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Effect of lactic acid in-process crystallization of lactose/protein powders during spray drying

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ABSTRACT

Spray drying of acid whey is a major problem for the dairy industry due to its high content of lactic acid. This study has investigated the effect of lactic acid concentration on the final product properties from spray-drying lactose solutions. A lactose/lactic acid solution, with various ratios of lactose to lactic acid, and at a 10% (w/w) lactose concentration has been spray dried using a Buchi-B290 mini spray dryer with an inlet temperature of 180 °C. Moreover, the crystallization kinetics of lactose/protein solutions have been studied for their in-process crystallization characteristics during spray drying, especially when the lactic acid is added to the solution to imitate the composition of a typical acid whey solution. Gravimetric moisture sorption tests were performed on the spray-dried powders processed under these conditions and compared with X-ray powder diffraction and modulated differential scanning calorimetry (MDSC) analyses to measure the degree of crystallinity. It has been found that very large changes in the degrees of crystallinity for the final spray-dried product have occurred with increasing the lactic acid concentration. The yields from spray drying have also been significantly decreased at higher concentrations of lactic acid. The results of this study have implications in choosing the processing conditions to produce and control crystallization in carbohydrate-containing dairy powders and also powders of acid-rich foods, such as fruit juices, during spray and fluidized-bed processing.

1. Introduction

Whey is a multicomponent solution of various water soluble milk constituents in water, which is the by-product remaining after the production of cheese or the removal of fat and casein (80% of the proteins) from milk (De Wit, 2001; Jelen, 2009). There are two basic types of whey: sweet and acid whey by acidification to below pH 5.0 (Table 1). While the main components of both sweet and acid wheys, after water, are lactose (approximately 70–72% of the total solids), whey proteins (approximately 8–10%) and minerals (approximately 12–15%), the main difference is found in the calcium and lactic acid contents (Panesar et al., 2007; Jelen, 2009).

Most milk plants do not have a fully-developed strategy to recycle whey, and around half the global production of whey is treated as a waste product (Salameh and Taylor, 2006). Due to the presence of economically-important components, such as lactose and the whey proteins, the disposal of whey solutions to drain is no longer acceptable. Whey and whey products as functional ingredients in food and pharmaceutical applications, and as nutrients in dietetic and health foods, have received considerable attention (De Wit, 2001).

Different types of whey products contain some of the most valuable milk nutrients, but the processing of whey into marketable attractive final products faces major economical obstacles (Jelen, 2009). Among the different ingredients of milk powders, amorphous lactose is the most hygroscopic and unstable material. The stickiness of powders containing amorphous materials relates to composition and their associated T_g values (Roos and Karel, 1991; Boonyai et al., 2004; Paterson et al., 2005). Reducing stickiness in materials can be achieved through partial or complete crystallization of the sticky components (Nijdam et al., 2008; Imtiaz-Ul-Islam and Langrish, 2009; Yazdanpanah and Langrish, 2011b). Due to the rapid removal of water in spray drying, amorphous solids may be formed (Roos, 1995). Amorphous materials have disordered and tangled atomic or molecular arrangements and may exist as solid "glasses" or paste-like "rubbers". The transition between these states occurs at or above the glass-transition temperature (T_g), which is specific for each material (Roos and Karel, 1991).

Above the glass-transition temperature, the molecular mobility increases, as shown by a decrease in viscosity (Burnett et al., 2004). Water (moisture content) acts as a plasticizing, or softening agent,

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Table 1
The typical composition of sweet and acid whey.

Components	Sweet whey (g/l)	Acid whey (g/l)
Total solids	63–70	63–70
Lactose	46–52	44–46
Protein	6–10	6–8
Calcium	0.4–0.6	1.2–1.6
Phosphate	1–3	2–4.5
Lactate	2	6.4
Chloride	1.1	1.1

(*Source:* Jelen, 2009; Panesar et al., 2007).

in amorphous food materials, significantly lowering the overall glass-transition temperature (Roos, 1995). The extent of T_g depression depends on the concentration of the plasticizer and its interaction with the amorphous material (Roos, 1995). Above the glass-transition point, amorphous materials of low molecular weight will crystallize, and the water sorption capacity decreases, resulting in mass loss since excess water is desorbed during crystallization (Roos and Karel, 1991; Burnett et al., 2004). Crystallization from amorphous materials in the solid state occurs when molecules of the amorphous solids rearrange themselves into an orderly structure (Jouppila and Roos, 1994a). The transformation from solid amorphous to solid crystalline products has been investigated by several researchers. They suggested that the crystallization rate of various amorphous sugars can be estimated by using one of the following approaches: the Williams–Landel–Ferry (WLF) equation (Williams et al., 1955), the Avrami equation or the activated-state model (Das and Langrish, 2012). The difference between the glass-transition temperature (T_g) of the materials and the process temperature (T) has been widely accepted as a best indicator to avoid this stickiness during drying (Bhandari et al., 1997; Islam et al., 2010). According to the Williams–Landel–Ferry (WLF) equation, a higher particle temperature and lower particle glass-transition temperature increase the crystallization rate of the particles during the spray-drying process.

It has been found that the spray drying of acid whey is not an easy task because powder sticks to the dryer and cyclone walls due to the high content of lactic acid and the low pH (Modler and Emmons, 1978; Salameh and Taylor, 2006; Bhandari, 2008). If the drying conditions and the degree of lactose crystallization are similar, then differences in drying properties can be linked to the lactic acid–lactate content of these products (Modler and Emmons, 1978). Therefore, the rationale of this study is to investigate the effects of lactic acid concentration on the degrees of powder crystallinity during spray drying of lactose solution with various acid concentrations.

The main ingredients of the acid whey are lactose, protein, and lactic acid. Due to the importance of lactose in the food and pharmaceutical industries, there have been extensive studies on crystallization behavior of lactose (Roos and Karel, 1992; Jouppila et al., 1998). The effect of lactic acid on crystal growth of lactose should have been studied due to its fundamental and commercial importance in food processing. However, there is little known about the crystallization behavior of lactose in the presence of lactic acid (Bhandari, 2008), and it has been found that presence of protein at low concentrations (<5 wt.%) significantly affects the product yield and crystallinity (Yucel and Coupland, 2010). The crystallization kinetics of lactose/protein solutions, especially when lactic acid is added to the solution, for example acid whey, have not been studied in terms of in-process crystallization during spray drying. It is difficult to predict the performance of the crystallization of commercial acid whey due to the presence of other impurities. Therefore in one series of experiments in this paper, only solid-phase crystallization of lactic acid and lactose was carried out. Then further experimentation has been done on the crystallization behavior of powders produced during spray drying using solutions of lactose/protein/lactic acid to imitate the composition of typical acid whey solutions. Different methods have been used to assess the degree of crystallinity of the spray-dried products produced under different concentrations of lactic acid.

2. Materials and methods

2.1. Sample preparation

In these experiments, pure α-lactose monohydrate crystals ($C_{12}H_{22}O_{11}\cdot H_2O$; analytical reagent, Australia), lactic acid 88% w/w ($C_3H_6O_3$; laboratory reagent from Chem-Supply, Australia), and whey protein isolate (Balance, Vitaco Health Ltd, Auckland, New Zealand) were used in this study and dissolved in deionized water. The package label of this WPI indicates the composition (per 100 g) as: 92 g protein, 0.4 g fat (total) including 0.2 g saturated fat, 0.5 g carbohydrate, sodium 0.6 g. One series of experiments was carried out by varying the composition of the solution with additions of whey protein isolate from 1% to 10% (w/w), while maintaining the level of lactose concentration in solution at 10% (w/w). Further experimentations has been done by adding lactose and whey protein isolate to create solutions with 15% (w/w) lactose and 5% (w/w) WPI (lactose lactose/WPI ratios of 75:25 (w/w)), and then varying the composition of the solution with additions of lactic acid from 1% to 20% (w/w). Each solution was made up to a total weight of 100 g. The composition of the solution was used at that ratio, so as to imitate the typical composition of whey solution(s) regarding the sugar and protein amounts (Panesar et al., 2007; Jelen, 2009). All solutions were magnetically stirred at the room temperature of 25 °C for at least 30 min, so a clear solution was obtained without any visible crystals being present. The clear solutions were then spray dried. The pH of the solutions was measured with a pH electrode, InPro 3250 series (Mettler Toledo, M 300, Switzerland).

2.2. Operating conditions for spray drying

The solutions were spray dried using a Buchi-B290 Mini Spray Dryer with an inlet gas temperature of 180 °C, an aspirator setting of 100% (38 m^3/h), pump rate of 25% (8 mL/min), and a nozzle air flow rate of 470 L/h (40 on the nozzle rotameter scale). These operating conditions caused the outlet gas temperatures to be 108 ± 3 °C and 112 ± 4 °C for lactose and lactose/WPI solutions, respectively. All experiments were performed in triplicate. Freshly spray-dried powder was collected from a collection vessel at the bottom of a cyclone and was immediately used for analytical tests (moisture content, MDSC analysis and gravimetric moisture analysis); otherwise the powders were kept in sealed bags and in a refrigerator for X-ray diffraction tests on the following day.

2.3. Powder characterization

2.3.1. Moisture sorption and oven drying tests

Crystallization behavior was studied with two repeat samples of the powders produced from spray drying. A mass of 1–2 g of the powder was placed on a 10-mm-diameter borosilicate glass Petri dish with a nearly monolayer particle thickness. The mass change as a function of storage time was recorded by computerized data-logging once per minute over a period of at least 6 h to reach a constant mass by using an analytical balance (±0.0001 g, Mettler Toledo, AB 204-S, Switzerland). The sample and the balance were placed in a sealed box where the relative humidity (70–75% RH) and the temperature (24.5–25 °C) were kept constant using a