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Online ice crystal size measurements by the focused beam reflectance method (FBRM) during sorbet freezing.

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Abstract

The ice crystal size distribution determines in part the textural properties of sorbet and ice cream. During sorbet and ice cream manufacturing, a narrow ice crystal size distribution with a small mean size is desired, in order to obtain a smooth texture in the final product. This research studied the influence of the mix flow rate, the evaporation temperature of the refrigerant fluid and the dasher speed on the ice crystal size and the draw temperature during sorbet freezing, so as to identify optimal operating conditions. The evolution of the ice crystal size was followed by the focused beam reflectance method (FBRM), which uses an in situ sensor that provides accurate and repeatable information about the chord length distribution (CLD) of ice crystals. Our results showed that the FBRM sensor is a promising tool which makes it possible to monitor online the development of the ice crystals in sorbets containing up to 40% of ice. Decreasing the refrigerant fluid temperature allows us to reduce the ice crystal size and to lower the product's temperature, due to the increase of the supercooling driving force. High dasher speeds slightly decrease the ice crystal chord length, due to the attrition of the bigger ice crystals, which produces new smaller ice nuclei by secondary nucleation. Also, an increase of the dasher speed slightly warms the product, due to the dissipation of frictional energy into the product. Low mix flow rates result in lower draw temperatures because the product remains longer in contact with the freezer wall extracting thus more heat from the product.

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Keywords: Ice crystal size; Focused beam reflectance method; Freezing; Scraped surface heat exchanger.

1. Introduction

The quality of sorbet and ice cream is determined in part by the ice crystal size distribution (CSD) of the product. A narrow ice CSD with small ice crystals (<50 μm) is desired to confer a smooth texture and

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good palatability to the product. Therefore, it is important to identify the operating conditions that affect the most directly the ice crystal size, so as to improve the quality of the final product. The mechanism of ice crystallization within a freezer is affected mainly by the process conditions, such as the evaporation temperature of the refrigerant fluid, the dasher rotational speed and the mix flow rate. The evaporation temperature of the refrigerant fluid provides the driving force that triggers ice nucleation and it determines the heat removal rate of the system. The scraping action of the blades originates frictional heat which may be dissipated into the product and lead to the increase of its draw temperature [1]. The mix flow rate dictates the residence time of the product within the freezer, affecting thus the ice nucleation and growth mechanisms of the ice crystals. In order to characterize the ice CSD, many methods have been used in literature. However, none of these methods has been able to measure directly the ice crystal size in the product's stream during the freezing process. Recently, online techniques such as the focused beam reflectance method (FBRM) have been developed for in situ monitoring of CSD in crystallization processes. In the case of ice crystallization, Haddad A. *et al*. [2] have successfully used the FBRM technique to follow the evolution of the ice CSD during the batch freezing of sucrose/water solutions. The FBRM probe is a laser reflection technique, which provides real time information about the chord length distribution (CLD) of particulates. One of the main advantages of this technique is its suitability for in situ measurements of particulates at high solid concentrations. However, the FBRM technique gives no information about the particle’s morphology and it measures a CLD rather than a CSD. But this measure can be useful to follow the evolution of crystals' size. The aim of this research is to use the FBRM technique to study in situ the influence of the operating conditions on the ice crystal chord length as well as on the draw temperature during the freezing of lemon sorbet.

Nomenclature

y	Predicted value of the response
β_0	Regression coefficient for interception effect
β_i	Regression coefficient for linear effect
β_{ii}	Regression coefficient for quadratic effect
β_{ij}	Regression coefficient for interaction effect
$X_i X_j$	Coded values of the experimental factors
c_{mean}	Mean chord length (µm)
n_i	Number of particles for each of the size classes *i*
c_i	Chord length (µm)

2. Material and Methods

2.1. Experimental setup – sorbet freezing

The freezing of lemon sorbet mix (25.7 % w/w sweeteners solids, 0.5 % w/w locust bean gum/guar gum/ hypromellose stabiliser blend) was carried out in a continuous pilot freezer at a laboratory scale (WCB MF 50) with a maximum capacity of 0.021 kg s^{-1}. The dasher speed range of the freezer was varied from 57 to 105 rad.s^{-1} and the evaporation temperature of the refrigerant fluid R22 (Chlorodifluoromethane), was varied within the range of -10 to -20°C according to experimental conditions.

2.2. Experimental design and data treatment

A central composite experimental design was used to assess the influence of 3 operating conditions: mix flow rate (MFR), dasher rotational speed (DRS) and evaporation temperature of R22 (TR22), on the response variables of ice crystal chord length distribution (CLD) and draw temperature (DT) of sorbet. The central composite experimental design was composed of a 2^3 factorial design with experimental points at ±1, a 'composite' design with points at the extremes of the experimental region (±α, with α = 1.68) and a common central point of the two designs at zero [3].The experimental design was performed twice and 5 replicates of the central point were performed in order to provide enough information to estimate the experimental error. Table 1 shows the coded values of the experimental design and the real freezing operating conditions.

Table 1. Coded values of operating conditions for the central composite experimental design [a].

Process Conditions		*Coded Values*				
Factors	Coded variables	- α	-1	0	+1	+ α
MFR (kg.s^{-1})	X_1	0.007	0.010	0.014	0.018	0.021
TR22 (°C)	X_2	-10.60	-12.50	-15.25	-18	-19.90
DRS (rad.s^{-1})	X_3	57.07	62.83	78.54	94.25	104.72

[a] MFR = mix flow rate; TR22 = evaporation temperature of R22; DRS = dasher rotational speed.

Experimental data were analysed using response surface methodology and the second-order polynomial used to predict the experimental behaviour was the following:

$$y = \beta_0 + \sum_{i=1}^{3} \beta_i X_i + \sum_{i=1}^{3} \beta_{ii} X_i^2 + \sum \sum_{i<j=1}^{3} \beta_{ij} X_i X_j \quad (1)$$

where y is the response, β_0, β_i, β_{ii} and β_{ij} are regression coefficients for interception, linear, quadratic and interaction effects, respectively, and X_iX_j are coded levels of the experimental factors.

2.3. Ice crystal size measurements

The ice crystal chord length was measured online by using a Mettler-Toledo Lasentec® FBRM probe (Model S400A-8). This device generates a focused laser beam (780 nm) which scans a circular path at the interface between the probe's window and the particles in suspension. When a particle is intersected by the laser beam, it reflects the laser light throughout the time it has been scanned (cf. Fig. 1A). Simultaneously, the time period of reflection is detected by the FBRM probe and then multiplied by the tangential speed of the laser beam, yielding thus a distance across the particle, which is a chord length. The FBRM probe measures thousands of chords per second providing a CLD (number of counts per second sorted by chord length into 100 logarithmic size classes). Departing from this information, the mean chord length of the ice crystals was obtained by the following equation:

$$c_{mean} = \sum_{i=1}^{100} n_i c_i \Big/ \sum_{i=1}^{100} n_i \,, \tag{2}$$

where n_i is the number of particles for each of the size classes i of chord length c_i.

When performing our experiments the FBRM probe was inserted into the outlet pipe of the freezer with a 45° angle relative to the flow (cf. Fig. 1B), making it possible to renew continually the sorbet flow that was being measured. In order to avoid condensation at the inside surface of the FBRM probe window, a purge was carried out with nitrogen at 1 bar, with a flow rate of 5 l/min. Once the steady state of the freezer was established, the chord length acquisition data was synchronized with draw temperature and recorded every 5 seconds for a period of 10 minutes.

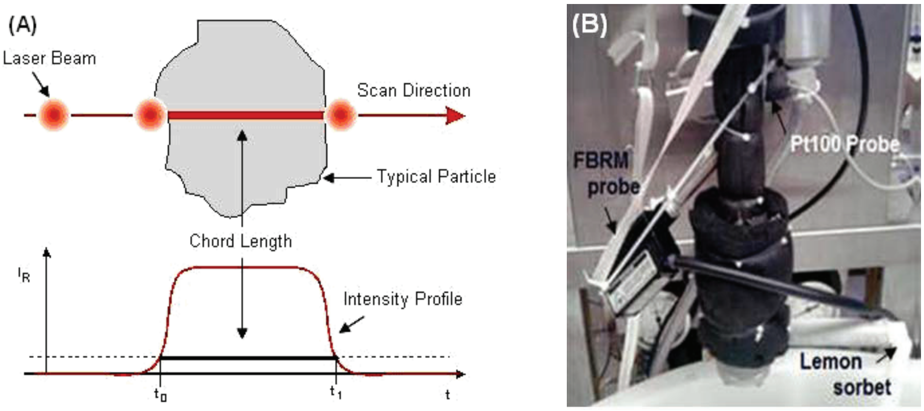


Fig. 1. (A) Measurement principle of a particle's chord length by the FBRM probe (Mettler-Toledo); (B) FBRM probe inserted at the outlet pipe of the freezer

2.4. Draw temperature measurements

The draw temperature of the product was measured online by means of a calibrated Pt100 probe (Baumer®, accuracy of 0.1°C). The Pt100 probe was inserted into the outlet pipe of the freezer before the product's exit (cf. Fig. 1B). In order to establish the relationship between the draw temperature of sorbet and its equilibrium ice mass fraction, the sorbet's equilibrium freezing point curve was previously determined in our laboratory using differential scanning calorimetric (DSC) measurements.

3. Results and Discussion

Table 2 shows the real operating conditions under which the FBRM measurements were taken and the mean values of the obtained responses. Each condition was performed twice and 5 times for run 10. Results in table 2 show that the use of the FBRM sensor makes it possible to monitor online the development of the ice crystals in sorbets containing up to 40% of ice, which was one of the objectives of this research.

Table 2. Real freezing conditions during measurements and obtained responses [a]

Run	***Coded values***			***Factors***			***Responses***		
	MFR	TR22	DRS	MFR $kg.s^{-1}$	TR22 °C	DRS $rad.s^{-1}$	MCL µm	DT ºC	IMF %
1	1	1	1	0.018	-18.04	94.25	6.04	-5.26	33
2	1	1	-1	0.018	-18.01	62.83	6.25	-5.34	32
3	1	-1	1	0.018	-12.55	94.25	7.32	-3.89	21
4	1	-1	-1	0.018	-12.48	62.83	7.31	-3.85	20
5	-1	1	1	0.01	-18.07	94.25	5.93	-6.26	37
6	-1	1	-1	0.01	-18.08	62.83	6.08	-6.67	39
7	-1	-1	1	0.01	-12.53	94.25	7.09	-4.73	28
8	-1	-1	-1	0.01	-12.49	62.83	7.51	-4.83	29
9	-α	0	0	0.007	-15.31	78.54	6.31	-6.02	36
10	0	0	0	0.014	-15.31	78.54	6.47	-4.89	29
11	+α	0	0	0.021	-15.42	78.54	6.53	-4.15	23
12	0	0	-α	0.014	-15.24	57.07	6.40	-4.93	30
13	0	+α	0	0.014	-19.83	78.54	6.00	-6.27	37
14	0	-α	0	0.014	-10.55	78.54	8.10	-3.58	17
15	0	0	+α	0.014	-15.35	104.72	6.32	-4.86	29

[a] MFR = Mix flow rate; TR22 = Evaporation temperature of R22; DRS = Dasher rotational speed; MCL = Mean chord length; DT = Draw temperature; IMF = Ice mass fraction.

3.1. ANOVA analysis of the responses

The global ANOVA analysis in table 3 shows for the mean chord length response, a significant model regression ($P < 0.0001$) with a value of $R^2 = 0.94$, a variation coefficient CV = 2.36% and does not show lack of fit ($P = 0.62$). The draw temperature of sorbet showed also a significant regression model ($R^2 = 0.99$, CV = 2.24%, $P < 0.0001$) and did not show lack-of-fit ($P = 0.83$). Therefore, both models can be used to predict the experimental behaviour of mean chord length and draw temperature responses, respectively.

Table 3. Global Analysis of Variance for Responses of MCL and DT [a]

Response	R^2 adjusted	CV (%)	F Value	P-value (model)	Lack-of-Fit
MCL	0.94	2.36	54.64	<0.0001*	0.62
DT	0.99	2.05	264.61	<0.0001*	0.83

[a] MCL = Mean Chord Length; DT = Draw temperature; CV = Coefficient of variation.

According to the regression coefficient analysis shown in table 4, it appears that the mean chord length was significantly affected at a 95% confidence interval by the evaporation temperature in its linear and quadratic terms ($P < 0.0001$ for β_2 and $P < 0.0001$ for β_{22}), followed by the dasher speed in its linear term ($P = 0.034$ for β_3). Whilst the mix flow rate did not show a significant effect. In the case of the draw temperature, the regression coefficient analysis in table 4 shows that the mix flow rate and the evaporation temperature of the refrigerant fluid had the most significant effect at a 95% confidence interval, for both their linear and quadratic terms ($P < 0.0001$ for β_1, $P < 0.0001$ for β_2, $P = 0.0002$ for β_{11} and $P = 0.0336$ for β_{22}) as well as their interaction ($P < 0.0284$ for β_{12}). The dasher speed also had a significant effect on the draw temperature for its linear term ($P = 0.0014$ for β_3) and for its interaction with the mix flow rate ($P = 0.0391$ for β_{13}).

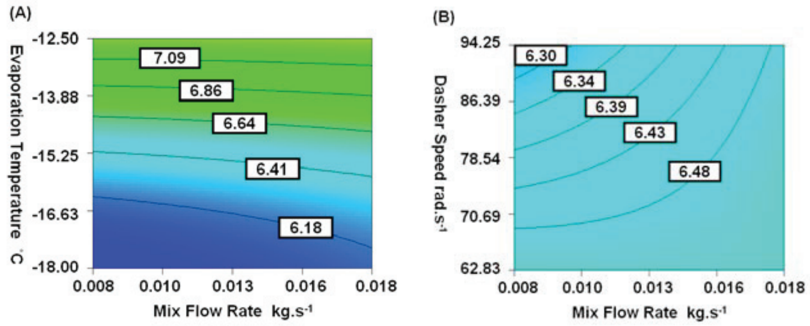
Table 4. Regression coefficients of the experimental behavior model and significance levels at 95% (P-values) for responses of MCL and DT[a].

	Interception	Linear			Interaction			Quadratic		
Response	β_0	β_1	β_2	β_3	β_{12}	β_{13}	β_{23}	β_{11}	β_{22}	β_{33}
MCL	17.896	-95.944	1.133	-9.05E-3	-2.714	0.731	-4.89E-5	334.98	0.028	-3.73E-5
P-value	<0.0001*	0.1636	<0.0001*	0.034*	0.4307	0.2329	0.9571	0.8658	<0.0001*	0.8157
DT	-4.863	271.36	0.248	0.029	-5.195	-0.856	-1.16E-3	-5597.52	-6.55E-3	-1.95E-4
P-value	<0.0001*	<0.0001*	<0.0001*	0.0014*	0.0284*	0.0391*	0.0626	0.0002*	0.0336*	0.0726

[a] MCL = Mean Chord Length; DT = Draw temperature; Coefficients (β) subindex : 1 = Mix flow rate; 2 = Evaporation temperature R22; 3 = Dasher rotational speed; * = significant influence at 95% confidence interval.

3.2. Effect of freezing conditions on the mean chord length

Fig. 2 shows the contour plots of the mean ice crystal chord length behaviour as a function of the evaporation temperature, mix flow rate and dasher speed. As we can see in table 4 (MCL β_2 and β_{22}) and Fig. 2 (A, C), the significant influence of the TR22 is directly proportional to the MCL, in other words, the mean ice crystal chord length becomes smaller when the evaporation temperature of the refrigerant fluid decreases (cf. table 2, runs 10, 13 and 14). This effect can be explained by the well known fact that at low refrigerant fluid temperatures the sorbet is exposed to a larger level of supercooling, which enhances a rapid heat removal rate and thus the ice nucleation mechanism. Similarly, Koxholt *et al.* [4] reported that low refrigerant fluid temperatures led to smaller values of ice CSD.



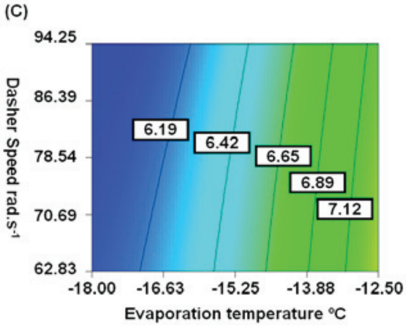


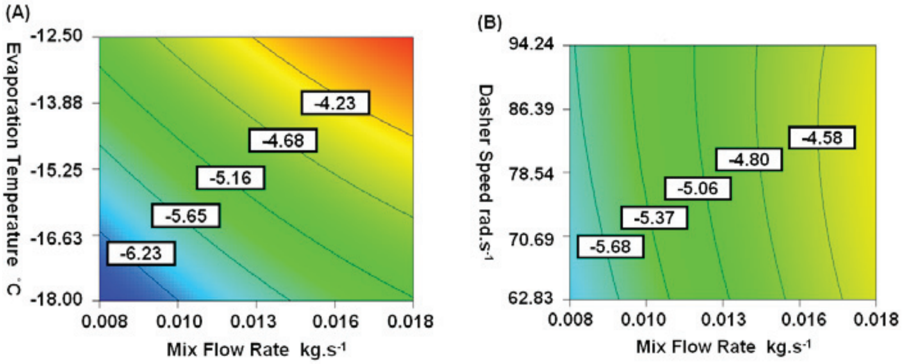
Fig. 2. Influence of operating conditions on the mean chord length of sorbet (in µm). (A) Dasher speed set at 78.54 rad.s^{-1}. (B) Evaporation temperature set at -15.31°C. (C) Mix flow rate set at 0.014 kg.s^{-1}

We can observe as well in table 4 (MCL β_3) and Fig. 2 (B, C), that the relationship between the dasher speed and the MCL is inversely proportional (cf. table 2, runs 10, 12 and 15). Thus, an increase of the scraping action of the dasher slightly decreases the ice crystal chord length. We believe that this influence is due to the higher rate of shear that is produced within the freezer, which may lead to a phenomenon of attrition of the bigger ice crystals, which produces new smaller ice nuclei by secondary nucleation.

It is generally thought that high mix flow rates (short residence times) produce smaller ice crystals [1, 4]. However, as we can see in our results (table 4, MCL β_1; and Fig. 2, A and B) the mix flow rate did not show a significant effect on the ice crystal chord length. Consequently, it is our opinion that this result is due to a compensatory effect that is produced within the freezer: when low MFR are used, the draw temperature of sorbet decreases (cf. table 4, runs 9, 10 and 11), which enhances the growth of ice crystals and increases the MCL. However, at the same time, when the sorbet's draw temperature is low, the ice mass fraction increases, as well as the viscosity of the product, producing a higher shear rate which leads to the attrition of the bigger ice crystals and generates new smaller ice nuclei by secondary nucleation.

3.3. Effect of freezing conditions on the draw temperature

Fig. 3 shows the contour plots of the draw temperature behaviour as a function of the evaporation temperature, mix flow rate and dasher speed. As we can see in Fig. 3, the relationship between the MFR and the draw temperature is directly proportional (cf. table 4, DT β_1). Thus, at a given refrigerant's fluid temperature, when lower MFR rates are used, the draw temperature of sorbet decreases.



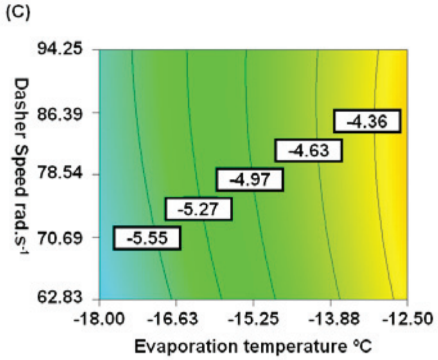


Fig. 3. Influence of operating conditions on the draw temperature of sorbet (in ºC). (A) Dasher speed set at 78.54 rad.s^{-1}. (B) Evaporation temperature set at -15.31°C. (C) Mix flow rate set at 0.014 kg.s^{-1}

We believe that this effect is due to the fact that at a lower MFR, the residence time is higher, and the sorbet is longer in contact with the freezer wall, so that more heat is extracted, decreasing the draw temperature and thus increasing the ice mass fraction (cf. table 2, points 9, 10 and 11). Likewise, Ben Lakhdar *et al*. [5] reported in the case of freezing of 30% sucrose/water solutions that high product flow rates lead to smaller ice mass fraction when the refrigerant fluid temperature was held constant.

We can observe as well (cf. table 4, DT β_3; and Fig. 3 B and C) that the relationship between the draw temperature and the dasher speed is directly proportional. Thus, an increase of the scraping action of the dasher slightly increases the draw temperature of sorbet (cf. table 2, runs 10, 12 and 15). We believe that this effect is due to the frictional energy originated between the blade tip and the freezer wall, which is dissipated to a certain amount into the product and slightly increases its temperature. Russell *et al*. [1] reported a warming of the ice cream, caused by an increase of the dasher speed, at a constant refrigerant fluid temperature.

4. Conclusions

Our research has shown that the FBRM technique is a convenient tool that allowed us to follow online the evolution of the ice crystal chord length in sorbets containing up to 40% of ice content. Our results demonstrated that the use of low evaporation temperatures makes it possible to obtain smaller ice crystals and to decrease the product’s temperature due to the higher level of supercooling applied to the product. An increase of the dasher speed slightly decreases the ice crystal chord length, due to the higher shear of the product which leads to the attrition of the ice crystals, and produces new smaller ice nuclei by secondary nucleation. High dasher speeds slightly increase the draw temperature, due to the frictional energy dissipated into the product. Low mix flow rates (long residence times) result in lower draw temperatures due to the fact that the product remains longer time in contact with the freezer wall, removing more heat from the product.

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