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Evaluating the crystallization of lactose at different cooling rates from milk and whey permeates in terms of crystal yield and purity

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ABSTRACT

The cooling rate of supersaturated lactose solution is one of the important parameters determining the yield and size distribution of lactose crystals. The influence of increasing cooling rate on lactose crystallization and quality of lactose crystals was evaluated in concentrated solutions prepared from deproteinized whey powder (DPW) and milk permeate powder (MPP). Concentrated permeates (DPW and MPP) with 60% (wt/wt) total solids were prepared by reconstituting permeate powders in water at 80°C for 2 h for lactose dissolution. Three cooling rates, 0.04°C/min (slow), 0.06°C/min (medium), and 0.08°C/min (fast) were studied in duplicate. A common rapid cooling step (80 to 60°C at 0.5°C/min) followed by slow, medium, and fast cooling rates were applied as per the experimental design from 60 to 20°C. After crystallization, the crystal slurry was centrifuged, washed with cold water, and dried. The dried lactose crystals were weighed to calculate the lactose yield. Final mean particle chord lengths were measured at the end of crystallization using focused beam reflectance measurement for slow, medium, and fast cooling rates, and observed to be not significantly different for DPW (27–33 μm) and MPP (31–34 μm) concentrates. Similarly, the lactose yield for slow, medium, and fast cooling rates in the DPW and MPP concentrates were in the range of 71 to 73% and 76 to 81%, respectively, and no significant difference between the 3 cooling rates was found. Qualitative analysis of dried lactose crystals exhibited no noticeable differences in the crystal purity with increasing cooling rate. This study evaluated the possibility of reducing the crystallization times by 8 h compared with current industrial practice without compromising the crystal yield and quality.

Key words: deproteinized whey, milk permeate powder, lactose crystal quality, focused beam reflectance measurement

INTRODUCTION

The growing demand for high-protein dairy ingredients led to fractionation of sweet whey and skim milk into a protein-rich fraction and protein-depleted fraction, often referred to as permeate. Milk permeate powder (**MPP**) and deproteinized whey (**DPW**) are the permeate fractions obtained during manufacturing of milk protein concentrate and whey protein concentrates, respectively, using UF and subsequent spray drying. Lactose is one of the major constituents in DPW and MPP, along with a small fraction of soluble minerals and proteins. Deproteinized whey is composed of 76 to 85% lactose, and 11 to 16% proteins and minerals, whereas MPP is composed of a higher lactose content of 78 to 88%, with 11 to 16% proteins and minerals (US Dairy Export Council, 2015).

Lactose is used as an ingredient in infant formulations, food products, and the pharmaceutical industry and is manufactured using the crystallization process from whey and milk permeates. The aim of crystallization is to recover lactose in the most stable and nonhygroscopic α-lactose monohydrate form and consequently prevent storage defects such as agglomeration and caking (Carpin et al., 2017). A typical lactose production at an industrial scale involves concentration of permeate to 60 to 65% TS followed by a gradual cooling, decantation, washing, and drying (Wong and Hartel, 2014). For an economical industrial production of lactose, maximum crystal yield is preferred. To avoid the loss of smaller crystals during decantation and washing steps, it is necessary to promote lactose crystal growth to maximize the lactose yield (Pandalaneni and Amamcharla, 2016).

Quality and yield of lactose are dependent on factors such as presence of impurities, agitation, cooling rate during crystallization, crystallizer design (Wong et al., 2012), and degree of supersaturation (McLeod et al.,

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2011). Presence of whey proteins and minerals negatively affected the quality and yield of lactose (Modler and Lefkovitch, 1986; Lifran et al., 2007; Chandrapala et al., 2016), whereas additives like soybean polysaccharide significantly improved the lactose yield (Sunkesula et al., 2017). Crystallization is considered the most important step in lactose recovery that includes approximately 6 h to fill the crystallization tanks and 14 to 18 h for the crystallization process, accounting for a total of 20 to 24 h of processing (Wong et al., 2012). As the supersaturated solution is gradually cooled, lactose solubility decreases and the supersaturation drive increases. The supersaturation driving force is required for the formation of lactose crystals (McLeod et al., 2011). When supersaturated lactose solution is rapidly cooled, nucleation is dominant and the risk of obtaining lactose crystals with smaller particle size is high. On the other hand, when cooled slowly, lactose crystal nucleation and growth occur simultaneously, resulting in bigger lactose crystals (McLeod, 2007).

During crystallization, if nucleation is spontaneously induced by supersaturated lactose, it is referred to as homogeneous nucleation. On the other hand, if nucleation is induced by a foreign substance, it is called heterogeneous nucleation. Heterogeneous nucleation is also dependent on the solubility of impurities present in the solution (Mullin, 2001). During crystallization, along with lactose, impurities also settle on the lactose crystal lattice (Lifran et al., 2007). Impurities settled over the lactose crystals are removed during the crystal washing step, but impurities settled in the crystal lattice can affect the quality of lactose crystals. Hence, understanding the effect of cooling rate on the lactose crystallization in presence of impurities is critical.

Because information is lacking on the crystallization of lactose in whey permeate and milk permeate at concentrations relevant to industrial practice, this study was focused on characterizing the lactose crystallization and quality of lactose crystals at different cooling rates in concentrated permeates. One of the traditionally used cooling rates (0.04°C/min) was compared with relatively faster cooling rates using focused beam reflectance measurement (**FBRM**) and other techniques. The influence of cooling rate and naturally present impurities in the concentrated permeates on lactose crystallization and quality were evaluated in this study.

MATERIALS AND METHODS

Experimental Design

The effect of cooling rate during lactose crystallization in concentrated DPW and MPP solutions was studied at 3 levels: slow (0.04°C/min), medium (0.06°C/min), and fast (0.08°C/min). All the experiments were conducted randomly and in duplicate. One of the 3 cooling rates (slow, medium, and fast) was applied during cooling from 60 to 20°C, accounting for the total crystallization period of 1,040, 706, and 540 min, respectively.

Preparation of Supersaturated Lactose Solution from Permeates

Deproteinized whey powder with 78.3% (dry basis) lactose and MPP with 86.6% (dry basis) lactose were acquired from Davisco Foods International Inc. (Le Seuer, MN) and Idaho Milk products (Jerome, ID), respectively. Concentrated DPW solution of 900 g with 60% (wt/wt) TS was prepared by gradually reconstituting 540 g of DPW into 360 g of distilled water at 80°C for 2 h under constant stirring. Complete dissolution of lactose was confirmed by microscopic examination. Concentrated MPP solution with 60% TS was also prepared following the same procedure as described for concentrated DPW. Fully solubilized permeate solution was then transferred into a crystallizer maintained at 80°C and 1 of the 3 cooling rates was used to crystallize the lactose in a permeate system. No lactose seeds were added during the crystallization in this study.

Experimental Setup and Lactose Crystallization

Lactose Crystallization. Lactose crystallization was carried out in a custom-built double-jacketed crystallizer attached to a programmable water bath (Polystat Standard, Cole-Parmer, Court Vernon Hills, IL) as shown in Figure 1. A custom-made 4-blade anchor propeller was attached to an overhead stirrer (Caframo, Georgian Bluffs, Ontario, Canada) to facilitate the agitation. From preliminary experiments (data not reported), an agitation speed of 140 rpm was selected to keep the crystals in suspension with minimal secondary nucleation due to breakage of crystals observed from microscopic images. A temperature probe connected to a data logger (HOBO, Bourne, MA) was used to acquire the crystallizer temperature every 10 s. The crystallizer was covered with a lid to avoid any water loss. Three cooling rates were studied with a same initial flash cooling from 80 to 60°C at 0.5°C/min. The initial common cooling rate was used in this study to simulate flash cooling step commonly used in the industrial practice. For in situ monitoring of lactose crystallization, FBRM (Particle Track E25, Mettler-Toledo AutoChem Inc., Columbus, OH) was installed in the crystallizer at a $30 \pm 5^{\circ}$ angle as described by Pandalaneni and Amamcharla (2016).