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INVESTIGATION OF THE PROPERTIES OF MARMALADE WITH PLANT CRYOADDITIVES DURING STORAGE

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Abstract. This article presents the results of studies of the properties of marmalade with natural plant cryoadditives during storage for 3 months (90 days). To improve the organoleptic characteristics and antioxidant properties of marmalade, plant additives of cryogenic origin were used, namely cryopastes from quince, apple, carrot, pumpkin, grapes, and cryopowders from rose hips, sea buckthorn, and grapes. It is shown that the use of plant cryoadditives makes it possible to increase the biological value and antioxidant properties of new types of marmalade. For new types of marmalade with cryoadditives, during its shelf life, these properties have not been studied earlier. However, it should be noted that such studies are necessary and relevant for solving the problem of preserving the quality indicators and antioxidant capacity of marmalade with cryoadditives. New kinds of marmalade have high organoleptic characteristics during the whole storage period. It is shown that the storage conditions of marmalade, according to current regulatory documentation, are suitable for new marmalade products, since the physico-chemical and microbiological indicators vary, but remain within acceptable limits. Thus, the moisture content decreases to almost 31%, the acidity rises by 12–16%, compared to freshly prepared samples. The content of reducing substances increases by 30–64%. It is determined that the value of the antioxidant capacity of all the samples developed is much higher – almost 2.3–8 times than the same figure for the control sample. After the storage period, the antioxidant capacity is reduced by 15–35%, but this data also significantly exceeds the antioxidant capacity of the control sample. Storage of marmalade with cryopastes and cryopowders for 90 days is possible, since all the basic physico-chemical, microbiological, and organoleptic indicators at the end of shelf life meet the requirements. Indicators of antioxidant capacity for them also remain at a fairly high level. New technologies of jelly-fruit marmalade with plant cryoadditives have been introduced at 4 enterprises. 2 utility model patents have been received, and regulatory documents have been developed and approved for the manufacture of new jelly products.

Keywords: marmalade, plant additives, cryogenic technology, cryopowder, cryopaste, antioxidant capacity.

ДОСЛІДЖЕННЯ ВЛАСТИВОСТЕЙ МАРМЕЛАДУ З РОСЛИННИМИ КРІОДОБАВКАМИ ПІД ЧАС ЗБЕРІГАННЯ

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Анотація. У статті наведено результати досліджень властивостей мармеладу з натуральними рослинними кріодобавками під час зберігання протягом 3 місяців. Для підвищення органолептичних показників та антиоксидантних властивостей мармеладу використовували рослинні добавки, отримані за низькотемпературними технологіями, а саме кріопасти з айви, яблук, моркви, гарбуза, винограду та кріопорошки з шипшини, обліпихи та винограду. Нові види мармеладу мають високі органолептичні показники протягом терміну зберігання. Встановлено, що величина антиоксидантної ємності нових видів мармеладу вище у 2,3–8 разів порівняно з контролем. Зберігання мармеладу з кріопастами та кріопорошками протягом 90 діб є можливим, оскільки всі основні фізико-хімічні, мікробіологічні та органолептичні показники наприкінці терміну зберігання відповідають вимогам та показник їх антиоксидантної ємності залишається на досить високому рівні.

Ключові слова: мармелад, рослинні добавки, кріогенна технологія, кріопорошок, кріопаста, антиоксидантна ємність.

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Introduction. Formulation of the problem

Marmalade is a jelly-like confectionery made from fruit and berry purée or an aqueous solution of gelling agents, sugar, and other ingredients. Depending on manufacturing techniques and on a structure-forming basis, marmalade is subdivided into two main types: fruit and jelly. The gelatinizer for fruit marmalade is pectin which is contained in fruit and berry purée (apple, plum, apricot, etc.). To produce jelly or jelly-

fruit marmalade, agar, agaroid, and pectins are used as gelatinizers [1].

Dyes and flavouring agents, usually of artificial origin, are included in formulae to provide high organoleptic parameters of marmalade. These components make the finished product look, taste, and smell attractive. But the nutritional value of most kinds of marmalade, unfortunately, remains quite low. Now much is known about the negative effect of artificial nutritional supplements on a person's health [2]. In work [3], it is shown that synthetic dyes used in production of foodstuff slow down the growth of nervous cells. To make fruit or jelly-fruit marmalade, fruit or berry purée, or precooked fruit and berry mass is used. But usually, these components have already passed the sulphitation stage and heat treatment for better crushing (grinding) quality, that is why their biological value is considerably decreased. This is a good reason for developing new types of marmalade with the use of plant additives obtained by means of cryogenic technologies. Moreover, the main attention should be paid to plant raw materials of domestic origin, which, after cryogenic treatment, can be used to give food products colour, odour, taste, antioxidant properties, as well as increase their biological value. It was found that, during cryogenic grinding of plant raw materials at a temperature of –10°C or lower, there is an increase in the concentration of biologically active substances as compared to their concentration in the initial raw material [4]. But we must take into account that natural food additives are usually unstable, and degrade in the process of storage of the finished product. That is why it is relevant to study organoleptic, physico-chemical, microbiological indices and antioxidant properties of new types of marmalade with plant pastes and powders obtained by cryotechnologies during the storage period.

Analysis of recent research and publications

Recently, scientists have developed a number of new technologies of marmalade using plant raw materials or products of their processing. For example, to enrich marmalade with vitamins and biologically active substances, persimmon [5], red currant [6], and honey [7] are added into it. To increase the content of vitamin C, foreign scientists have developed marmalade technologies with the addition of banana, ginger [8], and *Sonneratia apetala* fruits of the Lythraceae family [9]. To enrich the products with β-carotene, pumpkin purée and wild sweet ashberry [10], or the pulp of the fruits of the 'prickly pear cactus' (Indian fig opuntia) [11] are added. Adding the pulp of a cactus of the genus Stenocereus graysus (better known as pitaya) [12] makes it possible to increase the content of iron, calcium, and phosphorus, as well as vitamins B, C, and E. Marmalade with the grumichama (Brazil cherry) pulp added contains a lot of useful substances: provitamin A, B1, B2, C, iron, phosphorus, and calcium [13]. There is another technology when aloe vera and lime purée is added to marmalade, thus allowing the use of jelly products in therapeutic and prophylactic nutrition [14], etc. Nowadays, in the food industry, the use of pastes and fine powders of fruits, berries, vegetables obtained by the cryogenic technology is very promising. The possibilities have been studied of using multicoloured powders of carotene, chlorophyll, and anthocyanin nature, obtained by means of cryogenic grinding, in the technology of jelly marmalade production [15]. New types of marmalade with cryopastes from quince, apples, carrots, pumpkins, grapes, and with cryopowders from rosehips, sea buckthorn, and grapes have been developed [16]. A number of studies have been carried out aimed at developing the technology and research of organoleptic, physico-chemical, microbiological, antioxidant properties of jelly-fruit marmalade with cryopastes and cryopowders of plant origin [17,18]. It is shown that the use of plant cryoadditives makes it possible to increase the biological value and antioxidant properties of marmalade. These properties of new types of marmalade with cryoadditives during shelf life have not been studied yet, while it is necessary and relevant to solve the problem of preserving the quality indicators and antioxidant capacity of marmalade with cryoadditives.

This work was aimed at investigating the organoleptic, physico-chemical, microbiological, and antioxidant properties of marmalade with cryopastes and cryopowders during shelf life and determining the optimal storage conditions. To achieve this goal, it was necessary to solve the following tasks:

– to determine the organoleptic, physico-chemical and microbiological quality indicators of marmalade with plant cryopastes and cryopowders during shelf life;
– to determine the antioxidant capacity of marmalade with herbal cryopastes and cryopowders during storage.

Research Materials and Methods

Cryoadditives are pastes or powders obtained with the use of cryogenic technologies. Technologies for obtaining cryopaste from quince, apples, carrots, pumpkins, and grapes are developed by KhSUFTT scientists. This technology includes freezing of raw materials at a temperature of -35 – -70°C and low-temperature grinding (resulting in particles with sizes of 10–50 μm), without using any chemical stabilizers [4]. Cryopowders from grapes, rosehips, and sea-buckthorn are produced industrially (LTD "Cryocon", Kiev). The cryopowder production technology includes cryogenic grinding of raw materials at a temperature of -80 – -15°C into particles with a size ranging 10–20 μm to 2–3 mm (depending on the type), cryosublimational fractionation, and low-temperature extraction of oil by liquefied gases (TI U 10.3-

38648982-001: 2013. Powders of cryosublimated vegetable, fruit and berry and vegetable raw material).

In order to broaden the assortment, improve the chemical composition, and increase the biological value of jelly products, 12 marmalade formulae using plant cryoadditives were developed. The traditional recipe includes sugar, molasses, fruit purée, apple pectin, sodium lactate, citric acid, and an essence [19]. The formulations developed by us do not contain synthetic essences. Purée is replaced with cryopaste. The content of gelatinizers is reduced by 20%, citric acid by 5–40% (depending on the type of the additive), sugar by 5% (with the addition of cryopowders). Cryoadditives are added both separately and in combinations. On studying the chemical composition of additives and products, and the strength and organoleptic characteristics of marmalade, rational dosages of cryoadditives are determined. Cryopastes are added in an amount of 10–20% of the total mass of the system, cryopowders, 1.5% (Table 1).

Table 1. Investigated marmalade samples

Abbreviation for a marmalade sample	Marmalade sample
MQ-1	Marmalade with quince cryopaste (10%)
MA-2	Marmalade with apple cryopaste (10%)
MC-3	Marmalade with carrot cryopaste (10%)
MP-4	Marmalade with pumpkin cryopaste (10%)
MG-5	Marmalade with grape cryopaste (10%)
MAC-6	Marmalade with apple (10%) and carrot cryopastes (20%)
MQP-7	Marmalade with quince (10%) and pumpkin (20%) cryopastes
MQRH-8	Marmalade with quince cryopaste (10%) and rose hips cryopowder (1.5%)
MARH-9	Marmalade with apple cryopaste (10%) and rose hips cryopowder (1.5%)
MCSB-10	Marmalade with carrot cryopaste (10%) and sea buckthorn cryopowder (1.5%)
MPSB-11	Marmalade with pumpkin cryopaste (10%) and sea buckthorn cryopowder (1.5%)
MGG-12	Marmalade with grape cryopaste (10%) and cryopowder (1.5%)

The mass fraction of moisture of the marmalade was determined by the refractometric method in compliance with the State Standard GOST 5900-2014, titrated acidity according to State Standart DSTU 5024: 2008, the content of reducing agents was determined by the ferricyanide method according to State Standart GOST 5903-89; the bromine antioxidant capacity was determined by the method of galvanostatic coulometry, based on the use of electrogenerated titrants, such as bromine [20].

Results of the research and their discussion

Organoleptic, physico-chemical and microbiological quality indicators of marmalade with cryoadditives have been studied. As a control, pectin-based jelly-fruit marmalade was used, with fruit purée added [19]. According to the requirements of regulatory documentation (State Standart GOST 4333-2004), marmalade should be stored in plastic films and cardboard boxes, in clean, well-ventilated rooms, at a temperature of no higher than 18°C and relative humidity of air no more than 75–80%, without exposing to direct sunlight. Storing goods with a sharp smell close to marmalade is undesirable.

The results of determining the organoleptic quality indices of jelly-fruit marmalade with cryopastes are given in Table 2, with cryopastes and cryopowders, in Table 3.

It has been established that during the storage of marmalade for two months, the organoleptic parameters of all samples remain the same as those of freshly prepared marmalade samples. They have the correct shape with a clear contour and no deformation, jelly-like consistency. The taste and smell of marmalade are clearly expressed. The samples retain a saturated colour (yellow, orange, violet, etc.), typical of the added fruit and vegetable additives.

After three months of storage, the marmalade also retains the taste and smell of fruits and vegetables, the correct form, gelatinous consistency, and can be cut with a knife. The colour of the products corresponds to the additives, but somewhat loses its brightness and saturation.

It is known that while storing marmalade products, the process of internal diffusion of the dispersion medium (an aqueous solution of sugar, molasses and acids) from the centre of marmalade to the outer layer is intensified. In the outer layer, a lot of sucrose molecules are concentrated. As a result, a crystalline crust is formed on the surface, and the moisture content in the marmalade products is correspondingly reduced by removing moisture. An excess of moisture in jelly products can affect their shape and cause the formation of a sticky surface. This, in turn, will affect the accumulation of total acidity and acid hydrolysis of sucrose, with the formation of reducing substances.

The measure of acidity and the content of reducing substances also significantly affect the shelf life of the marmalade products. With an increase in the concentration of reducing substances, the products can get overmoistened. And vice versa, a decrease in the content of reducing substances can lead to drying out (sugaring) of the products. That is why the physico-chemical quality indicators of marmalade with plant cryoadditives were studied during the shelf life. According to the requirements of the State Standart DSTU, the mass fraction of moisture should range within 15–24%, the acidity index should be within 7.5–22.5 degrees, the mass fraction of reducing substances should not exceed 28%.

Table 2. Organoleptic quality indices of jelly-fruit marmalade with cryopastes

Name of the parameter	Characteristics of marmalade							
	Control sample	MQ-1	MA-2	MC-3	MP-4	MG-5	MAC-6	MQP-7
Appearance	Correct shape, a clear contour, no deformation							
Consistence	Jellylike consistence. The inner surface of the marmalade sample is opaque when broken. No inclusions							
Taste and smell	Light fruit odour and taste. No foreign smell and taste			Light odour and taste of vegetables. No foreign smell and taste		Strong smell and sour taste of grapes. No foreign smell and taste	Light odour and taste of fruit and vegetables. No foreign smell and taste	
Colour	Light yellow			Orange	Light orange	Violet	Light orange	Orange

Table 3. Organoleptic quality indices of jelly-fruit marmalade with cryopastes and cryopowders

Name of the parameter	Characteristics of marmalade					
	Control sample	MQRH-8	MARH-9	MCSB-10	MPSB-11	MGG-12
Appearance	Correct shape, a clear contour, no deformation					
Consistence	Jellylike consistence. The inner surface of the marmalade sample is opaque when broken. No inclusions	Jellylike consistence. The inner surface of the marmalade sample is opaque when broken. With inclusions of cryopowder				Jellylike consistence. The inner surface of the marmalade sample is opaque when broken. No inclusions
Taste and smell	Light fruit odour and taste. No foreign smell and taste			Light odour and taste of vegetables. No foreign smell and taste		Strong smell and sour taste of grapes. No foreign smell and taste
Colour	Light yellow			Orange	Light orange	Bright violet

The results of determining physico-chemical indicators of quality of new types of marmalade and the control are given in Table 4.

From the data in Table 4, one can see that the physical and chemical characteristics of marmalade vary slightly during storage. The change in properties during storage is due to a whole complex of microbiological and physico-chemical processes. The transformations are a result of the hydrolysis of the hydrocarbon component, redox reactions, condensation, polymerization. Besides, the change in properties during storage depends on the content of free and bound moisture in the sample at the beginning and during this time. The mass fraction of moisture in the samples decreases, on the average, by 31% compared to the initial values. The loss of moisture is explained by the free moisture present in the samples, which, due to diffusion, is gradually released during the storage period. The greatest losses of moisture – almost 37%, compared to the initial value – are observed in the sample "control", which can be explained by the high content of free moisture in comparison with other samples. A greater amount of bound moisture in all the samples except for the "control" can be explained by introducing into their composition plant cryoadditives, which contain compounds capable of "binding" water. The content of reducing substances during shelf life rises by approximately 30–64%, compared to the initial value, because of hydrolysis of sucrose. The acidity increases by 12–16%, as compared to freshly prepared samples. However, all indicators are within the limits that meet the requirements of regulatory documentation for this type of product.

According to the research in the field of medicine [22], excess levels of free oxygen radicals are among the main causes of the development of many diseases and premature aging. One can reduce the effects of free radicals by the regular use of natural fruit, vegetables, other plant raw materials, or functional food made with the use of these natural additives and having significant antioxidant properties. The antioxidant capacity value (AOC) provides information on the antioxidant potential of chemical compounds of a complex food system as for its ability to suppress oxidation processes. These data are important when testing the quality of developed foods during shelf life and under different storage conditions. That is why we have determined the bromine antioxidant capacity of marmalade samples with cryopastes and cryopowders (Fig. 1).

The data in Fig. 1 testify that the antioxidant capacity of the control sample of marmalade is 10.5 C/100 g, marmalade with one type of cryopaste – 6.2–44.8 C/100 g, with combinations of cryopaste – 49.7–50.2 C/100 g and with combinations of

cryopastes and cryopowders – 50.2–80.7 C/100 g. Thus, the use of cryoadditives from natural plant raw materials in marmalade technology allows a significant increase in bromine AOC products in 2.3–8 times, compared with the control. The increase is probably due to the addition of vegetable supplements with a high content of water-soluble vitamins and other substances having high antioxidant properties. After the storage period, the AOC index of products with cryoadditives is reduced by 15–35% while AOC control is reduced almost to a half. Thus, it has been established that, at the beginning and after the warranty period of storage, marmalade with cryopastes and cryopowders has significant antioxidant properties in comparison with the control.

Table 4. Physico-chemical parameters of the quality of jelly-fruit marmalade with cryoadditives during storage

Marmalade sample		Moisture content,%	Acidity, degree	Mass fraction of reducing substances,%
Control sample	Freshly prepared	22.9±1.15	11.6±0.58	9.5±0.48
	After 3 months	14.5±0.73	14.3±0.72	14.7±0.74
MI-1	Freshly prepared	23,2±1.16	12.3±0.62	8.0±0.40
	After 3 months	16.0±0.80	16.4±0.82	14.0±0.70
MA-2	Freshly prepared	23.0±1.15	11.0±0.55	8.0±0.40
	After 3 months	15.8±0.79	13.6±0.68	14.0±0.70
MC-3	Freshly prepared	23.4±1.17	15.5±0.78	7.9±0.40
	After 3 months	16.1±0.81	17.5±0.88	13.5±0.68
MP-4	Freshly prepared	23.0±1.15	15.2±0.76	8.9±0.41
	After 3 months	15.9±0.80	17.8±0.89	14.6±0.73
MG-5	Freshly prepared	23.6±1.18	17.8±0.89	9.2±0.46
	After 3 months	16.4±0.82	19.9±1.00	14.9±0.75
MAC-6	Freshly prepared	23.1±1.16	16.0±0.80	10.7±0.54
	After 3 months	16.5±0.83	17.9±0.90	15.0±0.75
MQP-7	Freshly prepared	22.9±1.15	17.5±0.88	10.3±0.52
	After 3 months	16.8±0.84	18.6±0.93	15.1±0.76
MQRH -8	Freshly prepared	22.0±1.10	13.5±0.68	11.0±0.55
	After 3 months	16.0±0.80	16.9±0.85	15.9±0.80
MARH -9	Freshly prepared	22.2±1.11	12.2±0.61	11.3±0.57
	After 3 months	15.6±0.78	15.6±0.78	15.8±0.79
MCSB -10	Freshly prepared	22.3±1.12	16.3±0.82	10.9±0.55
	After 3 months	15.7±0.79	18.8±0.94	15.0±0.80
MPSB -11	Freshly prepared	22.1±1.11	16.2±0.81	10.9±0.55
	After 3 months	16.3±0.82	18.6±0.93	15.0±0.75
MGG -12	Freshly prepared	22.4±1.12	18.4±0.92	12.0±0.60
	After 3 months	16.2±0.81	20.0±1.00	15.5±0.76

Shelf life is one of the most important characteristics. It determines the competitiveness of any product on the market. Confectionery products, because of a high concentration of sugar used in their production, should not cause concern about their safety. However, it should be noted that the ingredients of marmalade, besides sugar, include other components. In addition, when processing raw materials, the amount of microorganisms initially present may either decrease as a result of heat treatment, or increase in the production process. Any activity of microorganisms leads to physical and chemical changes in products. As a rule, these changes are undesirable, since they lead to a decrease in quality – a change in consistency, acidity, organoleptic indices, and sometimes even damage to products. This confirms the need for microbiological quality assessment of products.

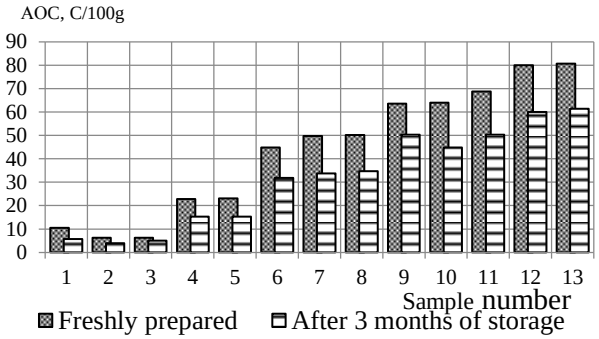

Fig. 1. Antioxidant capacity of new types of marmalade after storage: 1 – control sample (jelly-fruit marmalade), 2 – MC-3, 3 – MP-4, 4 – MQ-1, 5 – MA-2, 6 – MG-5, 7 – MQP-7, 8 – MAC-6, 9 – MCSB -10, 10 – MPSB-11, 11 – MGG-12, 12 – MQRH-8, 13 – MARH-9

The results of studies of microbiological quality indicators of jelly-fruit marmalade with cryoadditives, at the beginning and at the end of shelf life, are given in Table 5.

Table 5. Microbiological quality indicators of jelly-fruit marmalade with cryoadditives

Marmalade sample		QMAFAnM, CFU/g, not more than	Colibacillus (coliforms) in 0.1 g	Pathogens, including Salmonella, in 25 g	Molds, CFU/g, not more than
The value required by the standard		1×10^3	Not allowed	Not allowed	50
Control sample	Freshly prepared	1.7×10	Not found	Not found	12
	After 3 months	3.9×10			14
MQ-1	Freshly prepared	1.2×10	Not found	Not found	<10
	After 3 months	2.7×10			<10
MA-2	Freshly prepared	1.5×10	Not found	Not found	<10
	After 3 months	3.2×10			<10
MC-3	Freshly prepared	1.7×10	Not found	Not found	<10
	After 3 months	3.5×10			<10
MP-4	Freshly prepared	1.6×10	Not found	Not found	<10
	After 3 months	3.2×10			11
MG-5	Freshly prepared	2.7×10	Not found	Not found	12
	After 3 months	4.4×10			15
MAC-6	Freshly prepared	1.6×10	Not found	Not found	<10
	After 3 months	2.8×10			<10
MQP-7	Freshly prepared	1.6×10	Not found	Not found	<10
	After 3 months	2.7×10			13
MQRH-8	Freshly prepared	6.3×10	Not found	Not found	18
	After 3 months	7.1×10			22
MARH-9	Freshly prepared	6.5×10	Not found	Not found	22
	After 3 months	7.2×10			25
MCSB-10	Freshly prepared	4.5×10	Not found	Not found	12
	After 3 months	5.3×10			14
MPSB-11	Freshly prepared	4.7×10	Not found	Not found	13
	After 3 months	5.2×10			16
MGG-12	Freshly prepared	3.1×10	Not found	Not found	11
	After 3 months	5.9×10			16

As can be seen from Table 5, all samples of marmalade with plant cryopowders and cryopastes comply with current requirements for food quality, are safe from microbial contamination, and can be recommended for consumption without a risk to human health.

Approbation of research results. The technologies developed of jelly-fruit marmalade with herbal cryogenic additives have been introduced at 4 enterprises: the confectionery factory "SLADKIY MIR" (Kharkiv), CJSC "KONTI" (Konstantinovka), SP "Zhirko S. O." (Kharkiv), the hotel and restaurant complex "Antek" – Confectionery "Jaglo" (Poland). 2 utility model patents have been received, technical conditions and a technological instruction for the manufacture of new products have been developed and approved in accordance with the established procedure.

Conclusion

Organoleptic, physico-chemical, microbiolo-gical indicators of quality and antioxidant properties of jelly-fruit marmalade with cryoadditives from quince, apple, carrot, pumpkin, grapes, rose hips, and sea buckthorn were determined. It has been established that during storage of new types of marmalade with plant cryoadditives, their organoleptic, physico-chemical, and microbiological indicators of quality change to a certain extent, however, they meet all the requirements of the current normative documentation. The value of the antioxidant capacity of the newly developed samples and of those having been stored for 3 months is significantly higher than the same indicator for the control sample. Thus, the possibility of storing marmalade with increased antioxidant properties during the warranty period of 3 months is proved.

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