Effect of Freezing Point and Texture Regulating Parameters on the Initial Ice Crystal Growth in Ice Cream

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ABSTRACT

The objective of this research was to derive information on the technological influences on the ice recrystallization in ice cream after the freezer outlet during the sensitive temperature range from the freezing point to –8°C. The parameters studied were the total solids content and sucrose concentration, which determine the freezing point of the ice cream mix as well as the overrun, mix viscosity, and the fat content, which influence the texture of the ice cream. For the recrystallization experiments, the ice cream samples, taken directly after the continuous ice cream freezer, were treated at defined temperature time combinations. In the temperature range investigated, none of the parameters had a significant effect on the ice recrystallization. The contradictory results compared with previous research can be explained by the different experimental conditions and can reemphasize the importance of the hardening conditions as an influencing factor for the ice crystal sizes in ice cream. (**Key words**: recrystallization, freezing point, texture, ice cream)

Abbreviation key: $\mathbf{D_{50.3}}$ = median value of the volume-based ice crystal size distribution, **PEG** = polyethylene glycol.

INTRODUCTION

One of the main parameters that determines the mouthfeel of ice cream is the size distribution of the ice crystals. In an ice cream freezer, tiny ice crystals are formed that are much smaller than the crystal sizes measured at the freezer outlet. After a few minutes of storage at temperatures over –10°C, these ice crystals grow in size because of recrystallization processes (22). In the worst possible case, the ice crystals grow to such a size that the consumer can feel them as rough particles on the tongue (3). Several investigations (10, 16) have shown that samples containing crystals with a mean diameter of over 50 μm are perceived as rough.

It is generally accepted that the main factor influencing recrystallization and ice crystal growth is temperature. Based on an Arrhenius kinetics, Trgo (22) determined an activation energy of 225 kJ/mol for the recrystallization process of ice crystals in ice cream. This high activation energy indicates the high temperature dependency of the process; the higher the temperature, the faster the ice crystals grow. Temperature fluctuations, so-called heat shocks, especially promote recrystallization processes during the storage of the ice cream (9).

Many authors (1, 3, 11, 18) have suggested that the addition of stabilizers results in a slower ice crystal growth because of a lower water mobility in the unfrozen solution, which is caused by the higher viscosity. Some research groups (7, 8, 13), however, concluded that the stabilizers only influence the organoleptical perception of the ice crystals. Min (16), Hagiwara and Hartel (12), and Sutton and Wilcox (21) found that the effect of stabilizers on ice crystal growth and recrystallization was significant only for certain stabilizers and ice creams and under specific conditions but that there was no direct correlation between the mix viscosity and the crystallization rate.

Sweetners, depending on type and concentration, affect the freezing point of the ice cream mix, the amount of frozen water at a certain temperature, and the glass transition temperature. A direct correlation between the freezing point and the crystal growth rate was observed by Harper and Shoemaker (13) and by Hagiwara and Hartel (12). Slade and Levine (19) determined a relationship between the recrystallization rate and the difference of the glass transition temperature and the storage temperature. However, Hagiwara and Hartel (12) were not able to ascertain the existence of a general relationship for all experimental conditions and different sweeteners.

Arbuckle (1) suggested that the fat content can also influence the size of the ice crystals. Fat globules could mechanically impede the ice crystal growth.

Ice cream derives its typical creamy consistency from incorporation of air. Air bubbles are finely dispersed and have diameters ranging from 5 to 300 μm

Received July 7, 1998.
Accepted November 16, 1998.

(2). Although Arbuckle (1) and Windhab and Bolliger (23) suggested that the overrun influences the ice crystal size in that higher overrun leads to smaller crystals, Buyong and Fennema (8) did not believe that the air affected the ice crystallization.

Therefore, the objective of this research was to study the influence of the freezing point of the ice cream mix on the initial ice crystal growth as affected by the TS content and the amount of sucrose. Furthermore, the effect of the texturizing parameters such as fat content, mix viscosity, and overrun was investigated.

MATERIALS AND METHODS

Composition and Process

The standard ice cream mix formulation consisted of 10% milk fat, 11% nonfat milk solids, 10% sucrose, and 5% glucose syrup and demineralized water. No hydrocolloids or emulsifiers were added to avoid masking effects. All dry ingredients were mixed with demineralized water, and the water content of the fluid ingredients was taken into account. To investigate the influences of the certain parameters, the standard ice cream mix was varied. The formulations and freezing points of the different mixes are shown in Table 1.

After pasteurizing (85°C, 30 s) and homogenizing (170/40 bar, 70°C) the mix, ice cream was produced in a continuous ice cream freezer (Gelmark 80; Tetra Laval Hoyer, Milan, Italy). The overrun was 80%, and the drawing temperature was –6°C.

Recrystallization Experiment

The samples were collected directly from the freezer into screwable stainless steel tubes ($\varnothing$ = 12.7 mm) with a volume of 12 ml. Then the samples were treated in the first cold bath with defined temperature time combinations as designated in the legends of figures. For all of the different ice creams, the recrystallization temperature was reached within 0.5 min. Subsequently, the sample structure was fixed in the second cold bath at –25°C within 6 min (22). The samples were stored overnight in a freezing cabinet at –20°C, after which, the ice crystal analysis was performed.

Ice Crystal Analysis

The ice crystals were examined under a microscope (Axiovert 135; Zeiss, Oberkuchen, Germany) in a freezing chamber at –16°C. For the microscopy, the sample preparation was made with ethyl acetate as described by Donhowe et al. (10). For each temperature-time combination, two preparations were made. Two photomicrographs were taken from each preparation with a 100× magnification prior to photographic enlargement. Consequently, between 300 and 2000 crystals were analyzed per sample. The photomicrographs were evaluated by manual image analysis with a digitizing tablet (Podscat PT 3039; Podworld, Taiwan) and the Profi Partikel Manager software (version 3.0, 1993, Arndt & Baumgartl, Offenbach, Germany). Based on the assumption of a spherical shape, the software calculated a volume-based ice crystal size distribution. The size measurements were calibrated against photomicrographs of a Thoma counting chamber (Brand, Wertheim, Germany). All distributions were normal distributions (22). Therefore, the median value of the volume-based ice crystal size distribution ($\mathbf{D_{50.3}}$) of the ice crystal size distributions was used for the further evaluation of the experiments. The deviation of the median value ($D_{50.3}$) was ±3.5 μm when measured five times.

RESULTS AND DISCUSSION

Effect of the Total Solids Content on the Ice Crystal Growth

The variation of the TS affects several parameters of the ice cream simultaneously. A reduction of the dry matter results in an increase in the freezing temperature (40.8%: –3.0°C; 35%: –2.2°C; and 29%: –1.5°C) and, consequently, results in a change in the temperature behavior during freezing and hardening. Furthermore, a higher water content leads to a longer hardening time as more heat of fusion has to be eliminated. Additionally, the freezer outlet temperature is higher.

In Figure 1, the median values of the ice crystal size distributions ($D_{50.3}$) of ice creams with differing TS contents were determined during storage at –5°C. For all TS contents, the ice crystals grew over time. Although the freezing point of the samples with 29.2% TS and 40.8% TS varied by 1.5°C, and a 20% difference in the ice content occurred at analysis temperature –16°C, the ice crystals showed the same growth behavior. The amount of TS had no influence on the recrystallization of the ice crystals. However, Donhowe et al. (10) observed differences in the ice crystal size distributions for ice creams with different TS contents. The ice creams with a higher amount of TS had smaller ice crystals. The main difference between the experiments of Donhowe et al. (10) and

TABLE 1. Formulations, freezing points, drawing temperatures, and ice contents of the different ice cream mixes.

	Mix							
	0 (Standard)	1	2	3	4	5	6	7
Varied parameter	. . .	TS	TS	Sucrose	Sucrose	Viscosity	Fat	Fat
TS, %	35	29.2	40.8	45	49.2	40	30	25
Nonfat milk solids, %	11	9.18	12.82	11	9.18	11	11	11
Fat, %	10	8.34	11.66	10	8.34	10	5	0
Sucrose, %	10	8.34	11.66	20	28.34	10	10	10
Glucose syrup solids, %	4	3.34	4.66	4	3.34	4	4	4
Polyethylene glycol, %	. . .	. . .	. . .	. . .	. . .	5	. . .	. . .
Freezing point, °C	–2.2	–1.5	–3.0	–3.2	–4.8	Not measured	Not measured	Not measured
Drawing temperature, °C	–6	–5.3	–6.7	–9	–10	–6	–6	–6
Ice content at –16°C (analysis temperature)	0.44	0.54	0.35	Not measured	Not measured	Not measured	Not measured	Not measured

this work was found in the different temperature behavior. Donhowe et al. (10) filled the ice cream in 1.9-L packages and hardened the ice cream in cold air. As the freezer outlet temperature declined, the concentration of TS increased, and the temperature behavior of the individual ice creams might have been quite different, especially because of the slow hardening process in air.

During this study, the samples reached the same core temperature (–25°C) very rapidly (≤ 6 min) because of the small amount of sample material and the high heat transfer in the cold bath (22). When the ice cream samples were filled into 250-ml plastic cups, hardened in a deep freezer at –40°C, and tempered to –5°C for recrystallization studies, the results of Donhowe et al. (9) were confirmed. Thus, a higher amount of TS would lead to smaller ice crystals under industrial conditions because the ice cream is exposed to critical temperatures with high recrystallization rates for shorter periods.

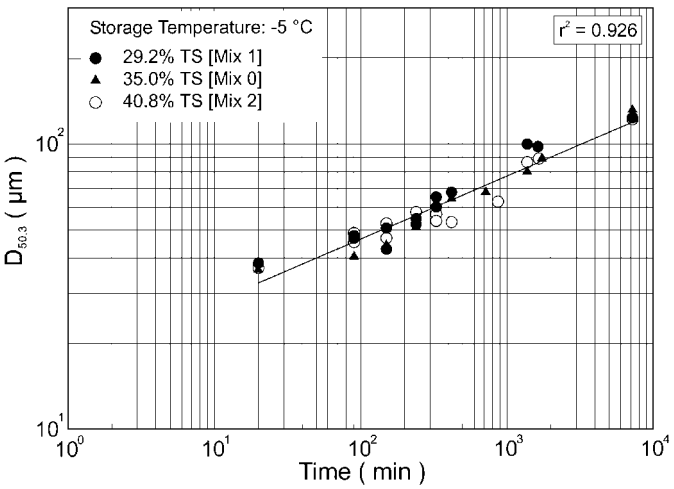

Figure 1. Figure 1. Median values of the ice crystal size distributions ($D_{50.3}$) at –5°C as a function of time after the freezer outlet for ice creams with different contents of total solids. The linear regression line was calculated from the data of a single batch of each of the three mixes.

From these results, it also can be concluded that the ice phase volume had no influence on the ice crystal sizes. The estimated difference for a median value $D_{50.3}$ = 20 μm based only on the 20% difference in the amount of ice would be in the range of 1 μm, which is within the deviation of the median value. An effect of the amount of ice frozen on the recrystallization rate as stated by Sutton et al. (20) must not be taken into account because of the short hardening time used.

Effect of the Sucrose Concentration on the Ice Crystal Growth

When the sucrose content was increased from 10 to 20% by substituting 10% water with sucrose, a 1°C-difference in the freezing point was observed (10% sucrose: –2.2°C; and 20% sucrose: –3.2°C), and the freezer outlet temperature decreased from –6° to –9°C. When the median values ($D_{50.3}$) of the two ice creams are compared (Figure 2), it becomes evident that there is no difference in the ice crystal growth or recrystallization.

To realize a more extreme difference in the freezing point, 20% sucrose was added to the ice cream mix with 29.2% TS. The freezing point difference was 3.3°C (29.2% TS: –1.5°C; and 49.2% TS: –4.8°C). These two mixes are the extremes of what is techno-

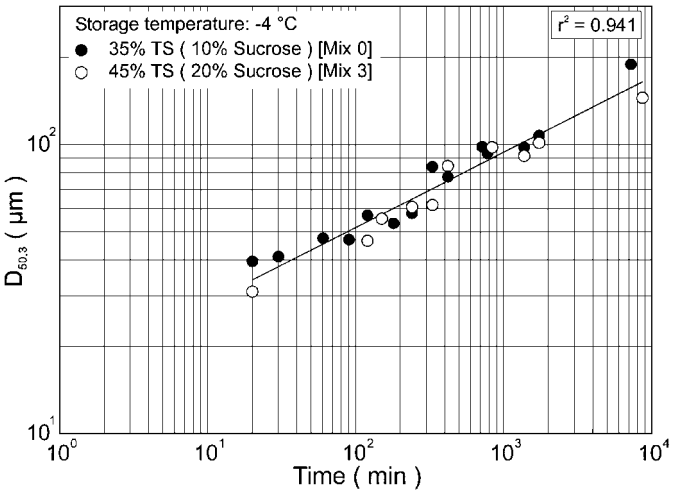

Figure 2. Median values of the ice crystal size distributions ($D_{50.3}$) at –4°C as a function of time after the freezer outlet for ice creams with different sucrose concentrations and a difference in the freezing point of 1°C. The linear regression line was calculated from the data of a single batch of each of the two mixes.

logically possible. Ice cream could not be produced under standard freezer conditions at a higher freezing point of the ice cream mix than –1.5°C because the amount of water that had to be frozen would have been too high. A freezing point lower than –4.8°C would have lead to an unstable and almost fluid ice cream. Figure 3 indicates that despite this extreme difference in the freezing point, it had no effect on the ice crystal growth. As recrystallization is mainly dependent on the migration of the water molecules from the surface of the ice crystals into the unfrozen solution, the above phenomena can be explained by the composition of this concentrated solution. The concentration of substances such as sucrose in the unfrozen solution is temperature dependent. If the starting concentration is higher, there is a smaller amount of frozen water at the same temperature. However, the end concentration is not dependent on the starting concentration, which means that only the storage temperature affects the concentration of the unfrozen solution to which the ice crystals are exposed.

Hagiwara and Hartel (12) determined a linear relationship between the freezing point temperature and the recrystallization rate. Additionally, they ascertained a linear relationship between the percentage of frozen water and the recrystallization rate. The different results might have been due to the different process technology and different ingredients. Hagiwara and Hartel (12) produced the ice cream in a batch freezer and stopped the freezing process when the ice cream had reached a certain consistency. Because they used different sweeteners to reach different freezing points, the ice cream was kept in the freezer for differing periods before hardening and storage. Furthermore, the hardening process in their research was performed in a walk-in freezer, which resulted in different hardening times.

Effect of Overrun, Viscosity, and Fat Content on the Ice Crystal Growth

In the following experiments, those parameters were to be investigated which did not have a direct relationship with water but which did influence the texture of the final product.

Overrun. Figure 4 shows that the overrun had no effect on the recrystallization. The ice crystals in a foam structure grow as fast as in a mix without air, which can be explained by the space between the air bubbles being much larger than the ice crystals. Therefore, there is no other driving force for the crystallization processes.

Viscosity. To investigate the influence of the viscosity of the ice cream mix on the recrystallization, 5% water from the standard formulation was substituted by polyethylene glycol (**PEG**). The PEG is an electrically uncharged macromolecule, which is inert to the other ingredients (14). The viscosity-increasing effect can be described as a fluid containing inert spheres. The addition of the 5% PEG caused a ninefold increase in mix viscosity (standard: 0.009

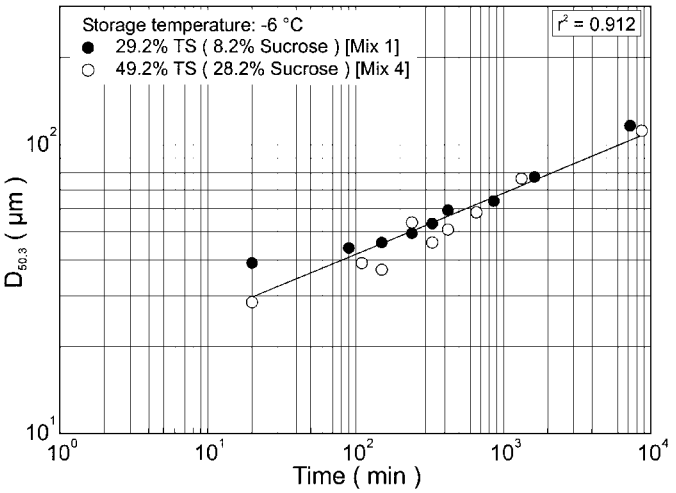

Figure 3. Median values of the ice crystal size distributions ($D_{50.3}$) at –6°C as a function of time after the freezer outlet for ice creams with different sucrose concentrations and a difference in the freezing point of 3.3°C. The linear regression line was calculated from the data of a single batch of each of the two mixes.

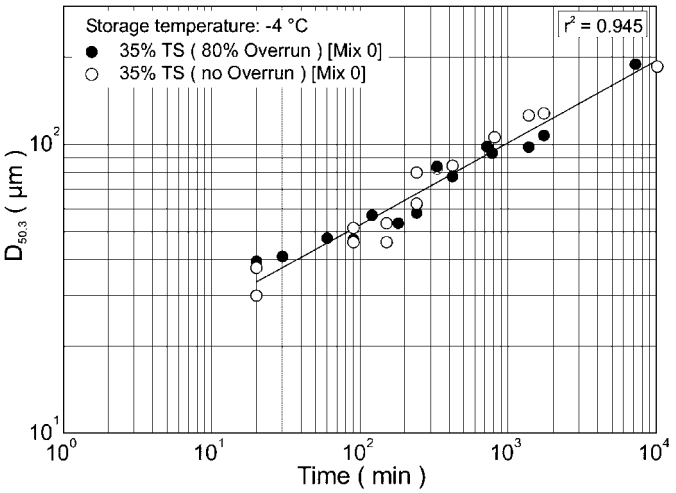

Figure 4. Median values of the ice crystal size distributions ($D_{50.3}$) at –4°C as a function of time after the freezer outlet for ice creams with different overrun values. The linear regression line was calculated from the data of a single batch of each of the two mixes.

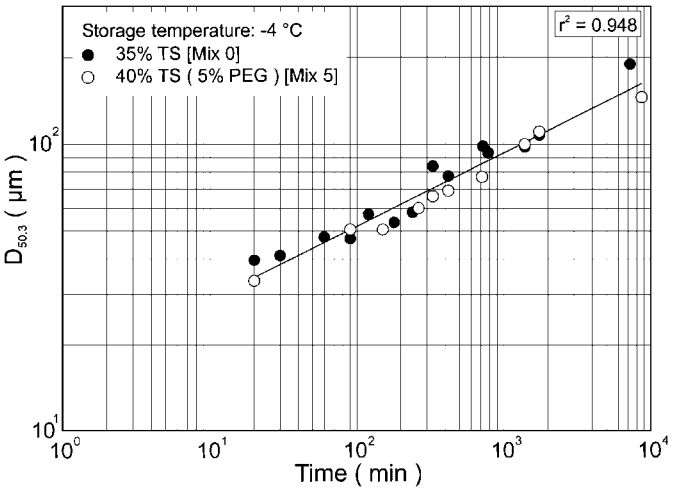

Figure 5. Median values of the ice crystal size distributions ($D_{50.3}$) at –4°C as a function of time after the freezer outlet for ice creams with different viscosities. The addition of polyethylene glycol (PEG) caused an 9-fold mix viscosity increase. The linear regression line was calculated from the data of a single batch of each of the two mixes.

Pa s; PEG: 0.097 Pa s). The ice cream exhibited a dry appearance, a high cohesiveness, and creaminess.

In Figure 5, the ice crystal growth in a standard ice cream and in an ice cream with 5% PEG is compared. It is evident that the viscosity did not influence the recrystallization rate or the ice crystal sizes, which confirms earlier results (22) in which an activation energy of 225 kJ/mol was determined for the recrystallization of ice crystals in ice cream. The high activation energy indicated that recrystallization cannot be a diffusion limited process that can be influenced by higher viscosities. Several authors (4, 5, 7, 8, 16, 17) also state that the retarding effect of some hydrocolloids on the ice recrystallization is not caused by higher viscosity. An approach on the structure of crystallization retarders might be the so-called antifreeze proteins, which stop or retard crystallization even in very small amounts (15).

Fat content. Figure 6 shows the effect of the presence and the amount of fat on the ice crystal growth in ice cream. Because of the unstable foam structure, the ice cream with 0% fat was produced without overrun. Although there was a dramatic difference in the sensorial parameters of texture, cooling effect, and iciness, there was no difference in the ice crystal sizes or the ice crystal growth. The different results as compared with Arbuckle (1) may relate to different temperature ranges used in the two studies. At lower temperatures, the volume fraction of the fat in the unfrozen solution and the possible stearic influence is higher. Furthermore, no emulsifiers were used in our study. The fat structure might have been more stable if emulsifiers were added.

CONCLUSIONS

Although the variation of the total solids content or the sucrose concentration caused significant differences in the freezing point and the amount of frozen water, there was no direct effect on the ice crystal

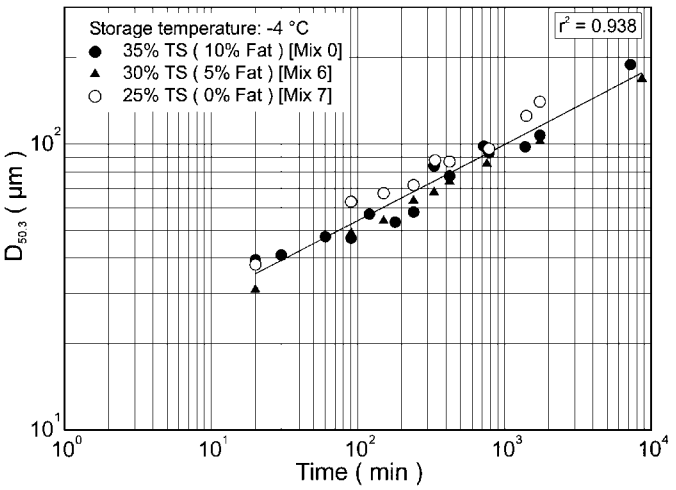

Figure 6. Median values of the ice crystal size distributions ($D_{50.3}$) at –4°C as a function of time after the freezer outlet for ice creams with different fat contents. The linear regression line was calculated from the data of a single batch of each of the three mixes.

sizes or the recrystallization rate under the conditions used in this study. From the contradictory results of other authors, it can be concluded that it is necessary to take basic process parameters into account such as the duration of time that the ice cream in the freezer and the temperature gradient in the freezer. For the production of ice cream, it is important to note that lower freezing points result in lower outlet temperatures from the freezer and a faster hardening behavior under constant freezer parameters. Thus, in practice, ice creams with a lower freezing point contain smaller ice crystals. Furthermore, the results reemphasize the need for rapid transfer of extruded ice creams to hardening temperatures.

The addition of PEG, which influences the viscosity of the mix and the unfrozen solution, did not affect the ice crystal growth, which confirms that the recrystallization is not a diffusion limited process, and, therefore, the retarding effect on the crystal growth of some stabilizers cannot be based on the higher viscosity.

A relationship between the fat foam structure and recrystallization was not observed. Neither the highly viscous foam lamellas nor the presence of air or fat globules and clusters had an effect on the recrystallization rate.

Future research should determine whether and to what extent ingredients that affect the ice and solution interface have a stabilizing influence on the product. Furthermore, investigations should be carried out to ascertain how water-binding substances, especially different protein structures like denatured and particulate whey proteins, affect the growth of ice crystals.

ACKNOWLEDGMENTS

This research project was supported by the FEI (Forschungskreis der Ernährungsindustrie e.V., Bonn, Germany), the AiF, and the Ministry of Economics. Project No.: AiF-FV 11201 N.

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