Journal of Food Engineering 209 (2017) 61–67

Contents lists available at ScienceDirect

Journal of Food Engineering

journal homepage: www.elsevier.com/locate/jfoodeng

How does particle size influence caking in lactose powder?

M. Carpin [a, b, c], H. Bertelsen [a], A. Dalberg [a], J.K. Bech [a], J. Risbo [b], P. Schuck [c, *], R. Jeantet [c]

[a] *Arla Foods Ingredients Group P/S, 6920 Videbæk, Denmark*
[b] *Department of Food Science, University of Copenhagen, 1958 Frederiksberg C, Denmark*
[c] *STLO, UMR 1253, INRA, Agrocampus Ouest, 35000 Rennes, France*

ARTICLE INFO

Article history:
Received 29 October 2016
Received in revised form
29 March 2017
Accepted 5 April 2017
Available online 9 April 2017

Keywords:
Crystalline lactose
Caking
Particle size distribution
Moisture sorption
Ring shear tester

ABSTRACT

Particle size distribution (PSD) is known to influence product properties such as flowability and compressibility. When producing crystalline lactose, different steps can affect the PSD of the final powder. The aim of this study was to investigate the influence of PSD on caking and the mechanisms involved. Smaller particles showed higher moisture sorption and a greater caking tendency, measured by dynamic vapor sorption and ring shear testing, respectively. Therefore, moisture sorption isotherms appeared as a valuable tool to predict the effect of PSD on humidity caking, as confirmed by the results of ring shear testing. Controlling the amount of fines, characterized by a higher content of impurities, a larger specific surface area and a broader span of the PSD, was found critical to limit caking. More precisely, both the total surface area and the span of the PSD require close attention as they can significantly influence humidity and mechanical caking.

1. Introduction

Among the whey-derived ingredients, lactose powder production is currently increasing significantly, with a production of nearly 1.26 million tonnes in 2013 and 1.5 million tonnes expected for 2017 (Affertsholt and Fenger, 2013). The numerous applications include e.g. dairy products, confectionery, baked goods and drugs (Lifran et al., 2000). Lactose powder has been produced since the beginning of the 20th century with the yield in focus (Paterson, 2009). The various applications in the food and pharmaceutical industries have led to different levels of quality requirements (Holsinger, 1988). However, these requirements have become stronger over the past decade with the increasing demand for infant formulae. Lactose is indeed the main ingredient in infant formulae as it provides an important source of carbohydrates to match the composition of human milk. Nowadays, quality is an essential differentiation factor in the highly competitive dairy market. One of the product parameters which lactose producers still strive to control to ensure constant quality is the particle size distribution (PSD) of the powder.

Particle size distribution is a very important characteristic as it governs several product properties (Ortega-Rivas, 2008) such as density, compressibility (Barbosa Canovas et al., 1987), flowability (Juliano and Barbosa-Cánovas, 2010), segregation (Barbosa-Canovas et al., 1985), rehydration (Gaiani et al., 2007), etc. However, obtaining the right PSD is often a challenge. The crystallization step is crucial for this purpose but by no means straightforward given the complex physicochemical processes involved (Schuck, 2011). Downstream, the PSD can be modified unintentionally (e.g. by particle attrition) or intentionally (e.g. by milling).

Caking, i.e. the unwanted agglomeration of powder particles observed as lumps of various sizes and hardness, is another product property which can be influenced by the PSD of the powder (Rogé and Mathlouthi, 2000). Caking has been an issue in numerous powder handling factories for many years and still leads to significant economic loss. The main caking mechanisms in food powders have recently been reviewed by Carpin et al. (2016). Briefly, the first mechanism, i.e. amorphous caking, involves the creation of viscous flow of amorphous material between particles as a result of an increase in temperature beyond the glass transition temperature (T_g). Moisture can plasticize the material and decrease T_g. Humidity caking is the other mechanism in which moisture plays a critical role. When the relative humidity (RH) of the air surrounding a crystalline material increases, water molecules are indeed adsorbed onto the particle surface and can form liquid bridges between particles. If the RH exceeds the deliquescence relative humidity

---

* Corresponding author.

E-mail addresses: melanie.carpin@arlafoods.com (M. Carpin), hans.bertelsen@arlafoods.com (H. Bertelsen), anders.dalberg@arlafoods.com (A. Dalberg), jens.kristian.bech@arlafoods.com (J.K. Bech), jri@food.ku.dk (J. Risbo), pierre.schuck@inra.fr (P. Schuck), romain.jeantet@agrocampus-ouest.fr (R. Jeantet).

http://dx.doi.org/10.1016/j.jfoodeng.2017.04.006
0260-8774/© 2017 Elsevier Ltd. All rights reserved.

(DRH) of the material, a phase transition from solid to liquid state takes place, leading to a dramatic increase in the amount of moisture adsorbed. The third mechanism, i.e. mechanical caking, is rather an aggravating factor and can dramatically intensify the two above mentioned mechanisms. Inter-particle interactions are indeed increased when particles come closer to each other, for example as a result of compression.

If the PSD is modified, for example due to changes in the crystallization process, how will the caking tendency of the final powder be impacted? This question frequently arises in the powder handling industries and the answer is not straightforward. Mathlouthi and Rogé (2003) investigated the effect of particle size on moisture sorption and caking of sucrose. The higher the amount of fine particles (<250 μm) added to a standard sugar sample, the more sorption was recorded. The particle cohesiveness was found to increase with a decreasing particle size. Fine particles had moderate flow as measured with a Jenike shear cell. This result was explained by their small size and increased hygroscopicity. In addition, Provent et al. (1993) reported that the main cause for caking of a particular pharmaceutical powder was the particle size. By mixing coarse and fine particles in different ratios, they showed that caking was affected by both the total surface area of the powder and the distribution of particle sizes. However, these studies were performed on powders other than lactose, and one could question if the results are still valid for this latter compound. For example, impurities are known to influence the caking behavior of a given powder (Carpin et al., 2017). It is therefore worth investigating the caking mechanisms specific to the lactose powder.

Listiohadi et al. (2005) investigated the effect of milling on the caking behavior of lactose. The hardness of the powder cakes was found to increase for a smaller particle size but moisture sorption was not affected by the particle size. More recently, Modugno et al. (2015) published a study on the influence of the PSD on lactose caking. They sieved the lactose powder to obtain different size classes which were subjected to caking tests. They reported that lactose crystals with a *d 50* smaller than 300 μm could cake easily if the water content was higher than 3%: they explained their result by a greater number of liquid bridges in the small size classes compared to the large size classes. However, this result cannot be applied to dried lactose powders for which the water content is usually below 0.5% (Hourigan et al., 2013). Moreover and as in most studies, the span of the size classes was not taken into account, which can greatly affect the packing density of the powder and thereby the number of liquid bridges. Therefore, even though articles have been published on the subject, it remains difficult to assess how the PSD influences caking of a given powder. The aim of the present study was therefore to investigate the influence of the PSD on caking of crystalline lactose and the mechanisms involved. The expected results should provide a clearer picture of what needs to be measured and controlled in order to limit caking.

2. Materials and methods

2.1. Production of lactose powders on a pilot scale

Decalcified and decolored ultrafiltered (UF) whey permeate was obtained from Arla Foods Ingredients (Viby J, Denmark). The solids content was raised to 60% in a Centritherm CT2 evaporator (Flavourtech, Griffith, Australia). For lactose crystallization control, the concentrate was cooled in a tank from 79 °C to 11 °C in about 18 h. Then, a Lemitec MD80 laboratory decanter centrifuge (Lemitec GMBH, Berlin, Germany) was used to produce five lactose powders with different washing grades. The slurry was first run through the decanter once without water for a pre-wash (Wash 0). This prewashed slurry was then mixed with water at different water/lactose slurry w/w ratios: 1/3 (Wash 0.3), 1/2 (Wash 0.5), 1/1 (Wash 1) and 2/1 (Wash 2). The different washing grades of the prewashed slurry were run through the decanter once more, and then dried in an Anhydro SFD 47 spin flash dryer (SPX Flow Technology, Søborg, Denmark) with an inlet temperature of 105 ± 2 °C and an outlet temperature of 85 ± 3 °C. Finally, the powders were packaged in two layers of plastic bags and a Kraft paper bag before transportation to the analysis laboratory where they were sieved and poured into airtight plastic containers of various sizes to minimize the headspace. The powders were stored at 20 °C prior to analysis.

2.2. Sieving

The lactose powders of different washing grades were sieved through a series of standard testing sieves (80, 160, 250, 355 and 500 μm) stacked in a sieve shaker (Retsch sieve shaker AS 200, Retsch GmbH, Haan, Germany) to separate them in different size fractions. The sieving was performed in batches of 500 g of powder for 5 min at a 1-mm amplitude.

For the investigation of the effect of the span on caking (Section 3.5.), two different size fractions of Wash 0.3 powder were produced by sieving; one by sieving Wash 0.3 through 100 and 355 μm sieves and another one by sieving Wash 0.3 through 160 and 250 μm sieves.

2.3. Measurement of particle size

The PSD of the different size fractions was measured by laser light scattering using a Malvern Mastersizer 2000 equipped with a Scirocco 2000 dry dispersion unit (Malvern Instruments, Worcestershire, UK). The span of the distribution was calculated as

$$\text{Span} = [(d\ 90 - d\ 10)/d\ 50], \tag{1}$$

where the diameters *d 10*, *d 50*, and *d 90* represent the particle size at 10%, 50%, and 90% in a cumulative size distribution respectively. The specific surface area was calculated from the surface weighted mean d_{32}.

2.4. Chemical composition

Protein and ash content were determined according to the methods described by Schuck et al. (2012). Total nitrogen content determined by Kjeldahl with a 6.38 conversion factor will be designated as protein content. Given the filtration steps in the lactose process, it is, however, unlikely that proteins remain in the final powder. Therefore, impurities formally expressed as protein may more likely be smaller nitrogen containing components such as peptides and amino-acids. Analysis of ash content was carried out in triplicate and the protein content was determined in duplicate.

2.5. Particle morphology — Scanning Electron Microscopy (SEM)

The surface morphology of the lactose samples was examined using a Scanning Electron Microscope (SEM, JEOL JCM-6000 — NeoScope II, Tokyo, Japan) operating at 15 kV. Samples were mounted on an aluminum stub and coated with a thin layer of gold (JEOL JFC-1300 auto fine coater) prior to analysis. The photomicrographs were taken at ×400 magnification.

2.6. Moisture sorption measurements

Sorption isotherms of powders were obtained with a Dynamic Vapor Sorption (DVS) Advantage (Surface Measurement Systems