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# Influence of pre-crystallisation and water plasticization on flow properties of lactose/WPI solids systems

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

This study investigated the influence of pre-crystallisation and water plasticization on flow properties of lactose/whey protein isolate (WPI) solids systems. Powder characteristics of lactose/WPI mixtures with different amounts of α-lactose monohydrate (1.01%, 11.18%, 29.20%, and 46.84%, w/w) were studied. Dairy powders with higher amounts of crystalline lactose showed larger tapped bulk density and particle density. Morphological characteristic study indicated that dairy solids with higher crystallinity had less rounded shape and rougher surface. Increasing protein content or crystalline lactose content could decrease the molecular mobility of dairy solids. Flow function tests indicated that dairy solid with 11.18% crystallinity was more easy-flowing than lactose/WPI mixtures with 1.01%, 29.20% and 46.84% crystallinity at 0% and 44% relative humidity (RH) storage conditions. Furthermore, dairy solids with higher amount of crystalline lactose showed better resistance to develop cohesive at high RH storage conditions. The friction angle of dairy solid with 1.01% crystallinity increased with increasing water content, while friction angles of lactose/WPI mixtures with higher crystallinity decreased with increasing water content.



## 1. Introduction

Flow properties of spray-dried dairy solids are very important in handling and processing operations [1,2]. Previous studies indicated that flow properties depend on the composition and physical properties of powders, such as particle size and shape, surface structure, amorphous lactose content, and water content [3–8]. Stickiness and caking of powders usually result from formation of liquid bridges between individual particles [9], and they are responsible for impaired flow properties [10]. Many studies showed that powders with greater amounts of amorphous components, such as amorphous lactose, were more sensitive to absorbing moisture, giving rise to lumping and caking problems [2,4,11–13].

Lactose in dairy systems can exist in various crystalline and non-crystalline forms. The crystalline state is a solid state having molecules well arranged in regular lattice. For lactose in amorphous state, the molecular arrangement is disordered. Amorphous lactose is thermodynamically unstable and hygroscopic, absorbing moisture from the surroundings and subsequently plasticizing, while crystalline lactose is thermodynamically stable and significantly less hygroscopic. Reducing stickiness in materials can be achieved through partial or complete crystallisation of sticky components [14]. Bronlund and Paterson [15] stated that crystalline lactose absorbed approximately 100 times less water than amorphous lactose in the same conditions. Therefore, pre-crystallising those amorphous materials during processing may help to resolve the problem of product stickiness and stability during subsequent storage [16].

Since lactose is around 70% of the dry matter in whey powder, the hygroscopicity of lactose makes whey powder become sticky and adhere to the chamber walls during spray drying [17]. Pre-crystallisation of lactose in whey concentrates before drying is a successful remedial measure in manufacturing process, and is widely used in the production of whey powder in dairy industry [18]. Powder hygroscopicity and caking are brought under control by lowering the level of amorphous lactose.

Moreover, previous studies indicated that particle shape affected the bulk behaviour and flow properties of dairy solids [5,19]. According to the study of Thomas et al. [20], morphological changes, such as surface deformation, occurred due to the build-up of lactose crystals in dairy powders. This difference in the particle shape of crystalline lactose and amorphous lactose may influence the flow properties of dairy powders and subsequently affect the handling and processing operations. Thus, comparing with amorphous lactose, crystalline lactose shows different physical properties and water sorption behaviour during processes of production and storage [15,19], which may finally influence the flow properties of dairy solids.

However, how pre-crystallisation and crystalline component content, such as α-lactose monohydrate, that affects the flow properties of dairy solids has not been reported so far. The objectives of this

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

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study were to investigate the effect of crystalline lactose content on the flow properties of lactose/whey protein isolate (WPI) solids systems. Pre-crystallisation of lactose before spray drying was used to prepare dairy solids with different amounts of crystalline lactose in this study.

## 2. Materials and methods

### 2.1. Materials

α-Lactose monohydrate (>99% purity) was kindly donated by Arla Foods Ingredients (Sønderhøj 10-12, 8260 Viby J, Denmark). WPI, containing 71% β-lactoglobulin and 12% α-lactalbumin, was obtained from Davisco Food International (Le Sueur, MN, USA). Aluminum oxide calcined powder and α-lactose (≥99% purity) were purchased from Sigma-Aldrich (St. Louis, MO, USA).

### 2.2. Powder preparation

Solution of lactose and lactose/WPI mixtures at the ratio 4:1 was prepared in de-ionised water at 65 °C in a water bath for 2 h with a stirring speed of 500 rpm. The total solid concentration of lactose and lactose/WPI mixtures solution was 40% (w/w). Then the solution of lactose/WPI mixtures was cooled to room temperature (20–22 °C) and kept at room temperature (20–22 °C) for different hours to pre-crystallise. The stirring speed was 150 rpm during pre-crystallisation. The pre-crystallisation time for lactose/WPI mixtures was 0, 3, 15 and 20 h, respectively. They were defined as S2 (0 h), S3 (3 h), S4 (15 h) and S5 (20 h) according to the pre-crystallisation time. Pure lactose without pre-crystallisation and WPI were defined as S1 and S6, respectively. They were all spray-dried by an ANHYDRO spray dryer with a centrifugal atomizer (Copenhagen, Denmark) at the Teagasc Food Research Centre, Moorepark, Fermoy, Co. Cork, Ireland. The inlet air temperature was around 170 ± 2 °C and the outlet temperature 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 three months after spray drying.

### 2.3. Powder characterisation

#### 2.3.1. Determination of α-lactose monohydrate content in spray-dried lactose/WPI mixtures

The content of α-lactose monohydrate (% C°) in spray-dried lactose/WPI mixtures was determined according to the method of Schuck and Dolivet [21]. In this study, the content of α-lactose monohydrate was used to represent the crystallinity of dairy solids. The water of crystallisation (%) of a powder is the difference between total water and non-bound water. The formula is as below:

$$\%C^\circ = (BWL*19/L)*100 \tag{1}$$

where

BWL bound water content in the lactose (g/kg);
L lactose content (g/kg).

The bound water content in lactose was calculated according to the following formula:

$$BWL = TW - FW - (0.005*WPC) \tag{2}$$

where

BWL bound water content in lactose (g/kg);
TW total water content (g/kg);
FW non-bound water content (g/kg);
WPC whey protein content (g/kg); 0.005: 0.50 g of bound water per 100 g of whey protein.

Non-bound water content of lactose/WPI mixtures was measured using GEA Niro analytical method A 1 c [22]. The total water content of lactose/WPI mixtures was determined using a Karl Fischer Titration (Mettler Toledo International Inc., Im Langacher Greifensee, Switzerland). Each analysis was carried out in triplicate.

#### 2.3.2. Powder characteristics

Water content was determined using an HR83 Halogen Moisture Analyzer (Mettler Toledo International Inc., Im Langacher Greifensee, Switzerland). 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 venturi 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. Bulk density, particle density and porosity

Loose and tapped (100 taps) bulk densities ($\rho_{tapped}$) of lactose/WPI solids systems was measured as per GEA Niro [23], using a Jolting volumeter (Funke Gerber, Berlin, Germany). Particle density ($\rho_p$) was measured as per GEA Niro [24], using a Gas Pycnometer (Accupyc II 1340 Gas Pycnometer, Micromeritics Instrument Corporation, USA). Since the definition of porosity of a porous media corresponds to extra particle void space, the corresponding porosity of dairy solids was calculated as Eq. (3):

$$\varepsilon = 1 - \rho_{tapped}/\rho_p. \tag{3}$$

### 2.5. Morphological characteristics

Morphological characteristics were determined using a Malvern Morphologi G3 S (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, 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. 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.

### 2.6. Powder preparation for flow function test

Two moisture levels of lactose/WPI solids systems were prepared in a vacuum oven (OV-12, Medline Industries, Inc., Mundelein, Illinois,