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THEORETIC APPROACHES TO SUBSTANTIATE SHELF LIFE CAPACITY OF BUTTER AND SPREADS

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Abstract. The analysis of scientific information on the theoretical aspects and practical features of long-term freezing storage of butter and spreads is carried out. Means of mathematical modeling are substantiated, allowing prediction of the storage capacity of these foods based on the study of the mechanism and kinetics of the processes that determine the deterioration of quality (the set of relevant indicators) during storage. A detailed description of the proposed method for predicting the shelf life of butter and spreads using the full factorial experiment is given, which makes it possible to evaluate their storage capacity with sufficient reliability. A comparative assessment of the known physical, chemical and sensorial indicators affecting the storage capacity is given. The titratable acidity, acidity of the fat phase, degree of dispersion and distribution of moisture, and also the peroxide number were determined as basic storage stability factors.

Keywords: *butter, spreads, shelf life, quality factors, mathematical model, regression analysis, acidity, peroxide value.*

Introduction

The overall goal of the food industry consists in adequate providing all consumers with safe, affordable and nutritious foods. However, this problem cannot be solved only by increasing production volumes of the said products – a significant reduction in losses during the processing of food raw materials along the entire chain from field to table, a proper increase in the nutritional value of manufactured products, and an increase in their shelf life due to strict observance of safety requirements to food products and the full preservation of quality shall be also guaranteed [1, 2]. Industrial production of food products requires the correct application of scientifically based approaches to the storage of food products, that is, the possible suppression of their natural spoilage process. Of course, it is impossible to completely stop this process; however, changes in product quality during storage should occur in a predictable and controlled manner. This is facilitated by a significant improvement in the methods of preparation and processing of raw materials, packaging and distribution of food products [3]. Currently, research and development are held to create new packaging materials and food storage technologies, which are expected to positively affect compliance with food safety criteria [4] through the proper application of technologies for the obtaining, storage and processing of food materials using advanced scientific achievements and developments [5]. At the same time, it is important not only to comply with food safety criteria regarding production volumes, but also to ensure their compliance with the basics of healthy nutrition, quality and safety requirements through proper coordination of the profile activities of agriculture and the food industry [6].

Based on the above, it is important to comprehensively assess the requirements for the storage conditions of butter and spreads within the framework of market turnover and food reservations. This will allow developing rational approaches to the possible and appropriate consideration of the fundamentals of food security in technical regulation, in particular when developing standards of different levels and other regulatory documents.

Aiming at the full coverage of all segments of the solvent demand of consumers, the dairy industry, together with natural butter, has mastered the technology of producing recombinant butter using dried milk (whole and skimmed), as well as buttermilk. Despite the skepticism of adherents of healthy nutrition, the spreads, the raw materials for which are dehydrated milk fat, vegetable fats and dry buttermilk, find their numerous consumers. Technological schemes and production methods, the equipment used to obtain butter and spreads are similar. In the production of recombinant butter, only the initial operations associated with obtaining stable secondary milk-fat dispersions are somewhat different. The use of milk fat concentrates and skimmed milk powder in a certain way smoothes out the seasonality of production of natural butter [7].

Butter, as a concentrate of milk fat, contains phosphatides and unsaturated fatty acids such as linoleic, linolenic and arachidonic. Butter on storage, as a result of oxidative and hydrolytic reactions, accumulates by-products this causing deterioration in taste – especially when the butter is stored for a long time. Therefore, to slow down chemical processes and maintain product quality indicators during long-term storage, a number of measures are taken to establish the appropriate temperature regime, use effective packaging materials, etc. [8, 9]. F. Vyshemirskii, a well-known Russian expert in the field of butter production, noted [10] that the product should be reliably isolated from any external influences: light, microbiological, chemical, etc. At the same time, the packaging material must be environmentally friendly, neutral to the product itself and safe for human health. Butter does not have such a protective layer as cheese, and therefore requires proper packaging. Vegetable parchment paper is widely used for these purposes, this being a universal grease-resistant and moisture-resistant paper made from pure cellulose of coniferous and deciduous wood without the use of chemicals and moisture-resistant resins. The main requirements for parchment paper as a packaging material: sterility, environmental friendliness, resistance to high temperatures, chemical inertness, neutrality to the surface of the material, elasticity and flexibility, abrasion resistance, suitability for all types of printing, etc.

Regulatory regimes and practical features of long-term storage of butter and spreads

According to the Interstate Standard GOST 32261-2013 [11] this being in force in Russia and a number of CIS countries, long-term storage of butter is carried out in mode III (air temperature – minus (16 ± 2) ° C, relative humidity – not more than 90%) for 15 months, and provided that the temperature does not exceed minus 24 °C – within 24 months. According to the norms of the National Standard of Ukraine in force – DSTU 4399: 2005 [12] butter shall be stored at temperatures from 0 °C to −18 °C in freezers, the shelf life of the said is specified in the regulatory document to be from 3 to 12 months. Specialists of the Ukrainian State Research Institute of Nanobiotechnology and Resource Saving noted that during prolonged (for 24 months) storage of butter, the temperature regime is −25 ± 3 °C. Under the indicated storage mode, the product is packaged in a tight monolith of 24 kg in cardboard boxes previously covered with parchment paper [13], which is not provided for by the applicable national standard [12]. In this regard, in order to predict the shelf life and development of new packaging materials for butter and spreads with subsequent amendments to existing national standards, it is relevant to study sensorial and biochemical parameters when the said foods are stored in industrial freezers at −25 ± 3 °C. The long-term storage of a batch of butter weighing 20 MT, packaged in a tight monolith of 24 kg in cardboard boxes, previously covered with parchment paper was investigated [14]. Storage was carried out in an industrial freezer at a temperature of −25 ± 3 °C. Sensorial evaluation of the selected oil samples was carried out collectively by a 10-point evaluation system, and biochemical parameters: titratable acidity and fatty acidity were also determined. After storage of butter for 30 months, a decrease in sensorial values was observed: taste and aroma – 0.8 times, color – 0.7 times. The results of sensorial evaluation of butter after long-term storage are shown in Figure 1.

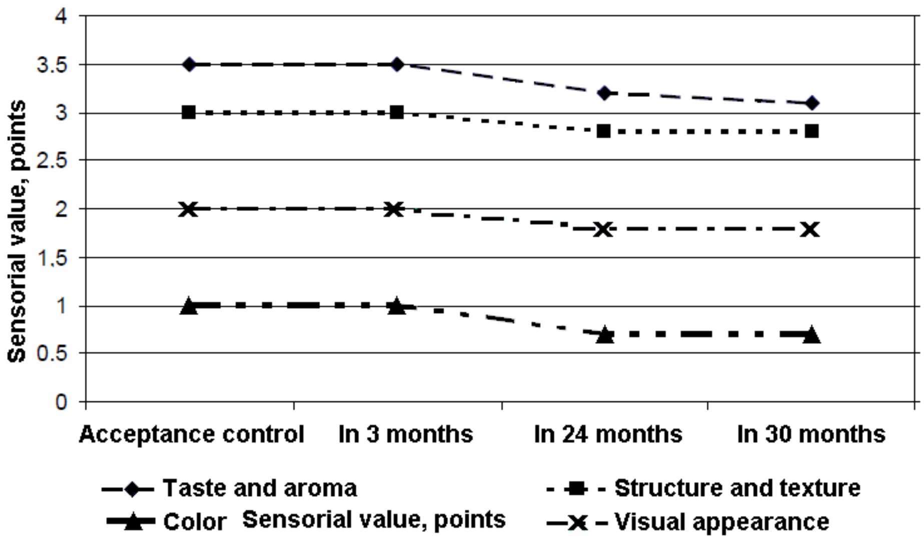

Figure 1. Results of sensorial evaluation of butter after long-term storage [14].

The study of biochemical parameters after long-term storage showed that the titratable acidity and the acidity of the fat phase increased 1.26 times and 1.32 times (Table 1). The indicated values correspond to the norms of the standard [12] in force, therefore there is every reason to assert that the temperature regime is acceptable −25 ± 3 °C in order to properly slow down biochemical processes in butter.

Table 1

Biochemical parameters of butter after long-term storage [14]

№№	Parameters	Storage duration			
		Control	3 months	24 months	30 months
1.	Acidity, °T (Degrees Turner)	15.9±0.8	15.9±0.7	16.7±0.8	20.00±0.9
2.	Acid fat phase, °K (Degrees Kettstofer)	1.79±0.08	1.80±0.09	2.00±0.1	2.37±0.1

The same long-term storage period was set for the "Gorodskoy" spread, stored at minus 25 °C. The quality of the product in transport and consumer packaging met the requirements of the regulatory documentation for it until the end of the planned shelf life (30 months). In [15] it is also indicated that experiments were started with long-term storage of spreads for 36 months.

The above information fully confirms the justification, both in terms of food safety and maintaining proper consumer properties, different temperature and humidity conditions and shelf life of butter and spreads. At the same time, full-scale studies of these foods and, accordingly, obtaining objective information about their storage capacity at present seem possible only in real time at the enterprises of the state reservation system. In this regard, substantiation of the algorithm for the numerical evaluation of food products, in particular butter and spreads, seems to be extremely expedient, based on their principal characteristics thereof.

Development of an algorithm for determining the compliance of the shelf life of a food product with regulatory requirements

In general, the shelf life of food products is the period of time during which they meet regulatory or other current requirements for sensorial, physical and chemical indicators, nutritional value, content of chemical and biological substances and their compounds, microorganisms and other biological objects. In another way, the shelf life of food products can be defined as the period of time during which the products retain the properties established in the regulatory and technical documentation. The vast majority of food products are complex multi-component biological and biochemical systems in which, as a rule, various microbiological, biochemical and physicochemical reactions take place – it is clear that their shelf life and quality retention duration depend on these and many other factors [16].

Mathematical modeling tools allow predicting the state of the object of study during storage under standard conditions, using the results obtained during storage under extreme conditions [16]. The indicated concept is based on the study of the mechanism and kinetics of processes that determine the deterioration of the quality (set of relevant indicators) of the test object during storage. Thus, for various food products, methods of mathematical modeling can be used, in particular, constructing and evaluating the quality of a regression model based on mathematical planning and processing the results of an active experiment [17, 18].

To establish a mathematical relationship between the shelf life of certain food products and factors influencing the said shelf life, a complete factor two-level experiment (CFE 2^n), including $N = 2^n$ experiments, is used [19]. In different experiments, within the framework of the experiment, all possible combinations of the levels of n factors affecting the response are fulfilled, and the response function is the value Y, which takes values y_1, y_2, y_3, … and determines the number of days during which the product properties do not go beyond the limits of the numerical ranges within which it can be considered usable. In other words, expiration is the acquisition by a product of such physical, chemical and sensorial characteristics that make its consumption impossible. First of all, these factors include the storage mode (under ordinary conditions, in cold room, in the frozen state) of food products and specific temperature conditions. For the proper execution of the experiment and the corresponding mathematical processing of its results, it is necessary that the factors are controllable, operational, unambiguous, compatible, independent, and the accuracy of the measurements is sufficient. The above requirement – controllability – means that the selected value (level) of the factor can be kept constant while the experiment continues. Another requirement is that the factor's operational requirement is defined as the sequence of operations by which its specific values (levels) are established. The choice of dimension and the accuracy of fixation of this factor are also associated with this definition. The uniqueness of a factor is the directness of its influence on the response, that is, the factor should not be a function of other factors. Proper factor compatibility means that all of their combinations are workable and safe. Proper independence of factors means the absence of a correlation between them.

For example, in the course of the study it was established that for this particular product there are a combination of 4 factors corresponding to the conditions listed above. One of the prerequisites for the application of regression analysis is the distribution of the modeled value according to the normal law, for example, with a probability of 95%. Then the predicted shelf life of the food product is determined from the regression equation – an incomplete fourth order polynomial.

$$
\begin{gathered}
y=b_0+b_1X_1+b_2X_2+b_3X_3+b_4X_4+b_{12}X_1X_2+b_{13}X_1X_3+b_{14}X_1X_4+ \\
+b_{23}X_2X_3+b_{24}X_2X_4+b_{34}X_3X_4+b_{123}X_1X_2X_3+b_{124}X_1X_2X_4+ \\
b_{134}X_1X_3X_4+b_{234}X_2X_3X_4+b_{1234}X_1X_2X_3X_4
\end{gathered}
\tag{1}
$$

To determine the unknown coefficients of equation Eq.(1), a complete experiment of two factors with uniform five-fold duplication of experiments in each row of the experimental design can be implemented. The basic characteristics of this plan are given in Table 2.

Table 2

Basic characteristics of experimental plan

Characteristics	Factors X_j			
	X_1	X_2	X_3	X_4
Basic level (experimental center)	a_1	c_1	d_1	e_1
Variation interval Δ_j	a_2	c_2	d_2	e_2
Lower level	a_3	c_3	d_3	e_3
Upper level	a_4	c_4	d_4	e_4
Notations of encoded factors	x_1	x_2	d_3	e_4

Based on the conditions set, the number of experiments in this experiment is $N = 2^4 = 16$. In order to move from the actual values of factors to encoded dimensionless quantities, the formula is used:

$$
x_j = \frac{X_j - X_j^0}{\Delta X_j}
\tag{2}
$$

where: x_j – encoded value of a factor;
X_j – present value of a factor;

X_j^0 – value of basic level;

ΔX_i – variation interval.

Thus, for each factor, the encoded value of the lower level corresponds to (-1), and the upper level corresponds to 1. Table 6 shows the experimental design matrix.

Table 3

Experiment design matrix

i	X_1	X_2	X_3	X_4	x_1	x_2	x_3	x_4
1	a_3	c_3	e_3	d_3	-1	-1	-1	-1
2	a_3	c_3	e_3	d_4	-1	-1	-1	1
3	a_3	c_3	e_4	d_3	-1	-1	1	-1
4	a_3	c_3	e_4	d_4	-1	-1	1	-1
5	a_3	c_4	e_3	d_3	-1	1	-1	-1
6	a_3	c_4	e_3	d_4	-1	1	-1	1
7	a_3	c_4	e_4	d_3	-1	1	1	-1
8	a_3	c_4	e_4	d_4	-1	1	1	1
9	a_4	c_3	e_3	d_3	1	-1	-1	-1
10	a_4	c_3	e_3	d_4	1	-1	-1	1
11	a_4	c_3	e_4	d_3	1	-1	1	-1
12	a_4	c_3	e_4	d_4	1	-1	1	1
13	a_4	c_4	e_3	d_3	1	1	-1	-1
14	a_4	c_4	e_3	d_4	1	1	-1	1
15	a_4	c_4	e_4	d_3	1	1	1	-1
16	a_4	c_4	e_4	d_4	1	1	1	1

As the next step, coefficients of the regression equation are to be found, where the variables are encoded values of the factors:

$$\begin{aligned} y= &\tilde{b}_0+\tilde{b}_1 x_1+\tilde{b}_2 x_2+\tilde{b}_3 x_3+\tilde{b}_4 x_4+\tilde{b}_{12} x_1 x_2+\tilde{b}_{13} x_1 x_3+\tilde{b}_{14} x_1 x_4+ \\ &+\tilde{b}_{23} x_2 x_3+\tilde{b}_{24} x_2 x_4+\tilde{b}_{34} x_3 x_4+\tilde{b}_{123} x_1 x_2 x_3+\tilde{b}_{124} x_1 x_2 x+ \\ &\tilde{b}_{134} x_1 x_3 x_4+\tilde{b}_{234} x_2 x_3 x_4+\tilde{b}_{1234} x_1 x_2 x_3 x_4 \end{aligned} \quad (3)$$

In Eq. (3), the coefficients of linear terms indicate the significance of the influence of factors: the larger the numerical value of the coefficient, the more this factor influences the shelf life. In the case of a positive value of the coefficient, with an increase in the value of the factor, the shelf life increases, otherwise it decreases. The coefficients available for the products x_j determine the influences of the interaction of factors. For example, if the coefficient when producing two encoded factors is positive, then to increase the shelf life of the product, it is necessary to simultaneously increase or decrease the values of these factors. Accordingly, a reduction in the shelf life of a food product is accompanied by changes in factors in different directions. If the coefficient when producing two encoded factors is negative, then in order to increase the shelf life, factors must change in different directions, and to reduce, a simultaneous increase or decrease in the values of factors is required. Similarly, the signs of higher order interaction influences are interpreted.

Unknown coefficients for the dimensionless variables x_1, x_2, x_3, x_4 are found from the formulas below.

$$\tilde{b}_0 = \frac{\sum_{i=1}^{16} \overline{y}_i}{16} \tag{4}$$

$$\tilde{b}_{ju} = \frac{\sum_{i=16}^{16} x_{ij} \cdot y_i}{16}, j = 1,2,3,4 \tag{5}$$

$$\tilde{b}_{ju} = \frac{\sum_{i=1}^{16} x_{ij} \cdot x_{iu} \cdot \overline{y}_i}{16}, j < u, j,u = 1,2,3,4 \tag{6}$$

$$\tilde{b}_{juv} = \frac{\sum_{i=1}^{16} x_{ij} \cdot x_{iu} \cdot x_{iv} \cdot \overline{y}_i}{16}, j < u < v, j,u,v = 1,2,3,4 \tag{7}$$

$$\tilde{b}_{1234} = \frac{\sum_{i=1}^{16} x_{i1} \cdot x_{i2} \cdot x_{i4} \cdot \overline{y}_i}{16}, \tag{8}$$

where: $\overline{y}_i$ – average values of shelf life calculated from the results of 5 replications of i series of experiments;

x_{i1}, x_{i2}, x_{i3}, x_{i4} – encoded values of the factors not complying with i series of experiments.

If the measured random variable (expiration date) is distributed in accordance with the normal law in the entire studied range, then regardless of the average values $\overline{y}_i$ obtained in 5 replicates of each of the 16 series of experiments, the dispersion of this quantity, called the dispersion of reproducibility, will not change its quantities. Thus, line estimates of the indicated variance in different series of experiments should be homogeneous.

In order to test the hypothesis of homogeneity of dispersions s_i^2, the Cochran statistical test is used. If in accordance with it there is no reason to abandon the hypothesis of homogeneity of row-wise dispersion estimates, the experiments are considered reproducible.

In the case when the experiments are not reproducible, they try to achieve reproducibility by identifying and eliminating the causes of instability of the experiment, or by using more accurate methods and means of measurement. So, if among m replicates of a series of experiments there are results significantly different from other results of the same series, significant errors can be determined using the statistical *r*-test of the largest deviation. The result of such a check should be excluded from subsequent analysis. If there are significant errors, an additional experiment (or a series of experiments) is performed, ensuring that the experiment is carried out at $m_j = m$ = const.

Considering that the determination of the shelf life of experimental samples in each experiment is carried out with a certain error, the coefficients of the regression equation will be determined in accordance with the found error. The purpose of statistical analysis of the equation is to show with a predetermined probability that the obtained estimates of the coefficients of the equation are either greater or less than the error in their measurement. In the first case, they significantly differ from zero, in the second case – insignificantly, that is, they should be excluded from the equation.

In order to check the significance of the coefficients of the regression equation, the statistical Student's test is used. Components with negligible coefficients are excluded from the equation. The reason for obtaining an insignificant linear effect of any factor may be one of the following situations:

- a specific factor does not affect the expiration date;
- the variation interval is too small, and therefore the response change due to the change in the factor is proportional to random deviations caused by the influence of unaccounted factors;
- the value of a specific factor at the central point of the experiment (Table 3) corresponds to its optimal value; therefore, its equal increase or decrease by Δ_j will decrease the response values by approximately the same amount.

If, after checking the significance of the equation's coefficients, all N coefficients remain, then checking the adequacy of the model does not make sense, since the value of the expiration date calculated for such an equation for the conditions of any i^{th} series of experiments should coincide, within the limits of rounding accuracy, with the value accepted for calculation.

If the number of significant coefficients is at least one less than the number of series of experiments, it becomes necessary to statistically verify the adequacy of the obtained equation to experimental data. This verification is carried out using the Fisher test. The discovered inadequacy of the model can be due to both an unreasonably accurate description of the experimental data by the indicated equation, and the fact that the accuracy of the process description is significantly lower than the accuracy with which the experimental data were obtained. In the first case, the equation can serve as the basis for searching for optimal conditions, but cannot be used to test one or another hypothesis about the mechanism of the process under study.

To make it possible to determine the shelf life of a food product using the obtained regression equation in the case when the factors acquire real values, the encoded factors should be transformed according to the formula Eq. (1). In this case, the regression coefficients will change. At the same time, the interpretation of the influence of factors on the values and signs of the regression coefficients is excluded, since, due to circumstances due to the properties of the experimental design matrix, the coefficients of the equation will be determined depending on each other. However, upon receipt of the interpolation formula, this technique can be used. That is why, replacing the variables X_1, X_2, X_3, X_4 in the regression equation found, taking into account the data of Table 3, by the quantities $\frac{X_1 - a_1}{a_2}$, $\frac{X_2 - c_1}{c_2}$, $\frac{X_3 - d_1}{d_2}$ and $\frac{X_4 - e_1}{e_2}$ accordingly, an interpolation formula is obtained that allows, for specific values of the initial storage conditions of the food product to be stored, to predict the duration of its storage without violating the normative indicators of food safety and quality.

Justification of the main characteristics of the experiment plan for predicting the storage capacity of butter and spreads

Storage issues of butter have long been the subject of interest of scientists and experts in the food industry. For example, in the well-known work [20], such criteria for the shelf life of butter after storage (without freezing) are indicated, such as the degree of oxidation of fats, bacterial contamination and sensorial characteristics. It is clear that with the widespread introduction of freezing storage of food, including dairy and milk-containing fat products, approaches to ensuring and controlling their storage capacity have changed significantly. Fresh or chilled foods usually have similar spoilage factors – primarily microbial spoilage. Therefore, it is relatively easy to simulate temperature changes in the product and present models for the development and inhibition of the growth of microorganisms at known temperatures. As a result, it is possible to predict with satisfactory accuracy when the microbial load exceeds the safe limit, thereby determining the threshold safe value of the expiration date [21]. The situation with frozen foods is not so simple, so the search for adequate approaches to determining the expiration date continues. For example, in works [22, 23] introducing a technique based on the "fingerprinting kinetics" is recommended. The said technique allows giving a realistic estimate of shelf life while using several markers that are potentially associated with quality deterioration factors. To determine the expiration date, it is necessary to check the presence of key differences in the quality of food products during storage, to determine markers in terms of their significance in relation to the dynamics of spoilage, and use multivariate analysis to link selected markers with specific processes occurring in food products. This can help in: a) determining the appropriate quality attributes by reducing the number of markers needed to effectively predict shelf life, b) understanding the combination of factors that determine, along with individual factors, the shelf life, c) understanding the general mechanics of product quality deterioration. Prediction of shelf life is based on the fundamental principles of modeling the processes of loss of quality of a food product – first of all, on the kinetic modeling of various, well-studied, deterioration mechanisms in food systems. It is the models of multivariate analysis of variance and regression analysis using the methods of mathematical design of the experiment that can simultaneously take into account microbiological, sensorial, physical and chemical indicators in determining the shelf life of food products [24].

In the case, the task of predicting the periods of freezing storage of butter and spreads at the first stage is reduced to justifying the nomenclature of factors X_1 ... X_4, the most significant in the sense of determining the parameters that determine the quality of these products after specific periods of stay on the reservation.

In studies, the results of which are presented in [14], titratable acidity (°T), fatty acidity (°K) and sensorial indicators determined on a ten-point scale are used as criteria for the quality of butter after long-term storage: 1) taste and aroma; 2) color; 3) structure and consistency; 4) appearance. If it is entirely possible to agree with the use of the first two (objective instrumental) criteria, the appropriateness of using sensorial indicators raises certain doubts. Although there is an opinion about sensorial evaluation as an indispensable step in determining the quality of complex and variable food systems, the disadvantages inherent in sensorial methods are also well known. Firstly, serious sensorial research is quite laborious and expensive. And secondly, assessing the stability and quality of food products is impossible without the use of numerous analytical methods for detecting signs of hydrolysis and oxidation of [25].

The authors of [26] use in some way different nomenclature of indicators of quality criteria for butter after long-term storage. In addition to the titratable acidity (°T) and fatty acidity (°K) mentioned above, an indicator of thermal stability (units), as well as the degree of dispersion and distribution of moisture (classes I – III) are used. In our opinion, the thermal stability index is not a determining factor in the storage capacity of butter and spreads, but the degree of dispersion and distribution of moisture is an indicator that correlates well with the physicochemical properties of fat products when they are stored, as well as with the temperature regime at which this storage is carried out.

The most important indicator of lipid oxidation of fatty products is their peroxide number (mol / kg). There are recognized international methods for determining this indicator – both in the CIS regulatory system and in the national regulatory framework of Ukraine, the relevant standards of the International Organization for Standardization ISO are harmonized, allowing determination of the peroxide number with reasonable accuracy. The use of these standard methods, to some extent, is limited by the lack of norms regarding the peroxide value in the current technical specifications for oil and spreads, however, in the conditions of voluntary application of the standards, any reliable databases obtained by scientists and practitioners of dairy production can be used.

Thus, we consider it appropriate to use the following factors when calculating the storage capacity according to the above methodology:

X_1 – titratable acidity, °T;

X_2 – acidity of fat phase, °K;

X_3 – degree of dispersion and distribution of moisture, classes I - III;

X_4 – peroxide number, mol/ kg.

Having determined, as a result of practical experiments, the values of these variables, we can obtain an interpolation formula with which we can reliably predict the storage time of butter and spreads without violating the threshold values of food safety and quality of these products.

Conclusion

The determination of the reliable storage capacity of butter and spreads is of great economic importance, since these products are laid for long-term freezing storage. Mathematical modeling tools allows predicting the shelf-life of food products based on the study of the mechanism and kinetics of processes that determine the deterioration in the quality (set of relevant indicators) of butter and spreads during storage. The proposed method for predicting the shelf life of these fat products on the basis of a complete factorial experiment makes it possible to evaluate their storage capacity with sufficient reliability. The titratable acidity, acidity of the fat phase, degree of dispersion and distribution of moisture, and also the peroxide number were determined as basic storage factors.

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