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STUDIES OF WATER FREEZING FEATURES IN ICE CREAM WITH STARCH SYRUP

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Abstract. The purpose of the study is to research the process of water freezing in new types of ice cream with starch syrup at certain stages of the technological process. Starch syrup as a degradation product of corn starch is characterized by different values of the dextrose equivalent (DE). Starch syrup is a source of solids, sweetener, cryoprotectants (at high DE) and thickener (for low values of DE). The starch syrups with fundamentally different functional and technological properties are chosen for the study: high glucose-fructose syrup HGFS-98 (DE = 98) and low-sugar starch syrup GFS-30 (DE = 30). To determine the size of ice crystals in ice cream, a light microscope of the brand XS-2610 with a cooling chamber is used for an increase of x600, and the cryoscopic temperature is measured by cryostat and Beckmann thermometer (TL-1) to calculate the content of frozen out water. The regularities of the process of water freeze-out in ice cream with milk fat content of 3.5%, creamy fat content of 10% and filling with fat content of 15% in the temperature range from minus 6°C to minus 40°C are established. In particular, the content of frozen water in ice-cream at certain stages of the technological process is determined. The results are used to optimize the prescription composition of ice cream with starch syrup. It is recommended to use hydrocarbon complexes consisting of HGFS-98 and GFS-30 in the ratio of 30:70 to 80:20 to reduce the content of frozen water in ice cream of different chemical compositions. The results of the study are of practical importance and allow to obtain in production conditions the fine-crystalline structure of ice cream with starch syrup.

Key words: ice cream, starch syrup, cryoscopic temperature, water freezing.

ДОСЛІДЖЕННЯ ОСОБЛИВОСТЕЙ ВИМОРОЖУВАННЯ ВОДИ В МОРОЗИВІ З КРОХМАЛЬНОЮ ПАТОКОЮ

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Анотація. Досліджено процес виморожування води у нових видах морозива з крохмальною патокою на окремих етапах технологічного процесу. Патока крохмальна як продукт деструкції кукурудзяного крохмалю характеризується різними значеннями декстрозного еквіваленту (ДЕ). Патока є джерелом сухих речовин, підсолоджувачем, кріопротектором (за високих значень ДЕ) і загущувачем (за низьких значень ДЕ). Для проведення дослідження обрано патоку з принципово різними функціонально-технологічними властивостями: глюкозно-фруктозний сироп HGFS-98 (ДЕ = 98) і патоку карамельну GFS-30 (ДЕ = 30). Для визначення розмірів кристалів льоду у морозиві застосовували світловий мікроскоп марки XS-2610 з охолоджуючою камерою за збільшення x600, а для розрахунку вмісту вимороженої води вимірювали кріоскопічну температуру за допомогою кріостата і термометра Бекмана (ТЛ-1). Встановлено закономірності процесу виморожування води у морозиві молочному жирністю 3,5%, вершковому жирністю 10% та пломбірі жирністю 15% в температурному діапазоні від мінус 6°C до мінус 40°C. Зокрема, встановлено вміст вимороженої води у морозиві на окремих етапах технологічного процесу. Одержані результати застосовано для оптимізації рецептурного складу морозива з крохмальною патокою. Так, для зниження вмісту вимороженої води у морозиві різного хімічного складу, рекомендовано застосовувати вуглеводні комплекси, що складаються з HGFS-98 і GFS-30 за співвідношень від 30:70 до 80:20. Результати дослідження мають практичну значимість і дозволяють керувати одержувати у виробничих умовах дрібнокристалічну структуру морозива з крохмальною патокою.

Ключові слова: морозиво, крохмальна патока, кріоскопічна температура, виморожена вода.

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Introduction. Formulation of the problem

The dispersion of ice cream is free and bound water in its total content in the range of 58 to 75%. The physical and chemical properties of bound water influence the cryoscopy temperature (t_{cr}) and, accordingly, the nature of water crystallization during freezing, tempering and storage [1]. The water in the ice cream is actively bound by stabilizers, milk proteins, mono- and disaccharide, and practically does not crystallize during low-temperature processing [2]. However, about one third of the total content of bound water is characterized by rather weak link energy and also has the ability to freeze [3,4].

High quality ice cream contains small ice crystals of no more than 55–60 microns. The reason for the formation of the roughly crystalline structure of the product is the presence of large ice crystals. In the first place, the chemical composition of ice cream affects the degree of freezing of water: the content of sugars and fat [5,6]. The crude crystalline structure of ice cream may also arise due to slow tempering and migration recrystallization of water during storage at a temperature above-minus 12°C, in which the content in the product of frozen water becomes less than 50% [7-9]. That is why there is a need for additional study of the features of freezing water in ice cream for the variable fat content and various carbohydrate compositions of sweeteners during the technological process of its production.

Analysis of recent research and publications

For the production of ice cream and frozen desserts, sweeteners-cryoprotectants are widely used: disaccharides (sugars, lactose, maltose), monosaccharides (glucose, fructose), invert sugar (a product of an acidic glucose and fructose sucrose inversion product), starch syrup (a product of enzyme hydrolysis of starch, containing a complex of sugars of different molecular weights), etc. [3,10]. The process of low temperature treatment of mixtures and ice cream is accompanied by rapid and deep freezing of the water phase to temperatures in the average range of minus 20 to minus 40°C, which leads to a strong over-saturation of solutions of mono- and disaccharides. Due to lower solubility, disaccharides can form large crystals on the surface of ice cream based on sugar syrups [11]. That is why it is recommended to change the sugar content of such ice cream in the amount of 25–30% by 20–25% for starch syrup, invert sugar or monosaccharides [12]. However, this recommendation does not apply to dairy ice cream, in which the content of sugar and lactose reaches 14.0–15.5% and 4.0–5.5% respectively.

For the first time, the use of liquid starch syrup in ice cream for the partial replacement of sugar was proposed by O. Yakovleva et al. But this decision is technologically incorrect, because the authors did not take into account the most important physical and

chemical parameters of starch syrup – its dextrose equivalent and the content of dry substances [10].

The authors have previously investigated the possibility of replacing 25–50% sucrose with invert sugar in ice cream with a milk fat content of 3.5%. It is established that due to the reduction of the cryoscopic temperature (t_{cr}), resistance to melting of the product becomes smaller, which is supposed only for soft ice cream and ice cream type “bird milk” [14]. The disadvantage of using invert sugar is that it may contain oxymethylfurfural (OMF) – a product of deep acid hydrolysis of sucrose. Therefore, it was decided to choose as a sweetener in the ice cream starch syrup, which is a product of enzymatic hydrolysis of starch and does not contain OMF. The advantage of starch syrup, in particular low-sugars, compared to invert sugar, is also the ability to improve the texture of the ice cream and increase the perception of the aroma by the structuring of higher sugars [15-17]. It should also be noted that starch syrup is a cheaper sweetener and a source of dry substances, compared with white crystalline sugar. Starch syrups with different values of dextrose equivalent in large quantities are produced in Ukraine for the needs of the confectionery industry, which gives wide prospects for its use also in ice cream production. With the change in the composition of starch syrup in the content of median-molecular and low molecular weight carbohydrates can be regulated in ice cream cryoscopic temperature, the content of frozen water, the size of ice crystals, consistency, etc., which needs scientific confirmation.

The purpose of the study is to investigate the effect of starch syrup with different dextrose equivalent on the degree of water **freezing in milk-based ice cream during the production process.**

Research tasks are:

1. To investigate the regularities of the process of freezing water in ice-cream of different fatty content with starch syrup in a wide temperature range of thermo-mechanical processing;
2. To conduct mathematical modeling of the process of freezing water in ice cream of different carbohydrate composition at separate stages of the technological process;
3. To analyze the peculiarities of the cryoprotective abilities of starch syrup with different degree of saccharification and to choose the most effective variants of its application for managing the process of freezing water in ice cream.

Research materials and methods

For the study, dry starch syrups with fundamentally different functional and technological properties were chosen: high fructose corn syrup HGFS (DE = 98) and low-sugar starch syrup GFS-30 (DE = 30).

HGFS contains 54% glucose and 42% fructose from the total dry matter content and has a sweetness index of 0.98. The GFS contains 10% glucose, 20% maltose, 70% higher sugars and is characterized by a sweetness index of 0.3.

The basic composition of ice cream is: milk ice cream (fat 3.5%; sugar 15%; dry matter 29%); creamy ice cream (fat – 10%, sugar – 14%; dry matter 34%); plombir (fat 15%; sugar 14%, dry matter 39%).

In all ice cream samples, a universal stabilizing system Cremodan SE 406 (Danisco) was used in recommended amounts of 0.4% (plombir), 0.5% (cream) and 0.6% (milk).

According to the basic composition of the ice cream, formulations of experimental samples with a 100% replacement of sugar on the starch syrups HGFS-98 and GFS-30 and their compositions were developed. The ratio between the syrups in the compositions was taken from 10:90 to 90:10.

Ice cream mixtures were pasteurized at a temperature of $85 \pm 2^\circ\text{C}$ for 2–3 minutes, cooled to $4 \pm 2^\circ\text{C}$ and kept at this temperature for 12 hours. Plumbing mixes, which include butter, are homogenized on a valve type homogenizer at a pasteurization temperature and a pressure of 8–10 MPa in the first stage and 2.5–3 MPa in the second one.

Experimental ice-cream production was carried out on freezers brand FPM-3.5 / 380-50 "Elbrus-400" in terms of loading capacity of a screw chamber of 4 dm^3 . The duration of cooling (mode 1 with the speed of the auger mixer 270 min^{-1}) and the freeze of the mixtures (mode 2 with the speed of the auger mixer 540 min^{-1}) was 3 minutes. The samples were hardened and stored in the freezer "Caravell" A/S at temperature of minus 18–20°C.

The cryoscopic temperature of ice cream mixtures was determined using a cryostat and a Beckmann thermometer (TL-1) [3], the scale of which was calculated at 5°C and divided up to 0.01°C without constant zero point.

On the basis of Raoult's law for non-dissociated molecular solutions, the amount of frozen moisture at different temperature regimes was calculated according to the formula [4]:

$$\omega = (1 - (t_{cr}/t)) \cdot 100 \quad (1)$$

where ω – quantity of frozen water, %;

t_{cr} – cryoscopic temperature, $^\circ\text{C}$, t – temperature, $^\circ\text{C}$.

To determine the size of the ice crystals in the ice cream, a light microscope of XS-2610 with a cooling chamber was used with the temperature not higher than minus 12°C . For $\times 600$, microfilms were received. With the aid of an objective micrometer of an eyepiece grid with a price of $20 \mu\text{m}$ for the selected increase, the average sizes of ice crystals in the studied samples were weighed not less than in 5 fields of view.

Results of the research and their discussion

In the technological process of production of hardened ice cream for the control of its quality, it is

necessary to take into account the content of frozen water, the nature of its freezing and factors that affect this indicator. For this purpose, the features of freezing of water was investigated depending on the technological stages and the variability of the composition of the sweetener and the content of fat.

At the first stage of scientific research, the cryoscopic temperature of ice cream samples of the traditional composition with sugar and ice cream with a variable carbohydrate composition with complete replacement of sugar in the lower (control 1) and upper (control 2) ranges of fat content on the starch syrups HGFS-98, GFS-30 and their compositions (Fig. 1) was determined.

According to Fig. 1, the lack of a HGFS-98 for replacing sugar of a highly structured GFS-30 with a low dextrose equivalent does not allow to lower the cryoscopic temperature to the values adopted for ice cream of traditional species. The result of high values of cryoscopic temperature may be the excess freezing of free water with the formation of a coarse-crystalline structure. In the case of HGFS-98 alone, the reduction of cryoscopic temperature is too significant, which may be acceptable only for the production of soft ice cream and ice cream soufflé.

To assess the influence of sugary substances in the composition of starch syrups on the process of water freezing in ice cream for the main technologically significant stages of low-temperature processing, the following were adopted:

- stage № 1 – the temperature of freezing and yield of soft ice cream from the freezer (minus 6°C);
- stage № 2 – threshold temperature of storage of hardened ice cream (minus 12°C);
- stage № 3 – storage mode of ice cream for up to 10 months (minus 18°C);
- stage № 4 – storage mode of ice cream for up to 12 months (minus 24°C);
- stage № 5 – the bottom limit of the hardening of ice cream in a continuous manner (minus 30°C);
- stage № 6 – the upper limit of ice-cream drying in a continuous manner (minus 40°C).

Since two removable factors influence the content of frozen water in ice cream – the chemical composition of starch syrup and the temperature regimes that accompany the technological process, a two-factor regression analysis of the experimental data obtained for ice cream samples of classical species (milk ice cream (fat 3.5%), creamy ice cream (fat 10.0%), lombir (fat 15.0%)) content was carried out. An important condition for such an analysis is the lack of a functional connection between the factors.

Mathematical models were obtained in the form of analytical expressions that reflect the connection of factor characteristics with a productive feature.

In general, the equation of multiple regressions can be expressed as:

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \varepsilon, \quad (2)$$

where β_0 is a free member that defines the values Y_1, Y_2, Y_3 in the case when all independent variables X_1 are equal to 0.

The following mathematical models are obtained:

- for milk ice cream –

$$Y_1 = 76.65 + 7.17X_1 - 1.08X_2; \quad (3)$$

- for creamy ice cream –

$$Y_2 = 75.37 + 8.06X_1 - 1.17X_2; \quad (4)$$

- for plombir –

$$Y_3 = 74.92 + 8.29X_1 - 1.19X_2; \quad (5)$$

where X_2 is the cryoscopic temperature, °C;

X_1 is the processing temperature, °C;

Y_1 – the content of frozen water in milk ice cream, %;

Y_2 – the content of frozen water in creamy ice cream, %;

Y_3 – the content of frozen water in plombir, %.

To verify the models for adequacy, the adjusted determination coefficients are calculated according to the formula:

$$R^{-2} = 1 - (1 - R^2) \frac{n-1}{n-m-1} \quad (6)$$

where, R is the determination coefficient, which is found by the formula:

$$R = \sqrt{\sum r_{yx_i} \beta_{yx_i}} = \sqrt{r_{yx_1} \beta_{yx_1} + r_{yx_2} \beta_{yx_2}} \quad (7)$$

For samples of milk ice cream, creamy ice cream and plombir: $R_1 = 0,946$; $R_2 = 0,93$; $R_3 = 0,98$.

On the basis of the obtained mathematical models, graphic 3D models were developed demonstrating the features of water freezing at separate stages of the process (Fig. 2) for milk ice cream.

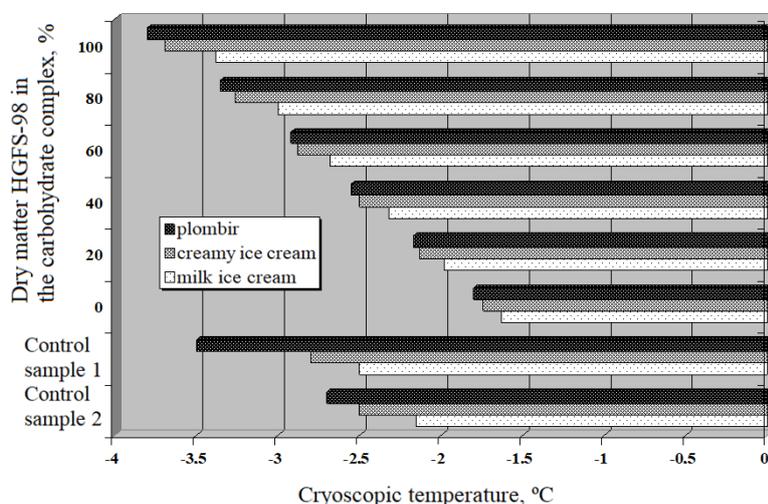


Figure 1. Cryoscopic temperature of ice creamsamples of traditional composition with sugar (Control samples) and ice cream with complete replacement of sugar on syrup HGFS-98 and GFS-30 and their compositions

Symbols: Control 1 – samples of ice cream with a minimum fat content (milk ice cream – 0.5%, creamy ice cream – 11.5%, plombir – 20.0%); Control 2 – samples of ice cream with maximum fat content (milk ice cream – 7.5%, creamy ice cream – 11.5%, plombir – 20.0%).

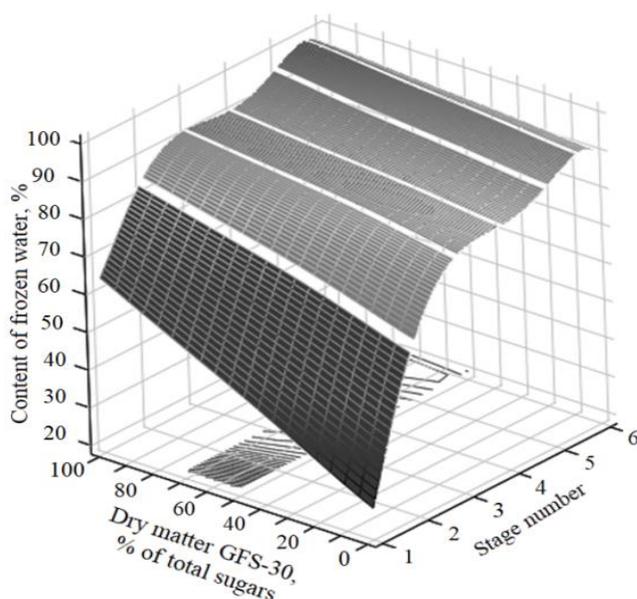


Figure 2. Graphic 3D model of water freezing features at individual stages of the technological process for milk ice cream

On X axes (Figure 2), the recommended range of GFS-30 in compositions with molasses HGFS-98 + GFS-30 have been identified, which meet the requirements for the content of frozen water for control samples of the traditional composition with sugar.

According to Fig. 1, the cryoscopic temperature of control samples of ice cream with sugar was:

- milk ice cream (from minus 2.17 to minus 2.5°C);
- creamy ice cream (from minus 2.5 to minus 2.8°C);
- plombir (from minus 2.7 to minus 3.5°C).

These values were listed in accordance with formula (1) to obtain rational values the content of frozen water.

For milk ice cream, it is possible to achieve the recommended value of frozen water at all stages of the process with the content of GFS-30 in the composition of starch syrup not less from 50 to 70%. For creamy ice cream, content of the GFS-30 in the composition should be from 40 to 60%, and for plombir is – from 20 to 50%. Of course, these limit values may be recommended for industrial production of ice cream. With increasing in the GFS-30 content, the cryoscopic temperature will decrease accordingly and it is rational for the production of soft ice cream and ice cream soufflé. It is also advisable to vary the content of the GFS-30 for increasing the degree of sweetness of the finished product.

Fig. 2 shows practically the same nature of water freezing for ice cream with different fat content. It is obvious that the main part of the water is frozen during stages No. 1–2 (up to 74–78%) for all samples of the recommended composition of starch syrup. At stages number 3–6, the crystallization process is slowed down, reaching its maximum at the upper limit of hardening by a continuous method of 92–93%. Thus, the most important role of the freezing process is

confirmed, during which a considerable part of free water is frozen, the content of which, in the first place, depends on the chemical composition of ice cream. It is also understandable to observe the minimum possible temperature regime of ice cream storage (minus 12°C), above which there is a threat of mass recrystallization of water to form a coarse-crystalline structure. Such temperature fluctuations are most dangerous during the transportation and sale of ice cream in the trading network.

For milk ice-cream with a high crystallization temperature in the zone of the beginning of crystallization during freezing, the amount of frozen water increases substantially (up to 50%), followed by a sharp braking. And for plombir and creamy ice cream with a low crystallization temperature, the freeze-out of water is somewhat slowed down. For these types of ice cream is characterized by a smaller amount of frozen water (within 37% and 46%). The cause of this effect is the different binding energy between the particles of the dispersion medium, which depends on a number of factors, primarily on the mass fraction of fat in the ice cream [9]. Considering the high water content (68–72%) for low-fat types of ice cream, the amount of hard water for milk ice cream is the smallest in comparison with the high-fat types of ice cream. This is the reason for the rapid emergence of ice crystals at the beginning of freezing, which then are grown in agglomerates of large sizes rising, which are then merged into larger agglomerates.

To illustrate the nature of the effect of higher structuring sugars and cryoprotectants-monosugars in the starch syrup composition, the size of ice crystals, in Fig. 3, micrographs of ice-cream samples of dairy classic type and milk with starch syrup are given. The samples of ice cream were subjected to the previous one low-temperature treatment – quenching at a temperature of minus 30°C and storage at a temperature of minus 18°C for 1 month.

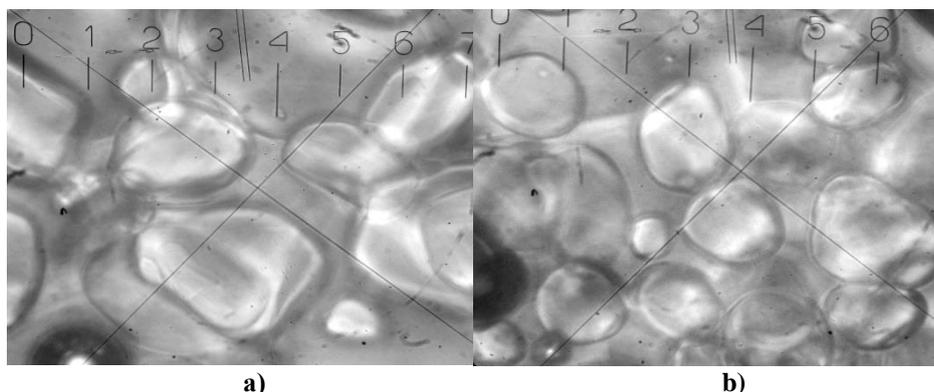


Figure 3. Microstructure of milk ice cream with sugar (control) (a) and with the composition of starch syrup (b) for an increase of 600 times (price of dividing 20 microns).

Obviously there is a significant technological effect in the case of complete replacement of sugar in the composition of milk ice cream on the composition of HGFS-98 + GFS-30. The average size of ice crystals

in ice cream decreased from 30,1 μm (control of sugar) to 24,2 μm (ice cream with starch syrup).

Consequently, to form the fine crystalline structure of milk ice cream, first of all, in the first stages of low

temperature treatment, it is necessary to adjust its chemical composition; in particular, it is recommended to replace the sugar on the composition of starch syrup. The role of the HGFS-98 is to bind active water and to change the cryoscopic temperature, and the role of the GFS-30 is to structure the water phase and to counteract the migration of water to the surface of small crystals of ice. So with the decrease in the content of fat in ice cream, the need for structured caramel syrup increases and vice versa. Introduction to the composition of ice cream dairy higher sugars in the composition of the starch syrup with low DE prevents the formation of a coarse-crystalline structure.

The prospect of further research is to conduct a three-factor regression analysis (taking into account changes in the content of fat) and to obtain a universal mathematical model that can be used for ice cream in a wide range of fat content – from 0.5 to 20.0%, in accordance with existing regulatory requirements.

Conclusion

The regularities of the process of water freezing in ice cream with various fat content and starch syrup in

the temperature range from minus 6°C to minus 40°C have been experimentally established. It has been revealed that the process of water freezing occurs more deeply for reducing the fat content and corresponding increase in the water content in ice cream. Established regularity confirms the important compliance with the temperature regimes of the production process of ice cream and the appropriateness of the use of ingredients with cryoprotective ability and structures the water phase.

Mathematical models of water freezing specifics at certain stages of the technological process have confirmed practically the same regularity of this process for milk ice-cream, creamy and cream-based ice-cream and a large role in the formation of the crystal structure of ice-cream temperature regimes of freezing and storage.

The expediency of using carbohydrate complexes consisting of HGFS-98 and GFS-30 in the ratio from 30:70 to 80:20, depending on the fat content, is proved in the ice cream. Complete replacement of sugar on starch syrup allows to control the process of water freezing in ice cream.

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