



Lactose crystallization as affected by presence of lactic acid and calcium in model lactose systems



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ABSTRACT

Acid whey is considered a hard to process stream mainly due to failure of lactose to crystallize, apparently attributed to the presence of lactic acid (LA) and calcium (Ca). Understanding the physical state and the thermal behaviour of lactose during crystallization in the presence of LA and Ca is important in order to develop adequate strategies to improve processability of acid whey and thus was the objective of this study. Presence of high concentration of LA (1% w/w) resulted in a decline in lactose crystal yields (~59%). This was alleviated by lowering the LA content (0.2% w/w) which improved the yield (~77%). At high LA content, small amounts of Ca (0.072% w/w) appeared to diminish the negative impact of LA presence by increasing the yield. Pure L solution yielded lactose particles of ~84 μm . Crystal size slightly increased to ~91 μm in the system containing high concentrations of LA along with low Ca concentration and although the presence of low concentrations of both LA (0.2% w/w) and Ca (0.072% w/w) resulted ~163 μm sized crystals. Water molecules in the hydration layer around lactose molecules appeared to play a significant role governing the crystallisation behaviour and characteristics of lactose crystals in the presence of LA and Ca and/or in combination. Hence, the removal of some Ca to concentrations $\leq 0.072\%$ w/w from acid whey may improve the crystallisation of lactose and thereby improve the processability of acid whey.

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1. Introduction

Whey, the serum phase of milk, is traditionally classified as *sweet whey*, a by-product of renneted cheese manufacturing, and *acid whey*, originating from the production of acid coagulated dairy products. In the past, the dairy industry faced a significant problem with the sweet whey disposal which created an enormous environmental problem due to organic pollution (De Wit, 2001). The problem was finally solved by concentrating sweet whey by evaporation to ~45–60% solids followed by crystallization of lactose and spray drying to produce a non-sticky free flowing whey powder, which is currently used in different food applications (De Wit, 2001). However, acid whey (AW) is considered a hard to process stream mainly due to failure of lactose to crystallize, which consequently remains in its amorphous form during concentration and interferes with spray drying (Gwenole et al., 2013). In comparison to sweet whey, acid whey contains higher levels of lactose and more minerals, but less proteins, and more importantly, more

lactic acid (Prazeres et al., 2012). Some of the industrial approaches reported lumping and caking of whey powder particles during spray drying of acid whey and such behaviour of lactose in AW has been attributed to the presence of lactic acid and Ca (Dec and Chojnowski, 2006).

Lactose in dairy systems can exist in various crystalline and non-crystalline forms. Storage stability and quality of certain foods are significantly affected by the physical state of lactose (Omar and Roos, 2007). Different food applications require varying extents of sugar crystallinity in food systems. As an example, achieving appropriate lactose crystallinity in dairy powders is very important (Islam and Langrish, 2010), which is also used to predict potential of dairy powders to cake. In solid confectionaries, the presence of a crystalline state or a thermodynamically unstable amorphous state affects both the appearance and textual properties (Kedward et al., 2000).

Lactose molecule has a hemiacetal structure and thus enables the molecule to interchange between α and β anomers by mutarotation. The α and β ratio depends on the compositional and processing conditions such as temperature, concentration, pH and presence of foreign substances (Hartel and Shastry, 1991).

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Occurrence of these two anomeric forms complicates the prediction of lactose functionality (Islam and Langrish, 2010). Various lactose crystallisation processes in the food industry are still far from optimized. Furthermore, lactose in food systems is often present in a mixture with other food components such as salts and proteins, which in turn affects the crystallisation process (Nijdam et al., 2007). Hence understanding the physical state and thermal behaviour of lactose crystals in different food systems with compositional variations has a great importance in development of proper processes and prediction of food quality (Hartel, 2001).

Lactose crystallisation is a key step in manufacturing of whey powders. Crystal characteristics affect the subsequent step of spray drying and whey powder quality (Mimouni et al., 2005). Every crystallisation process commences from a supersaturated solution and proceeds via 2 distinct phases (Wong and Hartel, 2014). The first phase, commonly termed nucleation, is the initial creation of zero-sized crystals (nuclei) which appear as a result of clustering and aggregation of molecules or ions in a supersaturated solution. Nucleation is usually described as either primary, initiated in presence or absence of suspended particles that promote formation of clusters, or secondary, which requires initiation by crystal seeding. The second phase is the growth of nuclei where the clusters of molecules or ions are steady and grow into noticeable sizes. The crystal growth includes three main mechanisms: mass transport, surface reaction and transport of latent energy away from growing crystals (Wong and Hartel, 2014).

Lactose crystallisation is usually retarded by presence of various impurities. For example, a type of salt and its concentration impaired lactose crystal formation (Herrington, 1934a; Jelen and Coulter, 1973b). Similarly lactic acid was considered an inhibitor of lactose crystallisation (Jelen and Coulter, 1973b), although there has not been a systematic study yet to prove it. In addition, the growth of lactose crystals appears affected by numerous process parameters and the condition of the solution such as degree of super-saturation, solution temperature, viscosity and pH (Bhargava and Jelen, 1996).

In the past, number of studies was conducted to elaborate on crystallization behaviour of lactose (Jouppila et al., 1998). However, there has been little new information regarding crystallization behaviour of lactose in the presence of lactic acid and calcium. A recent study focussed on the behaviour of spray dried lactose powders in model lactose solutions in the presence of lactic acid (Saffari and Langrish, 2014). However, the approach involved using up to 20% w/w lactic acid, which would be comparatively greater than that in acid whey systems. As no industrial solution for acid whey processing has been found, mainly due to the failure of lactose to crystallize, which thus remains in its amorphous form during concentration and hinders further processing, including spray drying and it is of utmost importance to understand behaviour of lactose in the presence of varying lactic acid and Ca concentrations. The main objective of the present study was to provide answers to a number of fundamental questions relating to crystallisation behaviour of lactose and its interactions with other constituents of acid whey in order to resolve the unprocessability of acid whey. Hence, the current study focused on establishing crystallisation behaviour of lactose in the presence of up to 1% w/w lactic acid as it reflected better lactic acid concentrations encountered in industrially obtained retentates that are further processed by spray drying. These retentates, frequently acquired by nano-filtration, contain varying concentrations of lactic acid and calcium, depending on the dairy production stream, which may influence lactose behaviour. Lactose crystallisation behaviour was assessed in lactose model systems containing lactic acid concentrations from 0.2% to 1% w/w and calcium concentrations of 0.12% w/w and 0.072% w/w. The outcomes of the present study would allow for

establishing necessary conditions to achieve appropriate lactose crystallization needed for further processing of acid whey and thereby lead to a solution to acid whey unprocessability.

2. Materials and methods

2.1. Materials

A commercial food grade lactose powder and an analytical grade lactic acid solution were obtained from Murray Goulburn Co-operative Co.Ltd (Brunswick, Australia) and Sigma–Aldrich Pvt. Ltd. (Castle Hill, NSW, Australia), respectively. Anhydrous calcium chloride with a purity of $\geq 97\%$ obtained from Sigma–Aldrich was used.

2.2. Preparation of model solutions

Appropriate amount of lactose powder was dissolved in Milli-Q water at room temperature ($22 \pm 3^\circ\text{C}$) to obtain a 5% w/w lactose solution (L). Lactose plus lactic acid (L + LA) solutions were prepared by the addition of 1, 0.6, 0.4 or 0.2% w/w lactic acid to the 5% w/w lactose solution, respectively. Lactose plus high calcium (L + high Ca) solutions were prepared by the addition of 0.12% w/w Ca and lactose plus low calcium (L + low Ca) solutions were made by adding 0.072% w/w Ca with the use of a 2.4 w/w % stock calcium chloride solution. The solutions were stirred using a magnetic stirrer for about 30 min until clear solutions were obtained where no crystals were visually observed. All solutions were filtered using 0.45 μm Whatman filter papers to remove any undissolved particles and the final weight was adjusted if required.

2.3. Concentration

Concentration of the samples was performed using a rotary evaporator (EYELA, Tokyo, Rikakikai Co. Ltd. Japan) to achieve $48 \pm 2\%$ w/w total solids. Temperature of the rotary evaporator water bath was adjusted to 55°C to imitate the industrial conditions. Regular refractometry measurements were performed measuring the Brix value using a laboratory refractometer (Atago abbe, Tokyo, Japan) for concentration determinations after calibrating it with pure MilliQ water.

2.4. Crystallization procedure

Exactly 50 mL aliquots of concentrated solutions were subjected to crystallisation using the method adapted by Mimouni et al. (2005). The solutions were rapidly cooled to the crystallisation temperature of 30°C . Then the solutions were slowly cooled down to 25°C , 20°C and 15°C over 3 h at a rate of 0.08°C per min using a water bath consisting of a cooling unit. The solutions were continuously stirred with the use of magnetic stirrer to avoid sedimentation of lactose crystals as well as to transport the supersaturated solution to the surface of the crystals. The stirring rate was fixed in all experiments. The samples were then kept at 15°C overnight with continuous stirring. The next day, the lactose crystals were separated using 0.45 μm filter papers. Lactose crystals were oven dried at 80°C for 4 h. The percentage yield of lactose was calculated as (Wong and Hartel, 2014):

$$\% \text{ Yield} = \frac{\text{Mass of lactose crystal obtained} \left(\frac{\text{g}}{100\text{g water}} \right)}{\text{Maximum theoretical crystal yield} \left(\frac{\text{g}}{100\text{g water}} \right)} \times 100$$

The final solubility was taken as 19.1 g lactose/100 g water

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