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# Roles of particle size on physical and mechanical properties of dairy model solids

Runjing Li [a, b], Yrjö H. Roos [b], Song Miao [a, *]

[a] Teagasc Food Research Centre, Moorepark, Fermoy, Co. Cork, Ireland
[b] School of Food and Nutritional Sciences, University College Cork, Cork, Ireland

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ABSTRACT

Lactose/milk protein isolate (MPI) solids systems were prepared by laboratory-scale spray dryer (defined as S-lactose/MPI solids systems) and pilot-scale spray dryer (defined as L-lactose/MPI solids systems). Particles of L-lactose/MPI solids systems had more rounded shape, and smoother surface than those of S-lactose/MPI solids systems. Water sorption study showed S-lactose/MPI solids systems sorbed 5–30% larger amount of water than L-lactose/MPI solids systems after equilibrated at different water activities (0.11 $a_w$–0.44 $a_w$). Besides, comparing the samples with same composition, dairy solids with small size particles showed higher steady water content at the end of lactose crystallisation. Moreover, S-lactose/MPI solids systems showed lower glass transition temperature ($T_g$) values than L-lactose/MPI solids systems after equilibration at 0.11 $a_w$, 0.23 $a_w$ and 0.33 $a_w$, which might be due to the higher water content of S-lactose/MPI solids systems. The study of mechanical properties indicated the stiffness of S-lactose/MPI solids systems was higher than that of L-lactose/MPI solids systems at 0.23 $a_w$ and 0.33 $a_w$ when temperature was below $T_g$ values. Water plasticization showed stronger effect on relaxation process of dairy solids with smaller size particles.



## 1. Introduction

Several single particle characteristics are important to powder properties. These include particle size, shape, surface, density, hardness, adsorption properties, and so on (Bronlund and Paterson, 2004; Fitzpatrick et al., 2004, 2007; Fu et al., 2012). Of these features, particle size is the most essential and important. Particle size of the powder is primarily determined by physical properties of the infeed emulsion (such as viscosity and solids concentration) and the atomization operating parameters, such as the rotational speed and wheel diameter in the case of centrifugal atomization and the orifice size and pressure in the case of nozzle atomization (Finney et al., 2002; Fang et al., 2005). According to Langrish et al. (2006), the dryer type and settings can also be used to control the morphology of the powder particle and the functional properties of the powder. They stated that the powders produced by the laboratory- and pilot-scale dryers were significantly different from the commercially dried powders in both surface composition and morphology.

Although the role of particle size is not clear, it is often desirable to produce large particles to facilitate rehydration (Ji et al., 2015). Small particles tend to disperse very poorly, especially in cold water, and instead form lumps on liquid surface. Large particles can be obtained through appropriate choice of spray dryer operating conditions or the use of agglomeration techniques (Ji et al., 2015). Powders in small particle size give a large surface area per unit volume, which is related to hygroscopicity (e.g., high degree of moisture absorption). The stability of a powder, in terms of physical and chemical properties, is usually impaired by increased water sorption (Bhandari and Hartel, 2005). The study of Haque and Roos (2004) showed that fine powders (size 3–15 μm for pure lactose and 8–20 μm for lactose/proteins mixtures) sorbed more water than coarse powders (size 15–28 μm for pure lactose and 15–35 μm for lactose/proteins mixtures) at relative vapour pressure (RVP) ≤ 33.2%. Besides, powder particle size could also influence the encapsulation efficiency of oils during spray drying (Jafari et al., 2007). Jafari et al. (2007) revealed that larger particles (>63 μm) retained more volatiles than smaller particles (<38 μm), but at the same time there was more unencapsulated oil at the surface of larger particles.

In addition, the bulk density, compressibility, and flowability of

* Corresponding author.
*E-mail address:* song.miao@teagasc.ie (S. Miao).

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a food powder are highly dependent on particle size and its distribution. According to Schulze (2007), the flow properties of a fine-grained bulk solid are mainly influenced by adhesive forces due to liquid bridges (if water is present) and van de Waals forces (dominating force for dry, fine-grained bulk solids). Both forces are proportional to particle size. Moreover, for quality control or system property description, it becomes paramount to present the particle size distribution of food powders.

The objective of this study was to study the effect of particle size on physical properties and mechanical properties of lactose/milk protein isolate (MPI) solids systems. In this study two kinds of spray dryers were used to produce dairy solids with different size particles. The aim was to build a better understanding of the roles of particle size on physical properties and mechanical properties of spray-dried dairy solids.

## 2. Materials and methods

### 2.1. Materials

α-lactose monohydrate (>99% purity) and MPI were kindly offered by Arla Foods Ingredients (Sønderhøj 10–12, 8260 Viby J, Denmark) and Kerry Ingredients & Flavours (Kerry Group, Tralee, Co. Kerry, Ireland), respectively. MPI contained ≥89% protein and ≤0.35% lactose. Aluminum oxide calcined powder (≥99% purity) was purchased from Sigma–Aldrich (St. Louis, MO, USA).

### 2.2. Preparation of lactose/MPI solids systems

Aqueous solution (15%) of lactose, and lactose/MPI mixtures (4:1, 1:1, and 1:4) were spray-dried using a laboratory-scale spray dryer BÜCHI Mini Spray dryer B-191 (BÜCHI Laboratoriums-Technik, Flawil, Switzerland) (defined as S-lactose/MPI solids systems) and a pilot-scale spray dryer ANHYDRO spray dryer with centrifugal atomizer (Copenhagen, Denmark) (defined as L-lactose/MPI solids systems), respectively, at Teagasc Food Research Centre, Moorepark, Fermoy, Co. Cork, Ireland. The inlet air temperature was around 170 ± 2 °C and outlet temperature was around 90 ± 2 °C. Spray-dried solids were kept immediately in evacuated desiccators over $P_2O_5$ at room temperature. Each analysis was carried out within 3 months after spray drying.

### 2.3. Powder characterisation

Protein content was determined by FP 628 Nitrogen Determinator (LECO Corporation, Lakeview Avenue, St. Joseph, Michigan, USA). Lactose content was determined by Automatic Polarimeter (Autopol 1, Rudolph Research Analytical, Hackettstown, NJ, USA). Chemical analysis of powders was carried out immediately after manufacture. Powder particle size distribution and specific surface area (SSA) were determined by laser light scattering using a Malvern Mastersizer 3000 (Malvern Instruments Ltd, Worcestershire, UK). Powder sample was added to the standard venture disperser with a hopper gap of 2.5 mm and then fed into the dispersion system. Compressed air at 0.75 bar was used to transport and suspend the powder particles through the optical cell. A measurement time of 10 s was used, and background measurements were made using air for 20 s. The laser obscuration level was at 2–10%.

### 2.4. Morphological characteristics

Morphological characteristics were determined by Malvern Morphologi G3S (Malvern Instruments Ltd, Worcestershire, UK). 5 $mm^3$ volume powder samples were dispersed on the glass plate. 2.5× objective was used for the measurement in this study. Circularity, elongation and convexity are three commonly used shape factors (Ji et al., 2015). One way to measure shape is to quantify how close the shape is to a perfect circle. Circularity is the ratio of perimeter of a circle with the same area as the particle divided by the perimeter of the actual particle image. Several definitions of circularity could be used but for accuracy the software reports HS Circularity (HS for high sensitivity) in addition to circularity. Circularity has values in the range of 0–1. A perfect circle has a circularity of 1 while a 'spiky' or irregular object has a circularity value closer to 0. Circularity is sensitive to both overall form and surface roughness. Elongation is defined as [1 − aspect ratio] or [1 − width/length]. As the name suggests, it is a measure of elongation and again has values in the range 0–1. A shape symmetrical in all axes, such as a circle or square, has an elongation value of 0; shapes with large aspect ratios have an elongation closer to 1. Convexity is a measurement of the surface roughness of a particle. It is calculated by dividing the convex hull perimeter by the actual particle perimeter. A smooth shape has a convexity of 1 while a very 'spiky' or irregular object has a convexity closer to 0. In this study, each sample was measured in triplicate to get the average value for circularity, elongation and convexity.

### 2.5. Water sorption and lactose crystallisation

Water sorption for each solid was measured using the static gravimetric method. Approximately 1 g powder was weighed into small glass vials (25 mL). Triplicate samples of spray-dried lactose/MPI solids systems were dried in a vacuum oven (OV-12, Medline Industries, Inc., Mundelein, Illinois, USA) at 50 °C for 48 h to remove residual water. All powders were then equilibrated for 168 h in evacuated desiccators over saturated salt solutions of LiCl, $CH_3COOK$, $MgCl_2$, $K_2CO_3$, $Mg(NO_3)_2$, and $NaNO_2$, giving RVP of 11%, 23%, 33%, 44%, 54%, and 65%, respectively. All desiccators were kept in incubators with temperature of 25 °C during equilibration. The samples were weighed at 0, 3, 6, 9, 12 and 24 h, and then at 24-h intervals. All vials were kept closed with caps after the vacuum was released in the desiccators before weighing. Water content of each powder was measured as a function of time and the mean weight of triplicate samples was calculated.

### 2.6. Differential scanning calorimetry

Glass transition temperatures, $T_g$ (onset), of L- and S-lactose/MPI solids systems were determined using a differential scanning calorimeter (DSC Q2000, TA Instruments, Crawley, UK). To determine the $T_g$, spray-dried dairy solids (1 g) were transferred to glass vials and dried in the vacuum oven at 50 °C for 48 h. The dehydrated powders were equilibrated in evacuated desiccators over $P_2O_5$ and saturated salt solutions of LiCl, $CH_3COOK$, $MgCl_2$, and $K_2CO_3$ for 168 h. Then 10–15 mg of equilibrated powders was transferred to Tzero pans. The DSC pans were hermetically sealed with Tzero hermetic lids and samples were analysed. An empty pan was used as a reference. At the first scan, the samples were heated over the glass transition temperature region at 5 °C/min and then cooled at 10 °C/min to below glass transition, a 2nd heating scan was then run to far above the glass transition temperature at 5 °C/min. Anhydrous samples were scanned using pans with punctured lids to allow evaporation of residual water during the measurements (Silalai and Roos, 2010b). At the first scan, the samples were heated at 5 °C/min to 100 °C and then cooled at 10 °C/min to below glass transition, a 2nd heating scan was then run to far above the glass transition temperature at 5 °C/min. The first scan was to evaporate the residual water, while the "anhydrous" state of powders during the subsequent heating scans was expected. All