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# Online ice crystal size measurements during sorbet freezing by means of the focused beam reflectance measurement (FBRM) technology. Influence of operating conditions

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## ABSTRACT

In ice cream and sorbet manufacturing small ice crystals are desired to deliver a product with a smooth texture and good palatability. This research studied the influence of the operating conditions on the ice crystal size and the draw temperature of the sorbet during the freezing process. The evolution of ice crystal size was tracked with the focused beam reflectance measurement (FBRM) technique, which uses an in situ sensor that makes it possible to monitor online the chord length distribution (CLD) of ice crystals in sorbets containing up to 40% of ice. The refrigerant fluid temperature had the most significant influence on the mean ice crystal chord length, followed by the dasher speed, whereas the mix flow rate had no significant influence. A decrease in the refrigerant fluid temperature led to a reduction in ice crystal size, due to the growth of more small ice crystals left behind on the scraped wall from previous scrapings. Increasing the dasher speed slightly reduced the mean ice crystal chord length, due to the product of new small ice nuclei by secondary nucleation. For a given refrigerant fluid temperature and dasher speed, low mix flow rates resulted in lower draw temperatures, due to the fact that the product remains in contact with the freezer wall longer. High dasher speeds warmed the product slightly, due to the dissipation of frictional energy in the product, the effect of which was in part moderated by the improvement in the heat transfer coefficient between the product and the freezer wall.

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## 1. Introduction

In the manufacturing process of frozen desserts such as sorbet and ice cream, three main steps can be distinguished: preparation of the mix, initial freezing, and hardening of the product. The first step includes the blending of all the ingredients, its pasteurization and homogenization, as well as the cooling and ripening of the mix at 5 °C. During the initial freezing step, the mix is pumped through a scraped surface heat exchanger (SSHE) or freezer. The evaporation of the refrigerant fluid in the jacket of the freezer cools down the temperature of the mix below its freezing point, and causes formation of an ice layer at the wall of the freezer barrel (Cook and Hartel, 2010). Subsequently, the scraper blades of the rotating dasher remove the ice layer from the freezer wall. The small ice crystals contained in the ice layer are dispersed into the centre of the freezer barrel, where they grow and become disc-shaped ice crystals that exit the freezer with a mean size of 15-27 um (Drewett and Hartel, 2007; Marshall et al., 2003; Russell et al., 1999; Sofjan and Hartel, 2004). At this stage, roughly half of the total amount of water is frozen (Hartel, 1996). Depending on the operating conditions of the process, the draw (exit) temperature of the product varies from -4 to -6 °C, having an adequately low viscosity to be pumped for moulding and packaging. Further on, in the hardening step, the product is introduced into a blast freezer to attain a core temperature of  $-18 \,^{\circ}$ C (Cook and Hartel, 2010), where roughly 80% of the amount of water is frozen (Marshall et al., 2003). Since the subcooling rate during hardening is not high enough to form new nuclei, the increase in the amount of ice formed with the decrease in temperature follows the equilibrium freezing point curve and leads to the increase in size of the existing





Abbreviations: ANOVA, analysis of variance; CLD, chord length distribution; CSD, crystal size distribution; CV, coefficient of variation; DSC, differential scanning calorimetry; DRS, dasher rotational speed; DT, draw temperature; FBRM, focused beam reflectance measurement; MCL, mean chord length; MFR mix flow rate; PSD, particle size distribution; PSP, polyamid seeding particles; SS, sum of squares; SSHE, scraped surface heat exchanger; TR22, evaporation temperature of r22.

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Nomenclature			
$\hat{Y}_i$ $Y_{ij}$ $\bar{Y}_i$ $\beta_0$ $\beta_i$ $\beta_{ii}$ $\beta_{ij}$ $X_i$	predicted value of the response experimental response mean response value regression coefficient for interception effect. regression coefficient for linear effect. regression coefficient for quadratic effect. regression coefficient for interaction effect. coded values of the experimental factors	C dj ni Ci X <sub>m.i.</sub> X <sub>ms.i</sub> X <sub>ms.f</sub>	experimental points replicated experimental points number of particles for each of the size classes <i>i</i> chord length ice mass fraction initial mass fraction of solute (sweetener content) final mass fraction of solute (sweetener content)

ice crystals (Marshall et al., 2003). Hence, the final ice crystal size of the product will be dictated to a large extent by the evolution during the hardening step of the ice crystals that were formed during the initial freezing. Consequently, the initial freezing process is the most critical step in ice cream and sorbet manufacturing.

The mechanism of ice crystallization within a freezer is affected mainly by the operating conditions of the freezing process, such as the evaporation temperature of the refrigerant fluid, the dasher rotational speed and the mix flow rate. The temperature of the refrigerant fluid provides the driving force that triggers ice nucleation and it determines the heat removal rate of the system. During freezing, ice nucleation occurs at the freezer wall, where there is enough subcooling (roughly –30 °C) between the refrigerant fluid and the mix to form ice nuclei (Hartel, 1996). On the basis of dendritic growth observations in quiescently-frozen sucrose solutions on a chilled surface, Schwartzberg and Liu (1990) suggested that due to the high rate of subcooling at the heat exchange cylinder wall, dendrites are likely to grow there, then are cut off and dispersed into the bulk flow by the scraper blades of the dasher. Subsequently, ice nuclei ripen and become disc-shaped ice crystals in the bulk warm region of the freezer (Cook and Hartel, 2010). Schwartzberg (1990) reported that the space between dendrites was proportional to the freezing rate to the -1/2 power, which means that high subcooling rates lead to a faster growth of more dendrites, with closely spaced branches and a thinner structure. More recently, on the basis of thermal conductivity measurements of a sucrose solution in a flowcell equipped with a scraper blade and a chilled surface, Zheng (2006) concluded that the ice layer formed at the freezer wall was in fact a slush layer composed of both ice and concentrated sucrose solution. Zheng (2006) also found that after each scrape of the blade, many ice nuclei grew rapidly from the ice debris remaining from previous scrapings, and continued to grow along the chilled surface before merging and growing vertically. Hence, a decrease in temperature of the refrigerant fluid would be expected to enhance the cooling rate, causing the faster formation of more ice crystals from the ice debris left behind from previous scrapings, which will grow with a thinner structure and lead to smaller ice crystal sizes.

The scraping action of the dasher improves the heat transfer rate between the freezer wall and the product (Ben Lakhdar et al., 2005). Higher dasher speeds would thus be expected to give lower draw temperatures and smaller ice crystals. However, an increase in dasher speed would also increase the amount of frictional heat generated by the blades, which could dissipate into the product, producing warmer draw temperatures, the melting of the small ice nuclei, and consequently, a reduction in the effective ice nucleation rate. Also, by increasing dasher speed, we increase the movement of the fluid within the freezer, which leads to the enhancement of ice recrystallization phenomena (Cebula and Russell, 1998). Furthermore, an increase in dasher speed may also lead to the attrition of the larger ice crystals (Haddad, 2009; Windhab and Bolliger, 1995), the remaining ice debris of which can lead to the formation of new ice nuclei through secondary nucleation. Sodawala and Garside (1997) used video microscopy to examine the freezing of a 10% sucrose solution on a cold surface with a rotating scraper blade. They observed the formation of ice flocs which grew parallel to the surface after each scrape, then merged and grew vertically. They also observed that an increase in the scraping frequency of the blade led to more frictional heat and to smaller flocs being cut off from the surface. Hence, increasing dasher speed would also be expected to produce new smaller ice nuclei formed from the remaining smaller ice flocs at the surface of the freezer wall.

The mix flow rate dictates the residence time of the product within the freezer, affecting the time available to remove heat from the product, and consequently, the ice nucleation and growth mechanisms of ice crystals. A number of studies in the literature have observed that high mix flow rates (short residence times) for a given draw temperature (by adjusting the temperature of the refrigerant fluid) and dasher speed produced smaller ice crystals due to the reduction in recrystallization phenomena in the bulk region of the product (Drewett and Hartel, 2007; Koxholt et al., 2000; Russell et al., 1999). For a given refrigerant fluid temperature (varying exit temperature) and dasher speed, Russell et al. (1999) also found smaller ice crystals produced at higher mix flow rates, the effect of which was attributed to the reduction in ice crystal coarsening. During the freezing of 30% sucrose/water solutions in an SSHE, Ben Lakhdar et al. (2005) reported that low product flow rates (long residence times) led to a reduction in the exit temperature of the product, and therefore to an increase of the ice mass fraction in the product.

Several studies have highlighted the importance of producing a narrow ice crystal size distribution (CSD) with a small mean size ( $<50 \mu m$ ) so as to confer a smooth texture to the final product and enhance consumer acceptance (Cook and Hartel, 2010; Drewett and Hartel, 2007; Hartel, 1996; Russell et al., 1999). It is therefore important to identify the operating conditions of the freezing process that most directly affect ice crystal size so as to improve the quality of the final product.

In order to characterize the ice CSD, many methods have been used. However, some of these methods partially destroy the ice crystal structure during sample preparation, and none of them have been able to directly measure the ice crystal size in the exit stream of the product during the freezing process. Recently, online techniques such as the focused beam reflectance measurement (FBRM) have been developed for in situ monitoring of CSD in the crystallization processes of chemical and pharmaceutical products (Barrett and Glennon, 2002; Negro et al., 2006). In the case of ice crystallization, Haddad et al. (2010) have successfully used the FBRM technique to follow the evolution of ice crystal size during batch freezing of sucrose/water solutions. The FBRM technique is based on the principle of a laser beam that is focused at the window of the tip of the measurement probe. The rotating optics inside the probe allows the laser beam to scan a circular path in Download English Version:

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