# Phase Transitions of Sweetened Condensed Milk in Extended Storage Temperature Ranges

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**Abstract.**
Sweetened condensed milk is a popular food in various climatic zones, including those regions where average winter temperature falls below –30°C. Such low temperatures can trigger crystallization because they disrupt the native structure of biopolymers. These processes spoil the quality of sweetened condensed milk. However, no scientific publications feature the cryoscopic temperature of sweet condensed milk or systematize the data on its low-temperature storage.

Sugar, sugar-milk, and milk solutions of various concentrations were frozen to determine their cryoscopic temperature by the thermographic method using a Testo 176T4 meter (Germany) with K-type probes (NiCr-Ni) at –78.5°C. The phase transitions were studied using a Mettler Toledo DCS 822e DSC analyzer.

The nucleation temperature, the cryoscopic temperature, and the subcooling degree depended on the concentration and the type of the solute. For sugar solutions, the cryoscopic temperature varied from –0.4 ± 0.1 to –6.4 ± 0.1°C; for sugar-milk solutions, it ranged from –2.1 ± 0.1 to –10.9 ± 0.1°C; for whole milk solutions, it was from –0.4 ± 0.1 to –4.6 ± 0.1°C. The thermographic method failed to obtain the phase transition and the cryoscopic temperature in analogue models of sweetened condensed milk. The loss of fluidity was about –30°C when the storage time exceeded 2 h. This effect was comparable to 54 min of storage at –35°C. The differential scanning calorimetry method showed that the phase transition occurred at –80°C.

This research opens new prospects for differential scanning calorimetry studies of phase transitions in condensed sweetened dairy products.

**Keywords.** Sweetened condensed milk, cryoscopic temperature, freezing, loss of fluidity, storage

# Фазовые переходы сгущенного молока с сахаром в расширенных температурных диапазонах хранения

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**Аннотация.**
Сгущенные молочные консервы с сахаром пользуются спросом у потребителей в различных климатических поясах, в том числе в регионах со среднегодовой температурой зимой более –30 °С. Хранение при отрицательных температурах может оказывать негативное влияние на качественные показатели продукта, что связано с процессами кристаллизации и нарушения нативной структуры биополимеров. Однако современные систематизированные данные о процессах в области низкотемпературного хранения, а также криоскопической температуры сгущенного молока с сахаром практически отсутствуют.

Объектами исследования являлись сахарные, сахарно-молочные и молочные растворы различной концентрации. Их замораживали и определяли криоскопическую температуру термографическим способом с применением измерителя Testo 176T4 с зондами K-типа (NiCr-Ni) при температуре охлаждения до –78,5 °С. Фазовые переходы в сгущенном молоке с сахаром исследовали с применением дифференциальной сканирующей калориметрии анализатора Mettler Toledo DCS 822e.

Установлено, что температуры нуклеации, криоскопическая температура и степень переохлаждения зависели от концентрации и вида растворимого вещества. Криоскопическая температура составила для сахарных растворов с различной массовой долей сухих веществ от –0,4 ± 0,1 до –6,4 ± 0,1 °С, для сахарно-молочных растворов – от –2,1 ± 0,1 до –10,9 ± 0,1 °С, для растворов цельного молока – от –0,4 ± 0,1 до –4,6 ± 0,1 °С. В моделях-аналогах сгущенного молока с сахаром зафиксировать фазовый переход и определить криоскопическую температуру термографическим методом не удалось. Однако установлено, что потеря текучести моделей-аналогов ориентировочно фиксируется при температуре –30 °С и хранении более 2-х часов. Данный эффект был сопоставим с хранением на протяжение 54 мин при температуре –35 °С. Исследования сгущенного молока с сахаром методом дифференциальной сканирующей калориметрии показали, что фазовый переход наступает при температуре около –80 °С.

Созданы предпосылки для глубокого исследования фазовых переходов в сгущенных молочных продуктах с сахаром с применением дифференциальной сканирующей калориметрии.

**Ключевые слова.** Сгущенное молоко, криоскопическая температура, замораживание, текучесть, хранение

## Introduction

Sweetened condensed milk is a strategic high-energy food product because it is rich in milk and sucrose [1–6]. It is especially popular in the Business-to-Consumer segment and highly applicable in the daily diet. Its use patterns in the industrial sector are also extremely diversified, the main consumers being the confectionery industry and the ice-cream production [7–13]. Sweetened condensed milk has a high nutritional value and a long shelf life, which explains why this product has become an integral part of the state food reserve, humanitarian aid, dry rations, etc. [4, 7, 11]. Regions with no dairy farming of their own are stable consumers of sweetened condensed milk. The indigenous peoples of the Russian Arctic are known to depend on sweetened condensed milk because they have to eat a lot of high-carbohydrate foods [6]. The air temperature in the region can fall much below –40°C. As a result, consumers cannot but violate

Table 1. Formulation of sugar-milk solutions

Таблица 1. Рецептурный состав сахарно-молочных растворов

| Component | Variants of solution models | | | |
|---|---|---|---|---|
| | Sucrose solution | | | |
| | A1 | A2 | A3 | A4 |
| Sugar, g | 15.0 | 30.0 | 45.0 | 68.0 |
| Distilled water, g | 85.0 | 70.0 | 55.0 | 32.0 |
| | Sugar-milk solution | | | |
| | B1 | B2 | B3 | B4 |
| Whole milk powder, g | 28,5 | | | |
| Distilled water, g | 85.0 | 70.0 | 55.0 | 32.0 |
| Sugar, g | 15.0 | 30.0 | 45.0 | 68.0 |
| Solids, % | 33.9 | 45.5 | 57.2 | 75.1 |
| | Whole milk solution | | | |
| | C1 | C2 | C3 | C4 |
| Whole milk powder, g | 12.5 | 25.0 | 37.5 | 50.0 |
| Distilled water, g | 87.5 | 75.0 | 62.5 | 50.0 |

the recommended storage conditions of 0–10°C. However, industry experts put stress on the universality of the use of sweetened dairy products, regardless of climatic conditions and geographical location, which explains the relevance of research on their low-temperature storage.

Our review of scientific and technical publications revealed no systematic data on the freezing point of sweetened condensed milk and its subsequent storage at freezing temperatures [14–19]. The only publication that mentioned low-temperature storage of condensed milk was that by Pavlova, where the cryoscopic temperature of fresh sweetened condensed milk ranged from –26 to –29°C [14]. When the product reaches the cryoscopic temperature, moisture begins to crystallize. The resulting crystals of various shapes can trigger the abiogenic degradation of macrocomponents, which implies a decrease in storage stability [20–23]. Low storage temperature also reduces the rate of biochemical reactions.

In theory, the freezing process for simple solutions means that the stability of one phase ends at a certain point that corresponds to a particular set of system variables. In practice, point phase transitions are abstractions that imply infinite ideal and defect-free systems [24]. In real systems, they are *a priori* blurred. The diffusion dilemma consists in determining the range of characteristic values. In some cases, this diffusion can be so weak that the point phase transition stops being an abstraction. In other cases, the phase transition can be so blurred that its limits are impossible to determine. Therefore, no boundary exists between phase transitions of the first and second levels. For instance, point phase transition can be determined for the system of water – extra pure sucrose but not for the system of water – food
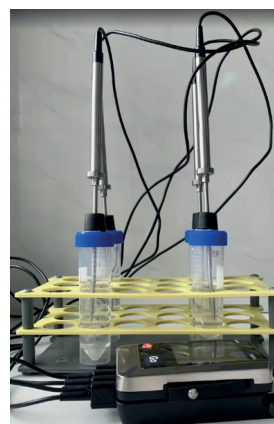


Figure 1. Experimental stand for determining the cryoscopic temperature

Рисунок 1. Экспериментальный стенд для определения криоскопической температуры

grade sucrose – milk powder, for which the probability of a point phase transition vanishes as the concentration of the components increases.

The present research objective was to analyze the change in the aggregate state of analogue models of sweetened condensed milk in the temperature range from +20 to –50°C and to determine its cryoscopic temperature.

**Study objects and methods**

The research was conducted at the Laboratory of Canned Dairy Products of the All-Russian Dairy Research Institute.

The study featured sugar, sugar-milk, and milk solutions of various concentrations, whole milk powder,
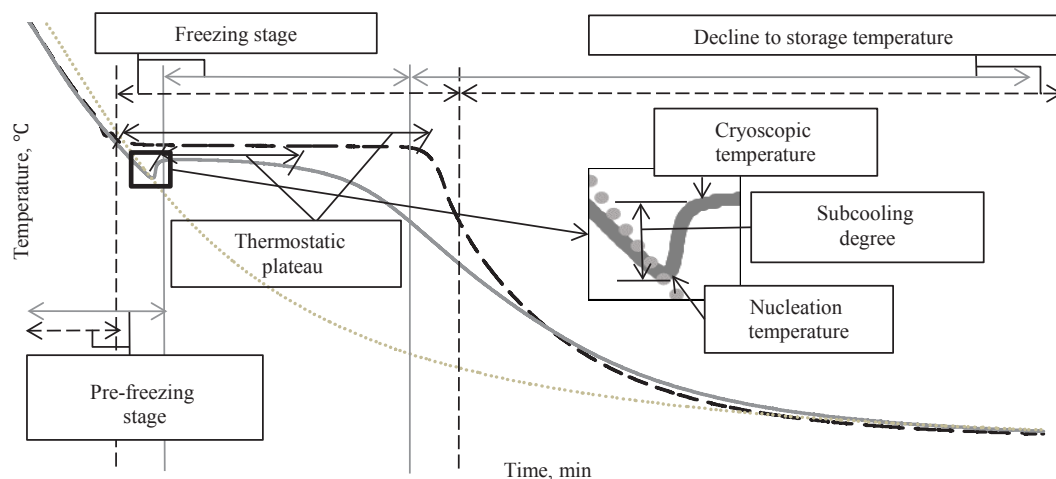
Figure 2. Freezing process

Рисунок 2. Схематическое представление процесса замораживания

and commercial samples of sweetened condensed milk (State Standard 31688).

The sugar solutions had a concentration of 15, 30, 45, and 68%, while the milk solutions had a concentration of 12.5, 25, 37.5, and 50%. They were prepared by dissolving a certain mass of sucrose and whole milk powder in a given amount of water at 25 and 40°C, respectively. The calculations ignored the moisture content in the powder.

To assess the effect of the milk solid concentration on freezing and defrosting, the sugar-milk solutions were obtained by restoring the whole milk powder at 40°C for 20 min, followed by adding sugar according to State Standard 33222-2015 (Table 1). The whole milk powder was produced according to State Standard 33629-2015: 96.46% solids, 26% fat, 26% protein, solubility index = 0.1 mL of crude residue. The low saturation of the solution was maintained by the controlled mixing rate of 27 min$^{-1}$ during the dissolution process.

All the solution samples weighed 35 g. They were poured into plastic flasks and sealed hermetically with lids with integrated temperature probes (Fig. 1).

The solutions were frozen in a low-temperature laboratory freezer Vestfrort VT 327 (Denmark) at –50 ± 1°C. Solid carbon dioxide in thermally insulated containers made it possible to obtain temperatures as low as –78.5 ± 0.5°C. The temperature was recorded every second using a combined Testo 176T4 meter (Germany). The device had four waterproof food probes made of K-type stainless steel (NiCr-Ni) with boundary values from –60 to –400°C. The samples were stored at the specified temperature. After they were taken out of the freezer, they thawed at 22 ± 1°C. Instrument readings and data export to Microsoft Excel for analysis and visualization were processed using the Testo-ComSoft Basic software.

The cryoscopic temperature was determined thermographically based the temperature curve plateau [25].

The fluidity of sweetened condensed milk analogue models was determined by freezing, followed by retrievability and visual assessment of the probes at –25 ... –50°C under refrigerator conditions.

The thawing kinetics of the analogue models was studied using a DSC822e Mettler Toledo differential scanning calorimeter under conditions of dynamic heating at a constant rate (https://www.mt.com). The results were processed using the STAR$^e$ software.

**Results and discussion**

We divided the freezing process into three successive stages to ensure the terminological uniformity (Fig. 2):
– pre-freezing stage: the period of time between the start of freezing and the cryoscopic temperature (freezing point). At this stage, the system is supercooled to the nucleation temperature to trigger the nucleation of ice crystals. The release of latent crystallization heat corresponds to the start of the thermostatic plateau;
– freezing stage: the temperature at the considered area of the product is almost constant because the heat removal makes a large amount of water turn into ice, i.e., phase transformation;
– decline to storage temperature: most of the water has frozen, and the temperature decreases to the required end temperature [26].

At the first stage, we determined how the time of rational low-temperature storage of solutions affected their aggregation state. The main criterion for determining the exposure time in the dynamic temperature – time – concentration system was to register all the stages of the freezing process. Figure 3 shows the data for sugar
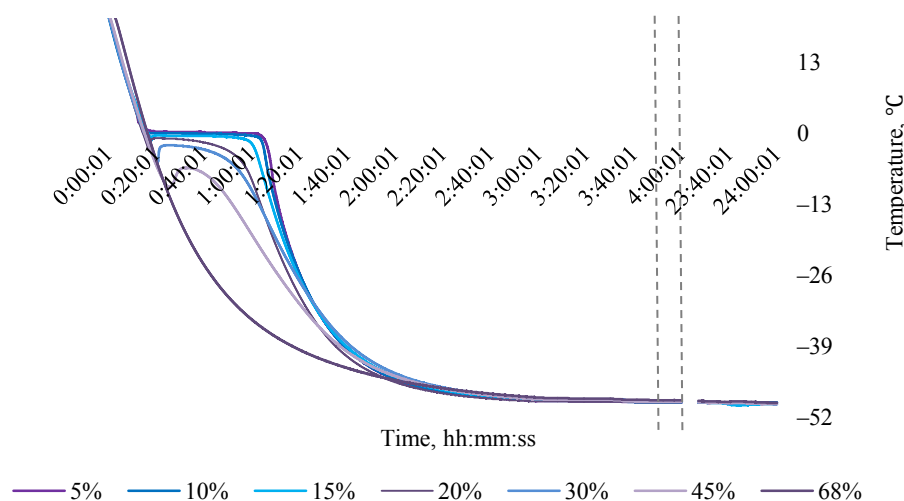
Figure 3. Thermograms of sugar solutions at various concentrations

Рисунок 3. Термограммы сахарных растворов различной концентрации

Table 2. Moisture crystallization criteria in sugar, sugar-milk, and milk solutions.

Таблица 2. Критерии кристаллизации влаги в сахарных, сахарно-молочных и молочных растворах

| Variants of solution models | Indicator | | | | |
|---|---|---|---|---|---|
| | Nucleation temperature, °C | Cryoscopic temperature, °C | Subcooling degree | Freezing time, s | Point phase transition, s |
| A1 | −1.4 ± 0.1 | −0.4 ± 0.1 | 1.0 | 2827 | 2922 |
| A2 | −5.3 ± 0.1 | −2.2 ± 0.1 | 3.0 | 2308 | 2500 |
| A3 | −9.9 ± 0.1 | −6.4 ± 0.1 | 3.5 | 1570 | 1885 |
| A4 | − | − | − | − | − |
| B1 | −2.1 ± 0.1 | −2.1 ± 0.1 | 0 | 2224 | 2224 |
| B2 | −5.0 ± 0.1 | −5.0 ± 0.1 | 0.9 | 1579 | 1927 |
| B3 | −10.9 ± 0.1 | −10.9 ± 0.1 | 1.3 | 1276 | 1635 |
| B4 | − | − | − | − | − |
| C1 | −0.4 ± 0.1 | −0.4 ± 0.1 | 0 | 2730 | 2730 |
| C2 | −1.2 ± 0.1 | −1.2 ± 0.1 | 0 | 2850 | 2850 |
| C3 | −2.6 ± 0.1 | −2.6 ± 0.1 | 0 | 2280 | 2280 |
| C4 | −4.6 ± 0.1 | −4.6 ± 0.1 | 0 | 1740 | 1740 |

solutions. Milk and sugar-milk solutions underwent the same procedure.

The onset of moisture crystallization process correlated with the temperature of the system: for 5% sucrose, the crystallization temperature was 0°C; for 45% sucrose, it was –9.9°C. Also, it was inversely dependent on the concentration of solids. The crystallization time decreased in proportion to the concentration of sucrose: for 5% sucrose, it was 39 min 14 s; for 45% sucrose, it lasted 6 min 40 s. However, no change in the aggregate state of the system was recorded as the sucrose concentration reached 68%. This effect was probably associated with the concentration features of the system, or inability to reach the critical temperature at which the phase transition occurs. Thus, it took the system four hours to stabilize completely and for the

temperature of the solutions to reach that of the external environment.

Table 2 shows the values of the obtained criteria that describe the process of freezing sugar, sugar-milk, and milk solutions.

Models A4 and B4 were analogues of sweetened condensed milk with sugar. They demonstrated no liquid-solid phase transition. However, a visual inspection revealed a change in the transparency of the solutions. All the samples had a firm texture, typical of frozen foods. The nucleation temperature, the cryoscopic temperature, and the subcooling degree directly depended on the concentration and the type of the solute. The dairy component had a strong impact on these indicators. The freezing time and the phase transition period declined as the concentration increased.
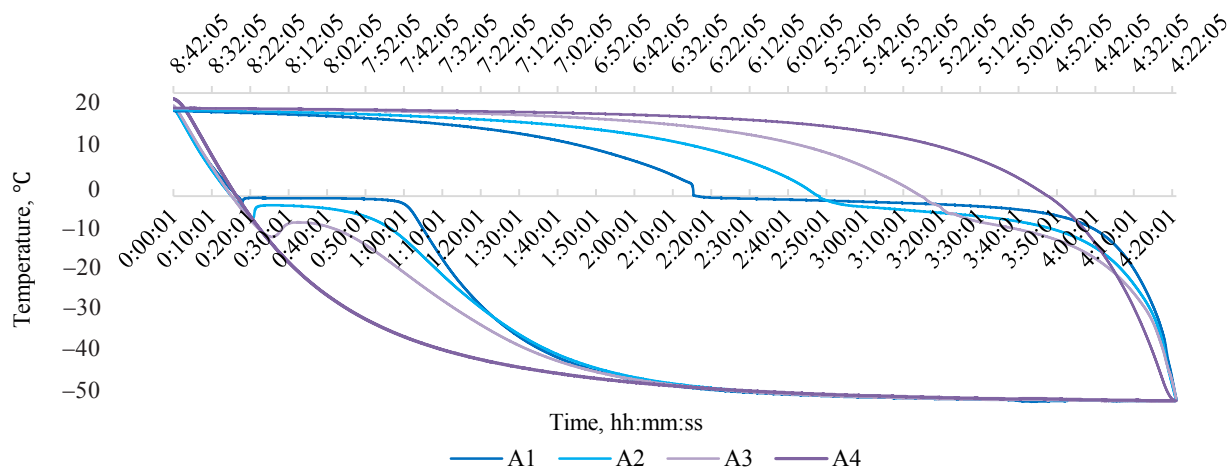
Figure 4. Freezing and defrosting curves for sugar solutions at various concentrations

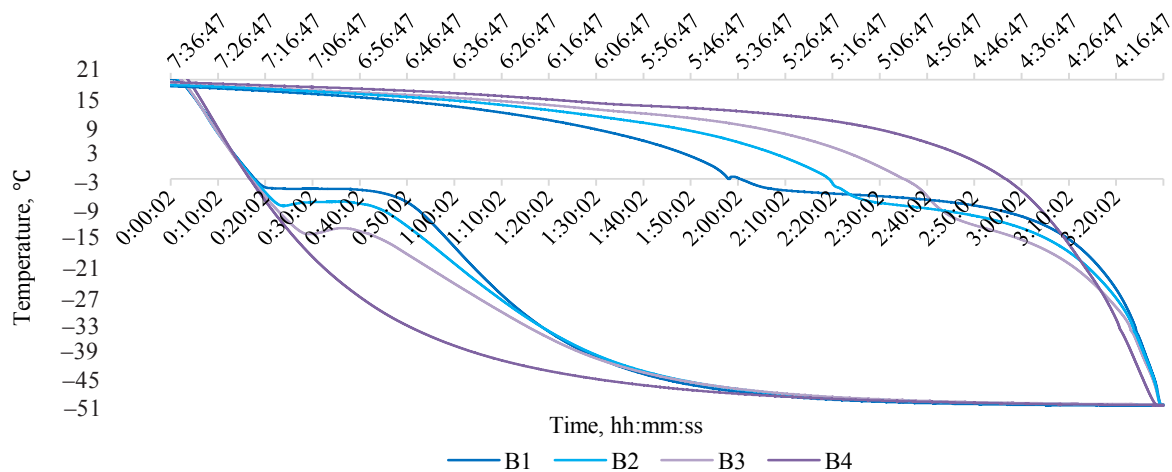Рисунок 4. Типовые кривые замерзания и размораживания сахарных растворов различной концентрации



Figure 5. Freezing and defrosting curves for sugar solutions at various concentrations

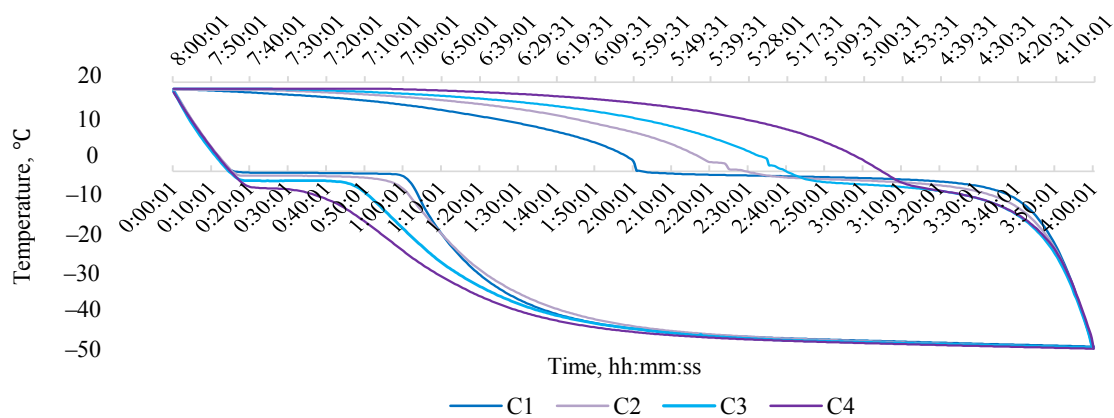Рисунок 5. Типовые кривые замерзания и размораживания сахарно-молочных растворов различной



Figure 6. Freezing and defrosting curves for milk solutions at various concentrations

Рисунок 6. Типовые кривые замерзания и размораживания молочных растворов различной концентрации
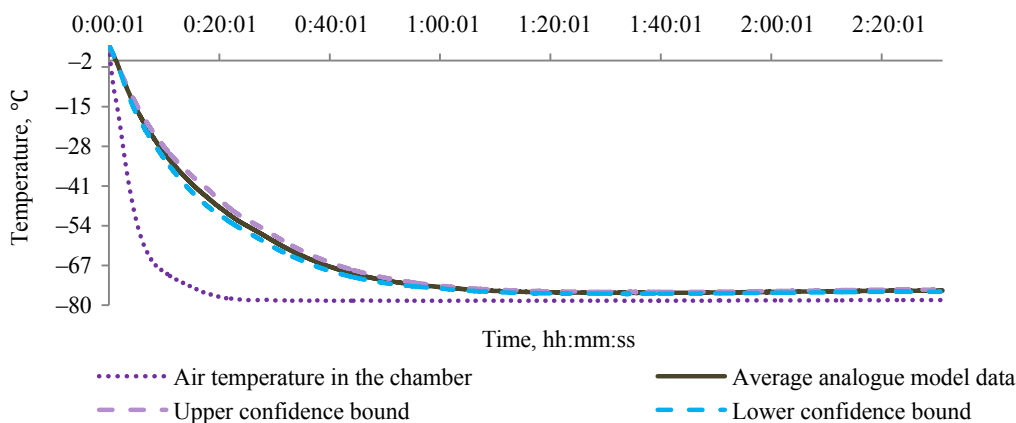
Figure 7. Thermograms of analogue models of sweetened condensed milk frozen with solid carbon dioxide

Рисунок 7. Термограммы моделей-аналогов сгущенного молока с сахаром при замораживании твердым диоксидом углерода



$$Y_1 = -8E{-}06x^3 - 0.0015x^2 + 0.0098x$$
$$R^2 = 0.9995$$

$$Y_2 = -7E{-}05x^3 + 0.0015x^2 - 0.0401x$$
$$R^2 = 0.997$$

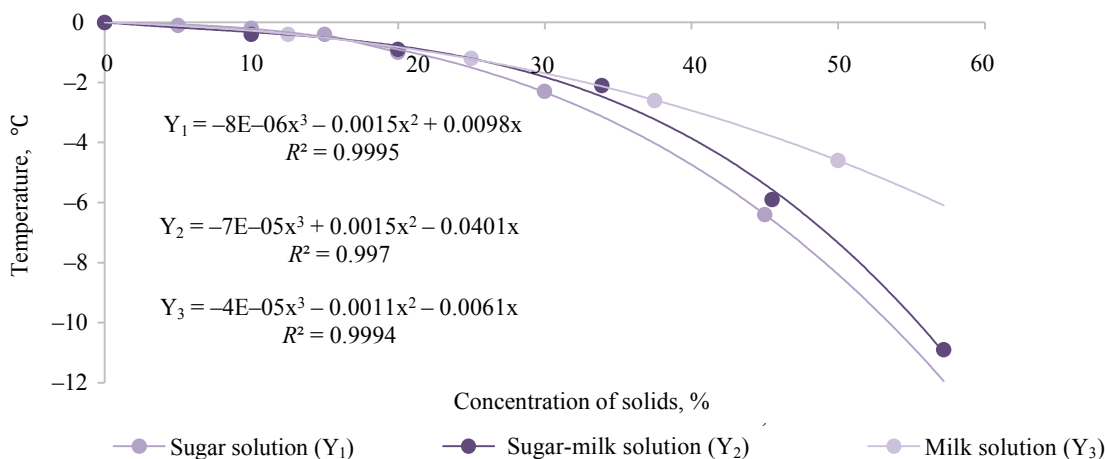$$Y_3 = -4E{-}05x^3 - 0.0011x^2 - 0.0061x$$
$$R^2 = 0.9994$$

Figure 8. Effect of concentration on cryoscopic temperature

Рисунок 8. Зависимость изменения криоскопической температуры от концентрации растворов

Figures 3, 4, and 5 visualize the typical freezing and defrosting curves for sugar, sugar-milk, and milk solutions, respectively. As the graphs show, when milk was introduced into the system, it reduced the water crystallization time, like in sugar solutions. The defrosting data were particularly remarkable. The phase transition time during defrosting was 2–2.5 times longer than during freezing. The defrosting time decreased after the milk component was introduced into the system, which resulted in a smoother phase transition. Thus, the milk component shortened the freezing/defrosting time. Probably, this phenomenon could be explained by the extra moisture-binding agents that entered the system, i.e., powdered milk components and, in particular, protein. Models A4 and B4 had a much faster defrosting rate.

As the previous stage revealed no phase transition, additional studies had to be performed. Solid carbon dioxide with a temperature of –78.5°C served as a refrigerant. However, this experiment also demonstrated no thermostatic plateaus typical for phase transitions (Fig. 7). The obtained results did not correspond to the data published by Pavlova in [14]. However, the multiple repetition of the experiment and the convergence of the values obtained made it possible to limit the scope of possible causal relationships to several options:

1. The rate of phase transition in this temperature range is less than one second. This value corresponds to the technical parameters of signal recorded by the device;

2. The technical parameters of the probes generate errors at the temperature range from –60 to –78.5°C;

3. Phase transitions occur at lower temperatures in complex polycomponent systems;

4. Diffusion dilemma.

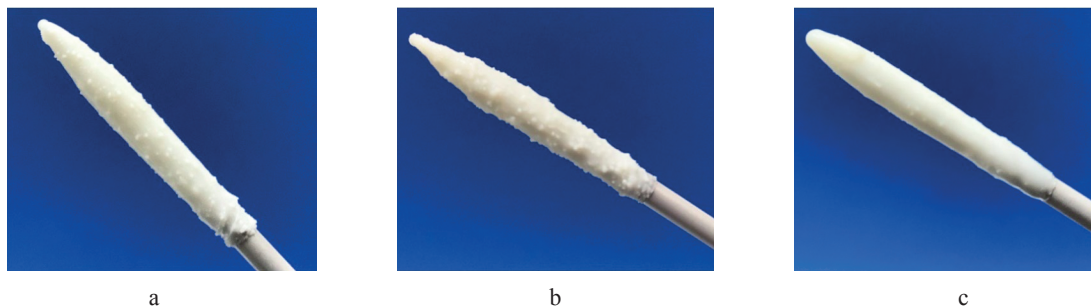Figure 9. Loss of fluidity after 54 min: a – at –30°C, b – at –32.5°C, c – at –35°C

Рисунок 9. Визуализация потери текучести исследуемых образцов моделей аналогов сгущенного молока с сахаром при хранении в течение 54 мин: a – при –30 °C; b – при –32,5 °C; c – при –35 °C



Lab: Mettler                                                                                          Stare$^e$ SW 15.00
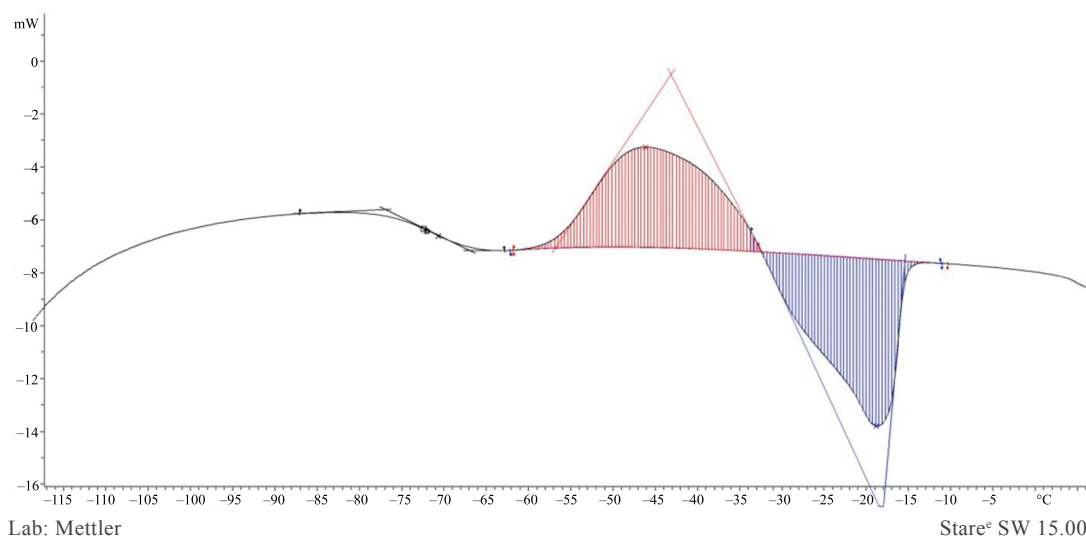
Figure 10. Differential scanning calorimetry of commercial sweetened condensed milk

Рисунок 10. Дифференциально сканирующая калориметрия промышленного образца сгущенного молока с сахаром

Figure 8 demonstrates the correlation between the effect of the concentration of solids on the dynamics of cryoscopic temperature in the solution. The dependences were non-linear, with three-power polynomials. Solutions with ≤ 20% solids showed no significant changes in the freezing point. Further increase in concentration led to significant changes related to the nature, concentration, and possible synergistic effects of the dissolved components.

Since the thermographic method failed to register the cryoscopic temperature of the analogue models, we decided to determine the temperature range when liquid turns solid. The loss of fluidity depended mostly on the ambient temperature and the storage time. At –30°C and ≥ 2 h of storage time, the effect was comparable to 54 min of storage at –35°C (Fig. 9). The appearance of crystal-like elements and the complete loss of fluidity under mechanical action were characteristic features of the structural change in the product. However, all samples thawed within a few minutes, regardless of temperature and storage time.

Figure 10 illustrates the differential scanning calorimetry of a commercial sweetened condensed milk (State Standard 3168). The solidification occurred at –82 ... –80°C. The pronounced crystallization and thawing peaks confirmed the heterogeneity of the system and the polycrystalline nature of the freezing process.

**Conclusion**

The research revealed some freezing/defrosting patterns of sugar, sugar-milk, and milk solutions, depending on the nature and concentration of the components dissolved. The cryoscopic temperature decreased following the increase in the concentration of solids. In sugar and sugar-milk systems, the freezing time and the phase transition period decreased as the concentration characteristics of the system increased. In whole milk solutions, the freezing time and phase

transition increased as the concentration rose from 12.5 to 25%. With a further increase in concentration, they decreased, which was probably due to the multicomponent composition of the system and the heat of crystallization. In sweetened condensed milk, the loss of fluidity occurred at –30°C if the storage time exceeded 2 h. This result was comparable to a 54-min storage at –35°C. The methods employed failed to establish the phase transitions in analogue models of sweetened condensed milk. The research created prerequisites for a more profound differential scanning calorimetry of phase transitions in sweetened condensed dairy products.

### Contribution

A.G. Galstyan and A.E. Ryabova developed the research concept and conducted a formal analysis. A.G. Galstyan developed the methodology and edited the manuscript. A.E. Ryabova and V.A. Tolmachev conducted the experiment and visualized the data. A.E. Ryabova drafted the manuscript. All the authors were involved in the study and agreed on the final version of the manuscript.

### Conflict of interest

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

### Критерии авторства

А. Г. Галстян и А. Е. Рябова разработали концепцию исследования и провели формальный анализ. А. Г. Галстян разработал методологию, рассмотрел и отредактировал рукопись. А. Е. Рябова и В. А. Толмачев провели эксперимент и визуализировали данные. А. Е. Рябова подготовила черновик рукописи. Все авторы были привлечены к исследованию, а также ознакомились и согласились с окончательным вариантом рукописи.

### Конфликт интересов

Авторы заявляют об отсутствии конфликта интересов.

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