

У виробництві морозива дисперсність кристалів льоду – важливий органолептичний показник – залежить від числа центрів кристалізації води на першій стадії заморожування (нуклеації). При подальшому заморожуванні вода, що залишилася, кристалізується на вже існуючих центрах. Наведено результати досліджень по обгрунтуванню інтенсифікації нуклеації шляхом збільшення швидкості заморожування в азоті та використання ефекту зародкоутворення, обумовленого присутністю частинок. Ініціаторами нуклеації розглянуті суспендовані частинки жирової фази та частково розчинного стабілізатора (мікрокристалічна целюлоза) та коагульованого білка. Встановлено, що найбільша дисперсність кристалів льоду досягнута при збільшенні швидкості заморожування при використанні азоту. При частці вимороженої води 40–50 % при імерсійному заморожуванні та наступному повітряному доморожуванні розмір кристалів льоду через 6 міс. зберігання не перевищував величину 37 мкм. Показано, що додатковим фактором ініціювання нуклеації при імерсійному та безконтактному заморожуванні у фрізері є частки жиру.

Виявлено позитивний вплив суспендованих частинок мікрокристалічної целюлози та коагульованого білка на дисперсність кристалів льоду в процесі виробництва морозива та зберігання протягом 6 міс. Середній діаметр кристалів льоду в процесі зберігання при використанні мікрокристалічної целюлози в вершковому морозиві склав 39 мкм, в кисло-молочному морозиві з використанням йогурту – 32–34 мкм.

Результати досліджень дозволяють визначити нові напрямки інтенсифікації нуклеації, що базуються на принципах підвищення швидкості заморожування та інтенсифікації нуклеації із застосуванням додаткових центрів кристалізації

Ключові слова: інтенсифікація нуклеації, заморожування, дисперсність кристалів льоду, рідкий азот, мікрокристалічна целюлоза, коагульований білок

NUCLEATION INTENSIFICATION IN THE ICE CREAM PRODUCTION

A. Tvorogova

Doctor of Technical Sciences*

E-mail: antvorogova@yandex.ru

T. Shobanova

Postgraduate Student, Junior Researcher*

E-mail: t.shobanova@yandex.ru

A. Landikhovskaya

Junior Researcher*

E-mail: anna.landih@yandex.ru

P. Sitnikova

PhD, Researcher*

E-mail: sitnikova.p.b@gmail.com

I. Gurskiy

Research Engineer*

E-mail: iixrug@yandex.ru

*All-Russian Scientific Research Institute of Refrigeration Industry – Branch of V. M. Gorbатов Federal Research Center for Food Systems of Russian Academy of Science Kostyakova str., 12, Moscow, Russia, 127422

Received date 27.01.2020

Accepted date 04.03.2020

Published date 24.04.2020

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1. Introduction

The name and definition of “ice cream” worldwide include not only the requirements for the origin and mass proportion of fat used in the production but also describe it as a “whipped” and “frozen” product. Given that ice cream is consumed frozen, it is important when freezing to reach a certain size of ice crystals and keep them dispersed during further storage. In this regard, in the first stage of freezing (the origin of the nuclei), it is necessary to obtain as many crystallization centers as possible. Ice formation typically begins when the product is cooled to the initial freezing point (cryoscopic temperature) or slightly below this point. The second phase of crystallization is the growth of crystals nuclei, followed by the construction of crystal blocks [1, 2].

The freezing process in the production of ice cream consists of 3 consecutive stages. The first stage is partial freezing of a specially prepared mixture during the freezing process to a temperature of minus 4–7 °C (soft ice cream is obtained). In the second stage, there is

freezing (hardening) of soft ice cream to a temperature of minus 12–18 °C in the freezer. The third stage involves post-freezing in the freezer to a temperature not higher than minus 18 °C, if this temperature has not been reached during the ice cream hardening process [2]. Ice cream is considered a finished product when the temperature reached in the production process is –18 °C. Freezing creates a dense consistency and structure characteristic of ice cream. A stable structure of ice cream is achieved if the main structural components of ice cream – fat, ice crystals, and air bubbles – have optimal dimensions, which in the process of storage do not change. In particular, ice crystals are not organoleptically felt if their size does not exceed 50 μm [3, 4].

In order to obtain and preserve highly disperse ice crystals, it is necessary to control the rate of germ formation in the first stage of freezing, as new crystallization centers are not formed in the later stages. To produce a large number of small crystals, freezing conditions should contribute to the formation of germs and minimize the growth of ice crystals.

Thus, it is obvious that some of the components used in the production of ice cream can act as initiators of nucleation, which makes it possible to manage this process when dealing with technological issues in cases of purposeful change in formulations.

The issue of initiating nucleation in the production of ice cream has not been sufficiently investigated, thus rendering relevance to studying the impact of the nucleation process on the dispersal of ice crystals during storage and to searching for new solutions for its intensification.

2. Literature review and problem statement

Factors influencing the decline in the quality of frozen foods are ice crystallization and recrystallization. Therefore, modern research is aimed at achieving uniform germ formation and crystallization of small ice crystals by improving known and creating new processes. Paper [5] describes the directions of modern research on inhibiting the recrystallization through the use of special additives acting as anti-freeze proteins. This kind of research is relevant. However, the creation of a large number of primary crystallization centers (nucleation) is a top priority.

Work [6] reports the results of research on the effects exerted on the growth of ice crystals in ice cream by emulsifiers, hydrocolloids, and carbohydrates.

The size of ice crystals in ice cream is greatly affected by the freezing process. During this period, a large number of crystallization centers are formed as a result of the transfer by rotating blades of ice crystals from the wall of the freezer, where crystals are created, into the mass of the product. Freezing is the only process in the production of ice cream, during which crystallization centers are created. When hardening ice cream – the process that follows freezing, crystal formation of moisture is difficult because of the effect of hypothermia. Therefore, the new ice crystals do not form their own crystallization centers and are located at existing crystallization centers. The more crystallization centers were formed during the freezing process, the smaller the ice crystals in the ice cream will be [3, 7].

Thus, the nucleation intensification at the primary freezing stage of the ice cream mixture in the freezer is the most effective way to create a high-dispersed phase of ice. It can be assumed that the nucleation can be intensified by increasing the freezing rate at its initial stage with the achievement of optimal (at least 50 %) share of frozen water and when using particles – the initiators of nucleation.

High heat transmission rates and, therefore, high freezing speed contribute to the formation of small ice crystals. With slow freezing, water molecules have more time to migrate to growing nuclei, resulting in large crystals forming [8].

It is possible to increase the freezing rate at cryogenic freezing. In food production, the most used cryogenic substances are nitrogen and carbon dioxide. Cryogenic fluids have very low boiling points. The boiling points of liquid nitrogen and liquid carbon dioxide are minus 196 °C and minus 79 °C, respectively. Cryogens are colorless, odorless, and chemically inert. They give a very large temperature difference and a high rate of heat transmission [9].

Paper [10] reports the results of studies on the effects of cryogenic nitrogen freezing on the structure of certain foods.

However, the effect of liquid nitrogen on the dispersal of structural elements in the ice cream was not studied.

Compared to germ formation, the growth of crystals has been relatively well studied and controlled [11, 12]. However, papers [8, 12, 13] show that it is advisable to conduct research to identify factors of nucleation intensification in the production of ice cream in the first stage of freezing in technologically accessible ways.

3. The aim and objectives of the study

The aim of this study is to choose and justify the effectiveness of technologically accessible and effective techniques for initiating nucleation in the production of ice cream in the first stage of freezing.

To achieve the set aim, the following tasks have been solved:

- to investigate the effect exerted on the nucleation by immersion freezing in nitrogen;
- to investigate the effect exerted on the nucleation by the fat phase;
- to investigate the effect exerted on the nucleation by the suspended stabilizer particles;
- to investigate the effect exerted on the nucleation by the coagulated protein.

4. Materials and methods to study the factors of nucleation intensification in the production of ice cream

The objects of our research were ice cream sundae and mixtures for its production, milk ice cream, fermented milk ice cream with yogurt, yogurt ice cream, a synergistic composition with carboxymethyl cellulose sodium salt (CMS) and microcrystalline cellulose (MCC).

The scientific research was conducted on the basis of the All-Russian Research Institute of Refrigeration Industry, a branch of the Federal State Budgetary Research Institution of the Federal Scientific Center for Food Systems named after V. M. Gorbatov at the Russian Academy of Sciences (VNIKHI – a branch of the FGBNU “FNC of Food Systems named after V.M. Gorbatov” RAS) and employed modern methods:

- rotational viscometry (programmed viscometer DV-II+PRO Brookfield (USA) – to determine the dynamic viscosity of the mixture;
- microstructural (CX41RF microscope (Japan) with an integrated camera and program control) – to study the state of the fat phase and ice crystals of the product, assess the distribution of coagulated protein in ice cream;
- determining the whipping of ice cream based on GOST 31457-2012;
- determining the titratable acidity in mixtures for ice cream based on GOST 3624-92 “Milk and dairy products. Titrimetric methods for determining acidity.”

In the production of fermented milk ice cream, the mixture was fermented with the sourdough cultures *Str. thermophilus* and *Lactobacillus delbrueckii subsp. bulgaricus* at 40–43 °C for 4–6 hours.

The fermentation process was carried out taking into consideration the need to achieve a titratable acidity in the finished product of 70–90 °T.

5. Results of studying the influence of various factors on nucleation in the production of ice cream

5.1. Studying the effect exerted on nucleation by the immersion freezing in nitrogen

Given that the shape and size of ice crystals may depend on the freezing rate, in this process, we used liquid nitrogen whose boiling point at atmospheric pressure is minus 196 °C. This freezing technique is used in the industrial manufacture of granular ice cream and soft ice cream in the catering network.

The immersion freezing in nitrogen was used instead of freezing at the initial freezing stage (a period of intensive nucleation) to the temperature of the product minus 4–5 °C and a share of frozen-out water of 40–50 %. This condition is typical for soft ice cream sold in the catering network. To receive hardened ice cream, we used a technique standard for enterprises – freezing in low-temperature chambers at temperatures minus 18, 30, and 40 °C [13].

The chosen object to be frozen was a mixture for ice cream sundae – a variety of the product with the lowest moisture content and the greatest dynamic viscosity needed to preserve and distribute structural elements.

Whipping and freezing the mixture for ice cream sundae with liquid nitrogen was carried out as follows: we poured the ready mixture for dessert in the bowl of the mixer and turned on a stirrer. When the product reached the required volume, liquid nitrogen was introduced into the mixer bowl, with the mixer operating, from a pipeline connected to the Dewar vessel. The amount of nitrogen supplied was regulated by a special valve on the pipeline.

Experimentally, it has been established that this technique of making frozen dessert contributes to the formation of small ice crystals, which is confirmed by the microscopy of specially prepared preparations (Fig. 1).

The effect of rapid liquid nitrogen freezing on the dispersal of ice crystals was investigated in ice cream sundae mixtures with a mass fat content of 12 % (sample No. 1), 15 % (sample No. 2), and 20 % (sample No. 3). In such varieties, additional crystallization centers can be the numerous fat particles suspended when cooling and freezing. In this regard, the probability of obtaining high dispersal of ice crystals without the additional introduction of stabilizers is high.

After the first phase of nitrogen freezing, the hardening and subsequent storage of samples continued in the refrigeration chambers at temperatures –18, –30, –40 °C in order to determine the effect of temperature on the dispersal of structural elements after hardening and storing. In all samples obtained during the immersion freezing, the dispersal of ice crystals even after 6 months of storage was higher than their dispersal in the industrially produced ice cream sundae over 1 month of storage. The average size of ice crystals did not exceed 37 μm with their organoleptic feel of 50 μm.

It has been established that at temperatures of minus 30 °C and minus 40 °C the dispersal of ice crystals in the storage process hardly changed. At a temperature of minus 18 °C, changes in the dispersal of structural elements, even in ice cream with the lowest mass fat share of 12 %, were insignificant (Fig. 2, Table 1).

Our study has identified the positive effect of liquid nitrogen on the formation and dispersal of ice cream structural elements, making it possible to use this re-

frigerant at the initial freezing stage and to eliminate the process of freezing.

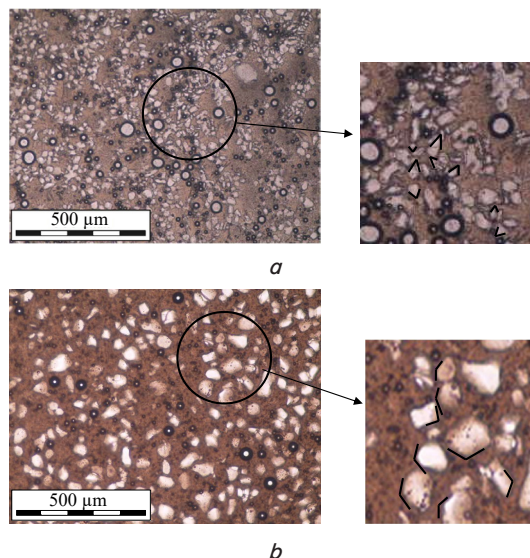


Fig. 1. Microphotographs of ice crystals in ice cream prepared at freezing: *a* – with liquid nitrogen; *b* – in a freezer

Table 1
Mathematical dependence of the distribution of ice crystals for size in ice cream sundae with a fat mass share of 12 %

Storage duration, month	Dependence	Approximation confidence coefficient, R^2
Ice cream sundae with a fat mass share of 12 %		
1	$y = -8E-07x^5 + 0.000x^4 - 0.006x^3 + 0.143x^2 - 0.555x$	0.978
3	$y = -6E-07x^5 + 9E-05x^4 - 0.005x^3 + 0.151x^2 - 0.923x$	0.981
6	$y = -6E-07x^5 + 8E-05x^4 - 0.005x^3 + 0.138x^2 - 0.828x$	0.984

Ice crystals in the samples obtained using liquid nitrogen are characterized by a greater presence of sharp angles than the crystals obtained in the standard way (at freezing and further hardening).

The effect of particles that are the nucleation initiators was considered using an example of the influence of fat particles, insoluble hydrocolloid, and coagulated protein.

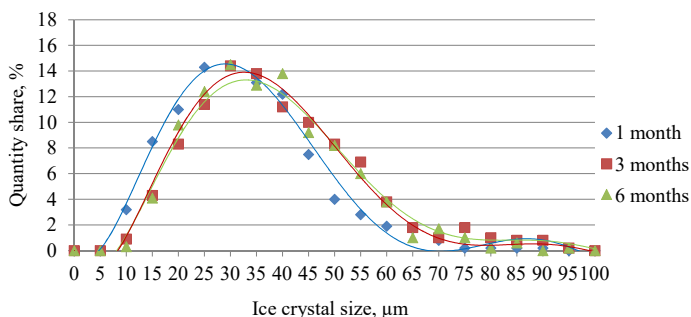


Fig. 2. Distribution of ice crystals for size in ice cream sundae with a fat mass share of 12 % during storage at –18 °C

5. 2. Studying the effect of fat particles on the process of nucleation in ice cream

The main number of fat particles in ice cream is characterized by a size not exceeding 1 μm. When the mixture is cooled, the fat transforms from liquid to solid state; it can be assumed that fat particulate matter is a phase of suspension. Therefore, they can act as particles that are the nucleation initiators. And the more of this kind of particles there are in the product, the more crystallization centers there will be. It is possible to increase the number of fat particles in ice cream by increasing homogenization pressure in the same variety or manufacture products with a high mass share of fat (at least 15 %). The effect of the fat phase on nucleation was investigated by conducting a comparative estimate of the distribution of ice crystals in ice cream with a mass share of fat of 15 % (sample 1) and 10 % (sample 2) with the same mass proportion of dry substances. To replenish dry substances in the product of 10 % fat, we used erythrite, a natural sweetener from the class of sugar spirits, which does not affect the viscosity of mixtures (Table 2).

Table 2

Ingredients of ice cream sundae and creamy ice cream

Indicators	Ice cream sundae	Creamy ice cream
Mass share of dry substances, %, not less, including:	39.38	39.18
milk fat	15.0	10.0
NDM	10.0	11.0
sucrose	14.0	14.0
dry substances of erythritol	–	3.8
dry substances of stabilizer	0.38	0.38

In the production of ice cream, depending on the mass share of fat, homogenization pressure is determined in order to obtain the main number of fat particles no larger than 1 μm. This size of fat particles was obtained in creamy ice cream and ice cream sundae. And, to eliminate the influence of different amounts of water on the crystallization process, the varieties of ice cream were made with the same mass proportion of moisture.

The distribution of ice crystals for size showed that the dispersal of crystals in ice cream sundae is much higher than that in creamy ice cream (Fig. 3).

As the data in Fig. 3 suggests, the main number of ice crystals in ice cream sundae is characterized by a size of less than 30 μm, in creamy ice cream – less than 40 μm.

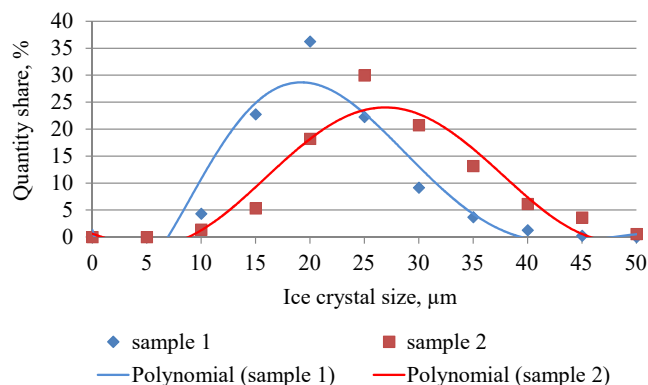


Fig. 3. Distribution of ice crystals for size: sample 1 – in ice cream sundae; sample 2 – in creamy ice cream

5. 3. Study of the effect exerted on nucleation by the suspended stabilizer particles

The high-molecular components used in the production of ice cream, stabilizers, may have a direct and indirect effect on nucleation. Stabilizers that are completely insoluble – directly; indirectly – stabilizers that are used at optimal concentration, which are in a colloidal degree of dispersion and which increase viscosity.

The synergistic composition of CMS and MCC was used as components influencing the nucleation.

Microcrystalline cellulose is purified cellulose (in crystalline form) derived from the reaction of acid hydrolysis of fibrous cellulose. This material is dried to a pure fine-dispersed form (for powder grades) or treated together with a water-soluble polymer (for example, cellulose gum). The polymer acts as a barrier dispersant so that the copolymer can be easily dispersed in water during intense agitation. This dispersion will recreate the colloidal form of microcrystalline cellulose. Such colloidal dispersions have very special properties compared to other soluble food hydrocolloids – they suspend solids, regulate the growth of ice crystals, stabilize emulsions, modify texture, heat resistant and form stable foams [4].

The effect of MCC and CMS on nucleation was investigated using an example of creamy ice cream, the composition was used in the amount of 0.93 %. The mass share of the composition of stabilizers was established experimentally, 0.2 % of CMS provides the required dynamic viscosity of the mixture, and 0.73 % of MCC – the presence of the required number of particles that are the nucleation initiators.

Taking into consideration the effect exerted by homogenization pressure on the dispersal of the fat phase and its relationship with the dispersal of ice crystals, the homogenization of mixtures was carried out at the optimal pressure of homogenization of 16 MPa at the first stage and 4 MPa at the second stage. Effective viscosity of the mixture after maturation was 258 mPa·s with a shifting gradient of 0.83 s⁻¹, the whipping of ice cream when unloaded from the freezer at a temperature of minus 4.2 is 89 %. The findings are typical of creamy ice cream with a fat mass share of 10 %.

We investigated the effect of MCC and CMS on the dispersal of ice crystals in the process of storage. The study results are given in Table 3.

Table 3

Change in microstructural elements in the ice cream storage process

Storage duration	Ice crystals		
	Average size, μm	Share, %, the size of	
		to 50 μm	to 70 μm
After hardening	31	98	100
1 month	31	97	100
3 months	39	85	99

As the data in Table 3 indicate, following the hardening of the samples, the average diameter of ice crystals was 31 μm. After 1 month of storage, the average size of ice crystals almost did not change, and after 3 months increased by 1.25 times and amounted to 39 μm, which is much less than the threshold of organoleptic perceptibility. Thus, it can be assumed that the use of the MCC+CMS complex contributes to the formation of small ice crystals.

5. 4. Studying the effect of coagulated protein on the dispersal of ice crystals in fermented milk ice cream

The production of fermented milk ice cream includes the process of fermentation of the mixture or the introduction of ready-made fermented milk products. In both cases, fermented milk ice cream contains milk protein in a coagulated state. Microscopy of the colored preparations for fermented milk ice cream established that the coagulated protein in it is distributed throughout the entire volume of the product in the form of suspended particles (Fig. 4). Therefore, similarly to microcrystalline cellulose, it can be the center that initiates nucleation.

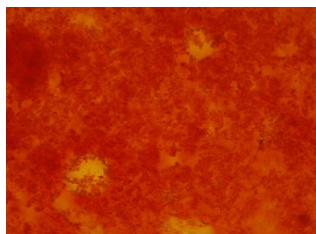


Fig. 4. Distribution of coagulated protein in fermented milk yogurt ice cream (microscope magnification x400)

The dispersal of ice crystals in ice cream with the same mass proportion of dry substances and fat was investigated: no fermentation (milk, sample 1), fermented milk ice cream with yogurt (30 % fermented base, sample 2), and yogurt ice cream (70 % fermented base, sample 3). The samples of ice cream were obtained by the conventional production technique; the formation of the structure took place in a freezer. It has been established that the dispersal of ice crystals, determined based on their greatest linear size, is the largest in a product containing 70 % of the fermented base (Fig. 5).

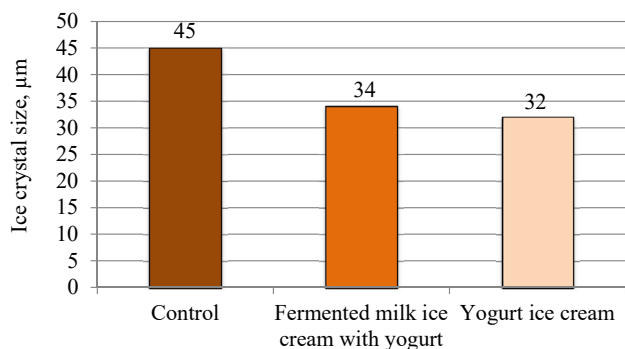


Fig. 5. The average size of ice crystals in ice cream: dairy (control) and fermented milk ice cream with yogurt and yogurt ice cream

As the data in Fig. 5 indicate, the size of ice crystals in milk ice cream is 1.4 times greater than that in fermented yogurt ice cream, which, given the same mass share of frozen-out water and the possibility of the formation of crystallization centers only at freezing, testifies to the larger number of crystallization centers in a fermented milk product.

6. Discussion of results of studying the directions of nucleation intensification in the production of ice cream

Our research addresses the task of preserving the dispersal of ice cream ice crystals – an important organolep-

tic indicator and a classification attribute of the product. In this case, the control of nucleation at the first stage of freezing was chosen as an effective technique. It was taken into consideration that the size of ice crystals in ice cream is largely determined by the number of crystallization centers formed exclusively at this stage at freezing (typically, 40–50 % of water). At post-freezing, the remaining water (30–40 %) crystallizes on existing ice crystals. Two areas have been justified to implement the task – increasing the freezing rate and using the particles that are the nucleation initiators. The first direction has been investigated using an example of immersion freezing in nitrogen. Suspended particles of the fat phase and microcrystalline cellulose and coagulated protein were considered as the particles that are the nucleation initiators. It has been experimentally substantiated that in all cases considered, the dispersal of ice crystals after production and during storage is noticeably higher than that in control samples (Fig. 1–3). The above, taking into consideration the characteristics of nucleation, allows us to argue that the specified factors have a positive effect on the nucleation.

The results of our study revealed additional factors for nucleation intensification in the production of ice cream (Table 3, Fig. 5). Their application would improve the quality indicators of the product, in particular the structure condition. A more detailed study into each of the specified factors of nucleation intensification could help control the dispersal of structural elements directly under production conditions. And the use of immersion freezing in nitrogen would give an impetus to the development of new technologies for obtaining ice cream with an adjustable proportion of frozen-out water at high dispersal of ice crystals. In addition, the study predetermines the possibility of substantiating new cryogenic substances and objects that initiate nucleation based on the effect of germ-forming agents.

7. Conclusions

1. The effect exerted on nucleation by immersion freezing in nitrogen has been investigated. It has been established that the use of immersion freezing in nitrogen in the first stage achieves a high dispersal of ice crystals, which persists during the storage process for at least 6 months.
2. The effect exerted on nucleation by the fat phase has been investigated. It has been established that as the mass proportion of fat increases with the same dry matter content in ice cream from 10 % to 15 % and the same proportion of frozen-out water, the average size of ice crystals decreases by 20 %, indicating the formation of the larger number of crystallization centers in a product with the higher mass proportion of fat.
3. The effect exerted on nucleation by the suspended stabilizer particles has been investigated. It has been shown that the use of the MCC+CMS stabilizer complex, which contains the suspended particles of MCC, contributes to the formation of small ice crystals (no more than 31 μm).
4. The effect exerted on nucleation by the coagulated protein has been investigated. It has been revealed that the size of ice crystals in fermented milk ice cream is 1.3 times less than that in milk ice cream with the same mass proportion of fat and dry substances.

Reference

1. Zhu, Z., Zhou, Q., Sun, D.-W. (2019). Measuring and controlling ice crystallization in frozen foods: A review of recent developments. *Trends in Food Science & Technology*, 90, 13–25. doi: <https://doi.org/10.1016/j.tifs.2019.05.012>
2. Kiani, H., Sun, D.-W. (2011). Water crystallization and its importance to freezing of foods: A review. *Trends in Food Science & Technology*, 22 (8), 407–426. doi: <https://doi.org/10.1016/j.tifs.2011.04.011>
3. Tvorogova, A. A., Konovalova, T. V., Spiridonova, A. V., Gurskii, I. A. (2016). The state of the ice crystals in the traditional ice-cream at storage. *Molochnaya promyshlennost'*, 8, 57–58.
4. Tvorogova, A. A., Konovalova, T. V. (2015). Grounds of technological functionality of native starches in ice cream production without food additives. *Holodil'naya tehnika*, 6, 39–42.
5. Gaukel, V. (2016). Cooling and Freezing of Foods. Reference Module in Food Science. doi: <https://doi.org/10.1016/b978-0-08-100596-5.03415-6>
6. Barey, F. (2007). Stabilizatsiya fazy kristallov l'da v morozhenom. *Pererabotka moloka*, 2, 27–29.
7. Casenave, C., Dochain, D., Alvarez, G., Arellano, M., Benkhelifa, H., Leducq, D. (2014). Model identification and reduction for the control of an ice cream crystallization process. *Chemical Engineering Science*, 119, 274–287. doi: <https://doi.org/10.1016/j.ces.2014.08.030>
8. Povey, M. J. W. (2014). Crystal nucleation in food colloids. *Food Hydrocolloids*, 42, 118–129. doi: <https://doi.org/10.1016/j.foodhyd.2014.01.016>
9. Zhu, Z., Luo, W., Sun, D.-W. (2020). Effects of liquid nitrogen quick freezing on polyphenol oxidase and peroxide activities, cell water states and epidermal microstructure of wolfberry. *LWT*, 120, 108923. doi: <https://doi.org/10.1016/j.lwt.2019.108923>
10. Estrada-Flores, S. (2016). Cryogenic Freezing of Food. Reference Module in Food Science. doi: <https://doi.org/10.1016/b978-0-08-100596-5.03175-9>
11. Ndoye, F. T., Alvarez, G. (2015). Characterization of ice recrystallization in ice cream during storage using the focused beam reflectance measurement. *Journal of Food Engineering*, 148, 24–34. doi: <https://doi.org/10.1016/j.jfoodeng.2014.09.014>
12. Lomolino, G., Zannoni, S., Zabara, A., Da Lio, M., De Iseppi, A. (2020). Ice recrystallisation and melting in ice cream with different proteins levels and subjected to thermal fluctuation. *International Dairy Journal*, 100, 104557. doi: <https://doi.org/10.1016/j.idairyj.2019.104557>
13. Konovalova, T. V., Tvorogova, A. A. (2017). Study of special features of the ice cream structure formation in a liquid nitrogen medium. *Holodil'naya tehnika*, 1, 58–64.