



Impurities enhance caking in lactose powder



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ARTICLE INFO

Article history:

Received 30 August 2016

Received in revised form

13 October 2016

Accepted 18 November 2016

Available online 18 November 2016

Keywords:

Caking

Lactose

Impurities

Amorphous

Moisture sorption

Ring shear tester

ABSTRACT

Caking of lactose and other dry ingredients is a common problem in the dairy and food industries. The lactose production process includes different purification steps, depending on the type of lactose produced. The aim of this study was therefore to investigate how the remaining impurities (i.e. non-lactose components) affect the caking tendency of the final powder. The results from a combination of different methods, including dynamic vapor sorption, characterization of the physicochemical composition and assessment of caking with a ring shear tester, suggested humidity caking. Larger amounts of impurities in the lactose powder resulted in enhanced moisture sorption and greater caking tendency. These findings emphasize the importance of controlling the washing and purification steps throughout the production process in order to limit caking in the final product.

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1. Introduction

As a consequence of trade globalization and significant advances in drying and powder handling technology, the volume of food ingredients produced in powder form has dramatically increased in the past decade. Indeed, as dry ingredients have better storage stability and are easier to transport, a major part of the recent investments in the dairy sector has been focused on dry products (International Dairy Federation, 2015). In particular, whey, which was traditionally considered as waste, has gained considerable attention, and its different constituents (whey proteins, lactose, lactoferrin, milk salts, etc.) are now separated and sold as high value products in the dry state. The range of applications for whey-derived dry ingredients has thus expanded considerably.

Among the whey-derived ingredients, lactose is used in various food and pharmaceutical applications. For example, lactose powder is the main ingredient of infant formulae and provides an important source of carbohydrates to match the composition of human

milk. Lactose can be found in different forms, but the most common and stable form is crystallized α -lactose monohydrate. α -lactose monohydrate is produced industrially by evaporation of whey followed by slow cooling in a crystallization tank. Typically, the harvested crystals are then washed and dried in a fluidised bed dryer. Different purification steps can make the process more complex, depending on the type of lactose produced. For example, calcium and phosphate are usually removed prior to evaporation in order to increase the running time of the evaporators and reduce fouling. The lactose production process has been described in greater detail by Hourigan et al. (2013).

The handling and storage of lactose and other dry food products can be complicated by a problem that is well known in the food industry, i.e. the unwanted agglomeration of powder particles observed as lumps of various sizes and hardness. This process, known as caking, results in non-conform products and significant economic loss. Although α -lactose monohydrate is generally considered to be a stable product, caking of lactose is a major problem in the dairy industry. The three most relevant caking mechanisms in food powders have recently been reviewed (Carpin et al., 2016). Amorphous caking is the main mechanism in amorphous powders whereby a temperature increase above the glass transition temperature (T_g) of the material leads to viscous flow to contact points. Due to the plasticization effect of water, storage at

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relatively high relative humidity (RH) can lower the T_g and initiate amorphous caking. Humidity is also a crucial factor in the second caking mechanism, called humidity caking. Water molecules are adsorbed on the surface of the particles and liquid bridges can be formed by capillary condensation. If the RH increases above the deliquescence relative humidity (DRH) of the material, the solid can dissolve in the surrounding water layer. A subsequent decrease in RH results in solid bridges and thus stronger links between particles. Finally, the third mechanism, mechanical caking, is an aggravating factor rather than a caking mechanism in itself. Mechanical pressure on a powder bed brings the particles closer to each other, thereby increasing the interactions between particles and the number of contact points. Mechanical caking therefore worsens any caking tendency due to humidity or the presence of amorphous material.

In view of the above three mechanisms, it is obvious that caking can be influenced by several parameters such as water content, particle size and shape, amorphous content, etc. Several studies have investigated how these factors affect caking of α -lactose monohydrate. Listiohadi et al. (2008, 2005a, 2005b) focused on the role of the different lactose polymorphs, amorphous lactose and the milling procedure. Bronlund and Paterson (2004) examined the effects of particle size and temperature on the moisture sorption characteristics of lactose powder and temperature-induced moisture migration in a bag of lactose (Paterson and Bronlund, 2009). As humidity has a crucial role in both amorphous and humidity caking, any impurity that can enhance lactose hygroscopicity, such as peptides and minerals, can be detrimental. This parameter has not been investigated to date. The aim of this study was therefore to characterize the effects of impurities on lactose 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). The concentrate was cooled in a tank from 79 °C to 11 °C in about 18 h for lactose crystallization purposes. For the washing step, 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). The 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 slurry were run through the decanter once more, and then dried on an Anhydro SFD 47 spin flash dryer (SPX Flow Technology, Søborg, Denmark) with an inlet temperature of 105 °C and an outlet temperature of 82–87 °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 poured into airtight plastic containers of various sizes to minimize the headspace. The powders were stored at 20 °C before analysis.

Pharmaceutical grade lactose (Lactochem[®] Crystals, batch number 663108) was purchased from DFE pharma (Goch, Germany) for comparison with the experimental lactose powders produced at different washing grades. Pharmaceutical grade lactose is produced industrially from edible grade lactose by re-dissolving the lactose in clean water followed by additional purification steps (Paterson, 2009). It is therefore the most pure lactose available on the market. Pharmaceutical grade lactose was mixed with distilled water to make a 15% (w/w) lactose solution which was left to stand at 40 °C for 1 h. The solution was then cooled to 20 °C and spray

dried on a pilot-scale spray dryer (GEA Niro A/S, Mobile Minor Dryer (MMD), Soeborg, Denmark) to obtain amorphous lactose. The inlet and outlet air temperatures were 200 °C and 90 °C, respectively, and the feed flow rate was 40 mL·min⁻¹.

2.2. Chemical composition

Protein, moisture 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 moisture and ash content was carried out in triplicate and the protein content was determined in duplicate. The lactose content was then calculated by difference. Individual minerals (calcium, phosphorus, sodium, potassium and magnesium) were measured by inductively coupled plasma optical emission spectrometry (ICP-OES) on an Optima 2000 DV (PerkinElmer, Waltham, Massachusetts, USA). Chloride was determined by potentiometry. All minerals were analyzed in duplicate.

2.3. Sieving and measurement of particle size

The lactose powders of different washing grades were sieved to separate 80, 160, 250, 355 and 500 μ m fractions. The particle size distribution of the powders was measured by laser light scattering using a Malvern Mastersizer 2000 equipped with a Scirocco 2000 dry dispersion unit (Malvern Instruments, Worcestershire, UK).

2.4. Moisture sorption measurements

Sorption isotherms of powders were obtained with a Dynamic Vapor Sorption (DVS) Advantage (Surface Measurement Systems Ltd., London, UK) equipped with a Cahn microbalance. The experiments were carried out in duplicate at a constant temperature (25 °C) using a nitrogen flow rate of 200 standard cm³·min⁻¹. Approximately 40 mg of powder was subjected to ramping of RH from 0% to 95% in 10%–RH steps with water as solvent. Equilibrium was considered to be reached if the rate of change in mass was less than 0.0002%·min⁻¹.

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 aluminium stub and coated with a thin layer of gold (JEOL JFC-1300 auto fine coater) prior to analysis. The photomicrographs were taken at \times 1000 magnification.

2.6. Solid-state nuclear magnetic resonance (NMR)

¹³C NMR spectra were obtained using proton decoupling, magic angle spinning (MAS) and cross polarization (CP). The spectra were recorded on a Bruker Avance I WB 300 MHz (7T) instrument (Bruker, Billerica, USA) at ambient temperature according to the method described by Gustafsson et al. (1998), with the following parameters: spinning rate 5 kHz, contact time 2 ms, acquisition time 147 ms, sweep widths 2190 ppm and delay between pulses of 3 s. For each spectrum, about 150,000 transients were cumulated with 49 k data points. The spectra were referenced to trimethylsilane (TMS).

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