



# The effect of water plasticization and lactose content on flow properties of dairy model solids



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## ABSTRACT

The flow properties of spray-dried solids depend on their composition and physical characteristics. This study investigated the influence of water plasticization and lactose content on flow properties of lactose/milk protein isolate (MPI) solids systems. Median particle size ( $d_{50}$ ) of lactose/MPI solids systems increased as lactose content decreased, and lactose showed the largest specific surface area (SSA). Particle shape of dairy solids with higher lactose content had less rounded shape, rougher surface and lower ratio of width/length.  $T_g$  values of lactose/MPI mixtures (protein content  $\leq 60\%$ ) showed only minor difference as comparing to  $T_g$  values of pure lactose in this study. Mechanical study showed that the higher was the lactose content in dairy solids, the more significant was the change in their modulus at glass transition region. Lactose/MPI mixtures with higher lactose contents showed better flowability in this study, but they gave bigger friction angles after storage at same relative humidity.

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## 1. Introduction

Spray-dried dairy solids are very important ingredients in many food and dairy products. There is a need for information about handling and processing characteristics of spray-dried dairy solids. Flow properties of spray-dried dairy solids are very important in handling and processing operations (Knowlton et al., 1994; Fitzpatrick et al., 2007a). Flow problems in hoppers, silos and transport containers are severe problems for engineers and process operatives (Marinelli and Carson, 1992; Fitzpatrick et al., 2007a). Food powders are commonly stored in bulk silos before packaging, dry-mixing with other powders or dehydration (Crowley et al., 2014b).

The flow properties of powders depend on their composition and physical characteristics, such as particle size distribution, particle shape, surface structure, particle density, bulk density, water content and chemical composition (Crowley et al., 2014b; Janjatović et al., 2012; Kim et al., 2005; Schulze, 2007). Many food powders and food ingredient mixes are rendered complicated by the fact that they contain many different components, and this makes it difficult to predict their flow behaviour (Fitzpatrick et al., 2007a).

Crowley et al. (2014a,b) demonstrated that composition and processing of milk protein concentrates across a range of protein concentrations resulted in powders with different physical characteristics, which, in turn, affected their flow properties. Study also showed the presence of free-fat on the powder surface was critical to the deterioration of dairy powder flowability (Kim et al., 2005). Fitzpatrick et al. (2007b) stated that the dominant compositional factors affecting the cohesiveness of dairy powders were moisture, amorphous lactose and state transitions.

In addition, stickiness of powder particles is responsible for impaired flow properties (Lazar et al., 1956). Stickiness and caking of powders resulted from formation of liquid bridges between individual particles (Peleg, 1977). Many studies showed that powders with larger amounts of amorphous components were more sensitive to absorbing moisture (Meste et al., 2002; Fitzpatrick et al., 2007a,b; Liu et al., 2006; Silalai and Roos, 2010). High moisture levels affect flowability negatively, due to increased liquid bridging and capillary interactions between particles. This would result in lumping and caking problems for powders. Moreover, as amorphous solid has a kinetically frozen liquid-like structure and is not in a thermodynamic equilibrium state, amorphous materials exhibit increased molecular mobility and rapidly decreasing viscosity above the glass transition (Roos and Karel, 1991; Champion et al., 2000; Roudaut et al., 2004). Several studies have confirmed that stickiness was controlled by the glass transition (Hennings et al.,

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2001; Ozmen and Langrish, 2002; Silalai and Roos, 2011a). Besides, changes in mechanical  $\alpha$ -relaxations of milk solids/maltodextrin systems were also associated with powder stickiness, which was also as a result of increasing molecular mobility above the glass transition of powder components (Silalai and Roos, 2011b). Thus, amorphous lactose content of dairy solids might affect their flow properties, as lactose causes caking and stickiness during storage and transportation.

However, there are only a few studies about the relationship between amorphous lactose content and flow function of dairy solids. The objectives of this study were to investigate the glass transition, mechanical properties and flow properties of lactose/MPI solids systems, to study the effect of water plasticization and amorphous lactose content on the flow function of dairy model solids.

## 2. Materials and methods

### 2.1. Materials

$\alpha$ -lactose monohydrate (>99% purity) and milk protein isolate (MPI) were kindly offered by Arla Foods Ingredients (Denmark) and Kerry Ingredients & Flavours (Listowel, Ireland), respectively. MPI contained  $\geq 89\%$  protein and  $\leq 0.35\%$  lactose. Aluminium oxide calcined powder ( $\geq 99\%$  purity) was purchased from Sigma–Aldrich (St. Louis, MO, USA).

### 2.2. Lactose/MPI mixtures preparation

Dairy solids with different ratios of lactose/MPI, were spray-dried at Teagasc Food Research Centre, Moorepark, Fermoy, Co. Cork, Ireland. These were lactose, lactose/MPI (4:1, 3:2, 1:1, 2:3, and 1:4), and MPI. Solution of lactose/MPI mixtures was prepared at room temperature while the solid concentration was 15% for lactose and lactose/MPI mixtures, and 10% for MPI (w/w). All sample solutions were spray-dried by an ANHYDRO spray dryer with centrifugal atomizer (Copenhagen, Denmark) at Teagasc Food Research Centre, Moorepark, Fermoy, Co. Cork, Ireland. The inlet air temperature was around 170 °C and outlet temperature around 90 °C. Spray-dried solids were kept immediately in vacuum 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, USA). Lactose content was determined by Automatic Polarimeter (Autopol 1, Rudolph Research Analytical, USA). Water content was determined by HR83 Hologen Moisture Analyser (Mettler Toledo International Inc., Switzerland). Ash content was determined after overnight incineration at 550 °C furnace. All 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, Worcestershire, UK). Particle density was determined using a Gas Pycnometer (Accupyc II 1340 Gas Pycnometer, Micromeritics Instrument Corporation, USA).

### 2.4. Morphological characteristics

Morphological characteristics were determined by Malvern Morphologi G3 S (Malvern Instruments, Worcestershire, UK). 5 mm<sup>3</sup> volume powder samples were dispersed on the glass plate. 2.5X objective was used for the measurement in this study.

Circularity, convexity and elongation are three commonly used shape factors. 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 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. 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. Elongation is defined as [1-aspect ratio] or [1-width/length]. As the same 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. In this study, each sample was measured in triplicate to get the average value for circularity, convexity and elongation.

### 2.5. Powder preparation for flow function test

In order to study the flow function of dairy solids with different water content, two moisture levels of spray-dried dairy solids were prepared in the vacuum oven (OV-12, Meline Industries, Inc., USA). For dairy solids with low moisture (LM) content, the powders were placed in the vacuum oven at 45 °C for 36 h. For dairy solids with high moisture (HM) content, spray-dried dairy solids were firstly dried at 45 °C in the vacuum oven for 36 h, and then equilibrated over saturated  $K_2CO_3$  solution (giving 44% relative humidity) at 25 °C for 5 days in the vacuum oven. During equilibration, all powders were put in petri dishes with thickness around 10 mm. The final water content was measured in triplicate using HR83 Hologen Moisture Analyser (Mettler Toledo International Inc., Switzerland) before measuring the flow properties.

### 2.6. Differential scanning calorimetry

Glass transition temperatures,  $T_g$  (onset), of spray-dried dairy solids were determined using a differential scanning calorimeter (DSC Q2000, TA Instruments, Crawley, UK). To determine the  $T_g$ , spray-dried solids (1 g) were transferred to glass vials and dried in the vacuum oven at 45 °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 144 h. Then 10–15 mg of equilibrated powders was transferred to DSC aluminium pans (Tzero pan and lid, Switzerland). The DSC pans were hermitically sealed 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, 2010). 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 rest heating scans was expected.  $T_g$  values of spray-dried solids with different moisture contents were determined by DSC before flow function test. All measurements were carried out in duplicate.

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