the growth processes always happen simultaneously and there is no clear definition to distinguish them, it is difficult to characterise the granules by the individual growth process (Hapgood et al., 2007). In that case, the granules with different particle sizes, which are caused by these growth rate processes, will be used to compare the structural and physical properties in this study.

While much research has been conducted into the fundamentals of granulation (Litster and Ennis, 2013; Salman et al., 2007), less attention has been given to the subsequent influence on functionality of powders. However, the agglomerated granules with modified structures are believed to potentially influence the rehydration behaviours, which include wetting, dispersing and dissolving phases (Hogekamp and Schubert, 2003; Richard et al., 2013). Each of them is closely related to the powders' physical and structural properties. Good wettability and dispersibility are both favoured by larges particles with high inter-particle porosity and high particle density (Forny et al., 2011; Goalard et al., 2006), while the dissolving behaviour is prone to the presence of small hydrophilic particles on the surface (Lillford and Fryer, 1998). Some studies showed that, agglomerated milk protein powders produced from fluidised bed display better wettability due to liquid being more easily penetrate into solids with porous structures (Ji et al., 2015). But for the high shear granulation, which has completely different granule growth mechanism from fluidised bed (Jacob, 2007), the similar studies haven't been reported so far. Consequently, it is essential to investigate the effect of its granule growth processes, on the individual wetting, dispersing and dissolving behaviours of milk protein powders.

In summary, the objective of this study is to investigate the effect of granule growth during the high shear granulation, on the rehydration abilities (wettability, dispersibility and solubility) of milk protein isolate powders. As part of this work, granules' structural modifications, including densities and morphology, were also examined.

#### 2. Materials and methods

#### 2.1. Materials

Milk protein isolate powders (MPI) were supplied by Kerry Ingredients (Kerry, Ireland). The composition is 86% milk protein, 1.5% fat, 6% ash, 5.2% moisture and less than 1% lactose. The lactose used in the high shear granulation, was purchased from Arla Food Ingredients (Viby J, Aarhus, Demark).

#### 2.2. High shear granulation

The MPI powders were agglomerated by a high shear granulator (4M8, Procept, Zelzate, Belgium). 200 g batch sizes of MPI standard powders were fed into the glass bowl and then were agitated by an impeller at a speed of 300 rpm and a chopper at a speed of 500 rpm during the granules formation stage. 100 mL 15% w/v lactose solution used as the binding solution was added into the bowl by droplets at the dosing speed of 4 mL min<sup>-1</sup>. When the binding solution had been used up, the product continued to be consolidated by the impeller and chopper at the same speed for another 10 min, which made sure the total granule size distribution was properly mono-dispersed. After that, a fluidised bed (VFC-Lab Micro flo-coater, Vector Corporation, Lowa, USA), which provided the air with a temperature of 50 °C and the flow velocity of 200 L min<sup>-1</sup>. was used to dry the granules until the moisture content was lower than 10%. Three batches of MPI granules were prepared respectively for the repeated measurements.

#### 2.3. Powder characterisation

The granules were subjected to a sieve analysis using a nest formed from 75, 106, 180, 425, 850 and 1000  $\mu$ m sieves (Endecotts, London, UK) to obtain five agglomerate size fractions (S1: -1000/+850  $\mu$ m; S2: -850/+425  $\mu$ m; S3: -425/+180  $\mu$ m; S4: -180/+106  $\mu$ m and S5: -106/+75  $\mu$ m). The particles of size larger than 1000  $\mu$ m or the smaller size than 75  $\mu$ m were removed. Therefore, including the standard MPI powder, six samples in total were investigated in this study. All the powders based on the different sizes were dried in a vacuum oven (Jeiotech, Seoul, Korea) at 60 °C temperature for 24 h and then kept in the desiccators to reach ambient temperature. The final moisture contents of samples before measurement were about 2–3%.

## 2.4. Physical properties

#### 2.4.1. Granule size, density and porosity

The particle sizes of granules were measured by laser light scattering using Malvern Masterszier 3000 (Malvern Instruments Ltd, Worcestershire, UK). At least three measurements were made and the average D (50) value was taken. The loose and tapped bulk densities were measured by a tapping machine with a graduated cylinder (Funke Gerber, Berlin, Germany). The volume occupied by 30 g powder was used to calculate the loose density while the tapped density was obtained by the volume after 100 taps. In addition, the apparent density of powder was measured by Gas Pycnometer (AccuPyc II 1340, Micromeritics Instrument Corporation, Georgia, USA). Sample was placed in the cell and purged with a flow of helium to degas the cell by ten pressurisation cycles. All the density measurements were repeated at least three times. Finally, the porosity was calculated using the tapped density and the apparent density.

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