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OPTIMIZATION OF PRESCRIPTION COMPOSITION OF JELLY MASSES USING THE SCHEFFE'S SYMPLEX PLAN

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Abstract: Manufacturers are faced with the task of creating new types of functional marmalades enriched with useful ingredients. The studies were carried out at the department of technology of bakery, confectionary, macaroni and grain processing industries of Voronezh State University of Engineering Technology. The formulation of "Jelly-fruit" marmalade was taken as control. A part of apple puree (the substance content is 15%) was replaced with juice from seabuckthorn berries (the substance content is 12%) and concentrated apple juice (the substance content is 80%) in terms of solids. The effect of various ratios of apple puree, concentrated apple juice and juice from sea-buckthorn berries on the process of gelation of jelly masses based on pectin and agar using the experimental-statistical approach has been studied. The values of plastic strength were chosen as the output parameters that characterize the properties of jelly mass. The experiments were carried out using the Sheffe's simplex-centroid plan. The use of the simplex optimization method allowed to determine the optimum values of the mass fraction of the introduced formulation components that provide the preparation of jelly masses with the maximum plastic strength of 31 kPa if based on agar, and 33.23 kPa if based on pectin. The organoleptic and physicochemical indicators of product quality have been determined. The content of antioxidant activity in marmalade has been determined experimentally. The nutritional and energy value of new products and the degree of meeting daily needs have been calculated. The products can be recommended for dietary and diabetic nutrition, as they do not contain sugar and molasses, as well as for all who look after their health.

Keywords: Optimization, the Scheffe's simplex plan, Rosenbaum concentration triangles, jelly masses, agar, pectin, stevioside

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INTRODUCTION

The Russian Federation pays much attention to the development of a healthy lifestyle for the population. Malnutrition is one of the main factors worsening the health of the nation. Adult people consume large amounts of food high in vegetable fat and simple carbohydrates, whereas there are not enough vegetables and fruits in the diet, which leads to an increase in overweight, obesity, increasing the risk of developing diabetes and cardiovascular diseases.

A great theoretical and practical contribution to the creation of technology of new food products was made by the scientists A.V. Zubchenko, A.P. Nechaev, L.I. Puchkova, L.M. Aksenova, Z.G. Skobelskaya, T.B. Tsyganova, L.V. Antipova, L.P. Pashchenko, S.Ya. Koryachkina, G.O. Magomedov and others.

Marmalade confectionery is loved by most consumers. Therefore, manufacturers are faced with the task of creating new types of this category of products enriched with useful ingredients.

Sea buckthorn juice containing carbohydrates, fats, proteins, fiber, organic acids, flavonoids, catechins, sterols, coumarins, vitamins A, C, H, PP, β-carotene, the vitamin B complex, minerals – potassium, calcium, phosphorus, silicon, iron, titanium, zinc, etc. [1]; concentrated apple juice and puree were chosen as a fruit filler. Puree contains a lot of mono- and disaccharides, vitamins C, E, PP, B_1, B_2 and minerals. Concentrated juice is rich in amino acids and monosaccharides, which decompose under the influence of a high temperature and low pH into components with the formation of 5-hydroxymethylfurfural.

The processing of confectionery masses is followed by the complex chemical, physico-chemical, thermophysical and mechanical processes, the study of which allows for effective and objective rheological control and the control of technological production cycles [2].

Thanks to the works of the Russian scientists P.A. Rebinder, M.P. Volarovich, K.P. Guskov, V.P. Kalugin, M.N. Karavaev, N.I. Nazarov, V.P. Koryachkin, Yu.A. Machikhin Yu.A., M.A. Taleysnik, N.E. Fedorov, S.E. Kharin et al. and a number of foreign scientists – U.L. Wilkinson, J. S. Reed, J. Koch, G.W. Scott Blair, R.K. Schofield, M.S. Bourne, S.E. Sharm, A. Finke, H.G. Muller, A.G. Ward, A. Kramer, W. Twigg, S.H. Linn, S.D. Morgan and others, the issues of rheology with regard to food technology have been widely developed.

The study aims at developing a marmalade technology with new types of raw materials, the selection of optimal proportions of a fruit filler (apple puree, juice from sea-buckthorn berries, concentrated apple juice) for jelly masses based on agar and pectin using mathematical modeling methods.

STUDY OBJECTS AND METHODS

The study was carried out at the Department of technology of bakery, confectionery, macaroni and grain processing industries of the Voronezh State University of Engineering Technology. The mass fraction of moisture in the raw materials (%) was determined using the refractometric method. To do this, the sample was ground, weighed and diluted with water in a ratio of 1 : 1, then an analysis was carried out using an IRF-454 B2M refractometer (Russia). Two drops of solution were applied to the lower prism of the refractometer and the percentage of solids was measured using a scale. The result was doubled.

The rheological studies consisted in determining the dynamics of the structure formation of jelly masses and their viscous properties using the method described in [3]. The plastic strength of the jelly masses was determined using an electronic structurometer ST-1 (Russia) every 30 minutes. A sample bottle with a sample was fixed on a turntable and raised upwards so that the sample moved close to the cone. The "Start" button was pressed and the indicator readings – the maximum force value when moving the table up and down – were taken. Then the plastic strength was calculated using a formula.

The viscosity of jelly masses was measured using a rotational viscometer Rheotest RN 4.1 (Germany), introduced into the State Register of Measuring Instruments of the Russian Federation (No. 22819-07). The viscometer consists of two cylinders – a stationary outer and a movable inner cylinder. The studied mass was placed in the gap between the cylinders and then they were fixed. The inner cylinder was rotated at a constant angular velocity by means of an electric drive. The dependence of the torque on the angular velocity of rotation of the movable cylinder was experimentally obtained.

The antioxidant activity was determined using the device "Tsvet Yauza 01-AA" (Russia).

The method is based on an amperometric method for determining the content of antioxidants. The weight of the sample for the analysis is 20 g. A filtrate was prepared for the analysis and five consecutive measurements of the signals of standard quercetin solutions were made. The arithmetic mean was taken as the result. A calibration graph was plotted in the coordinates: X is the signal of quercetin; Y is the concentration of quercetin, mg/dm^3. The antioxidant activity (mg/dm^3) of the study object was calculated according to the calibration curve of quercetin and calculated using a formula.

The optimal ratio of fruit fillers was chosen according to the Scheffe's simplex-centroid plan.

RESULTS AND DISCUSSION

The samples of jelly-fruit marmalade were prepared in laboratory conditions. "Jelly-fruit" marmalade produced according to GOST 6442-2014 was chosen as the basis. When preparing marmalade based on pectin, a part of apple puree was replaced with juice from sea-buckthorn berries and concentrated apple juice, and apple puree was completely replaced with juice from sea-buckthorn berries in marmalade based on agar. In addition, sugar and molasses were excluded from the formulation and replaced with stevioside [4].

Stevioside, which is derived from stevia honey grass, is low in calories and does not increase the blood glucose level. The low doses evoke a sense of a sweet taste, the high doses have an unpleasant bitter aftertaste. Stevia contains iron, potassium, phosphorus, calcium, zinc, magnesium, selenium, chromium, cobalt, vitamins E, P, C, D, the vitamin B complex, essential oils and amino acids. The use of stevia helps to reduce blood sugar, strengthen immunity and reduce cholesterol in the body.

Sea buckthorn berries are valuable and healthy berries with a pleasant sweet and sour taste and a unique aroma [1]. These berries improve metabolism, strengthen the protective properties of the body and help to optimize the work of the gastrointestinal tract. Flavonoids that are found in all parts of the plant are anti-cancer antioxidants and can be used for the prevention of cancer.

Apple juice is a means of preventing viral diseases; its use helps in recovery after fatigue, physical exertion, improves the cardiovascular system and normalizes all kinds of metabolic processes in the body. Table 1 shows the chemical composition of fruit and berry supplements.

To develop a new kind of marmalade, apple puree with a mass fraction of solids of 15%, juice from sea-buckthorn berries with a mass fraction of solids of 12%, concentrated apple juice with a mass fraction of solids of 80% were chosen.

One of the basic physicochemical processes in the production of marmalade products is a gelation process.

When stevioside is applied instead of sugar, there is a slight decrease in plastic strength of 4 kPa (Fig. 1 and 2).

1 **Table 1.** Chemical composition and energy value of enrichers

Parameters	Apple puree	Juice from seabuckthorn berries	Concentrated apple juice
Protein, g	0.6	1.1	0.14
Fat, g	0.2	5.0	0.1
Carbohydrates, g	19.0	5.2	11.54
Fiber, g	1.1	1.8	0.1
Organic acids, g	–	1.8	–
Minerals, mg			
Calcium	12.0	20.0	6.0
Potassium	124.0	180.0	126.0
Sodium	1.0	3.0	7.0
Magnesium	7.0	28.0	5.0
Phosphorus	17.0	8.2	7.0
Iron	1.3	1.2	0.26
Manganese	–	–	0.063
Selenium	–	–	0.1
Zinc	–	–	0.04
Vitamins, mg			
A, μg	–	240	–
B_1	0.01	0.02	0.003
B_2	0.02	0.04	0.015
B_5	–	0.10	0.063
B_6	–	0.9	0.033
B_9, μg	–	9.0	–
C	1.6	190.0	25.0
PP	0.5	0.4	0.038
F	0.2	4.0	–
H, μg	–	3.0	–
Energy value, kcal (kJ)	82 (342.76)	52 (217.36)	47 (196.46)

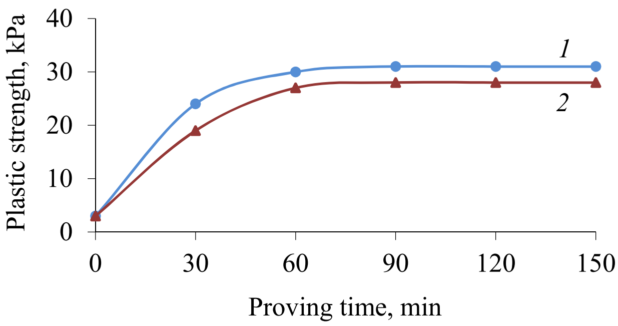

Fig. 1. Change in the proving of the plastic strength of jelly mass of the composition agar and: (*1*) sugar + molasses + apple puree; (*2*) stevioside + apple puree.

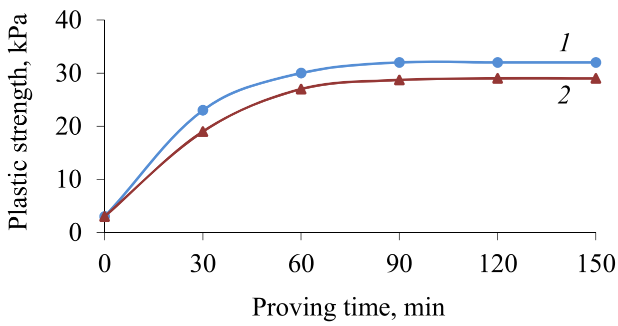

Fig. 2. Change in the proving of the plastic strength of jelly mass of the composition of pectin and: (*1*) sugar + molasses + apple puree; (*2*) stevioside + apple puree.

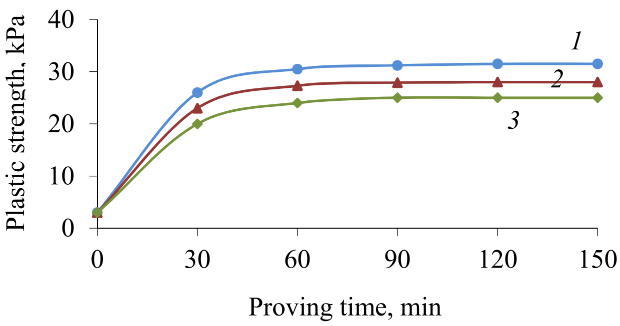

Fig. 3. Change in the proving of the plastic strength of jelly mass of the composition agar and: (*1*) molasses + sugar + apple puree (control); (*2*) stevioside + apple puree; (*3*) stevioside + juice from sea buckthorn berries.

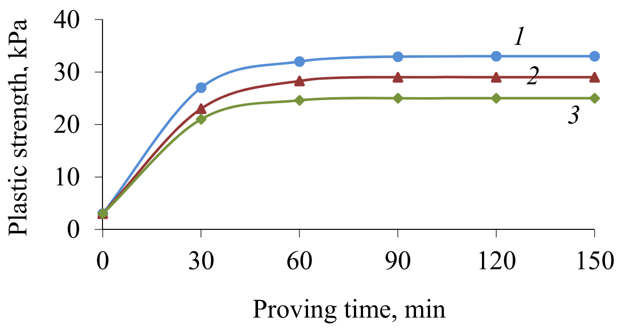

Fig. 4. Change in the proving of the plastic strength of jelly mass of the composition pectin and: (*1*) molasses + sugar + apple puree (control); (*2*) stevioside + apple puree; (*3*) stevioside + apple puree + juice from sea buckthorn berries + concentrated apple juice.

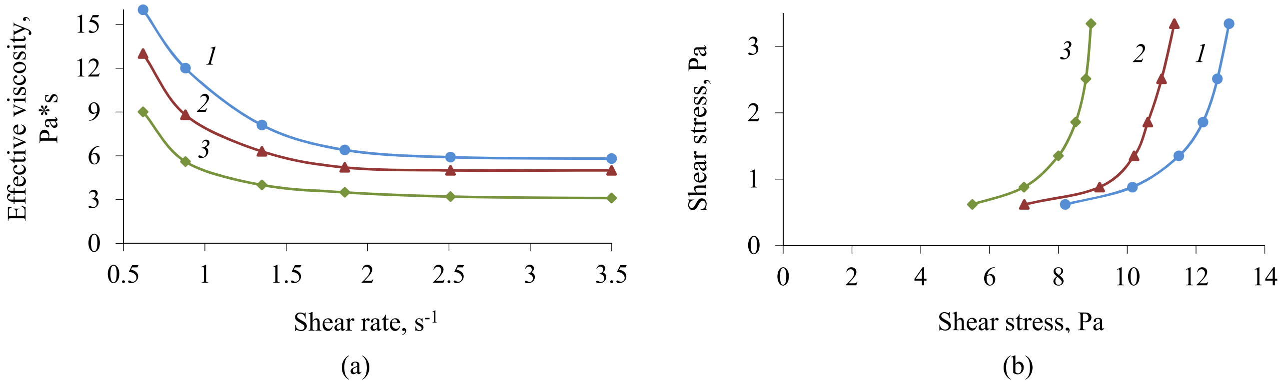

Fig. 5. Dependence of the effective viscosity of jelly mass based on agar on the shear rate (a) and flow curves (b) at t = 50°C of the jelly mass based on agar and: (*1*) molasses + sugar + apple puree (control); (*2*) stevioside + apple puree; (*3*) stevioside + juice from sea buckthorn berries.

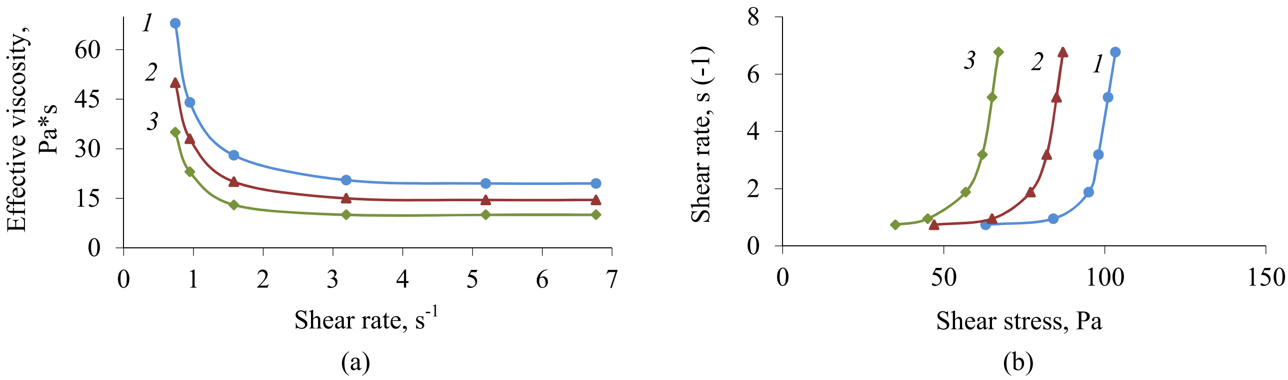

Fig. 6. Dependence of the effective viscosity of jelly mass based on pectin on the shear rate (a) and flow curves (b) at t = 70°C of the jelly mass based on pectin: (*1*) molasses + sugar + apple puree (control); (*2*) stevioside + apple puree; (*3*) stevioside + apple puree + juice from sea buckthorn berries + concentrated apple juice.

When sea buckthorn or concentrated apple juice is added to jelly mass, there is a decrease in plastic strength of 8.5 kPa. But this does not affect the form-retaining ability (Fig. 3 and 4).

The effective viscosity of jelly mass based on pectin and agar decreases with an increase in the shear rate by 20% and 30%, respectively. The samples have a lower effective viscosity compared with the control (Fig. 5 and 6).

The organoleptic and physicochemical indicators of product quality have been determined: the samples of marmalade have a pleasant taste and smell, the original color, a gelatinous consistency, the content of solids is: if based on agar – 78%, if based on pectin – 80%.

The effect of various ratios of apple puree, concentrated apple juice and juice from sea buckthorn berries on the process of gelation of jelly masses based on pectin and agar was studied using the experimental statistical approach [5].

Since in this case the term of independence of factors – the dosages of prescription components – is violated (that is, the content of each component depends on the sum of the rest), the traditional methods of planning (the full factorial experiment, its fractional replicas, orthogonal and rotatable planning) are unsuitable. Therefore, the decision has been made to use simplex-based planning, which allows us to take the following condition into account in the mathematical model

$$z_1 + z_2 + z_3 = 1, \qquad (1)$$

where z_1, z_2 and z_3 are, respectively, the mass fraction of apple puree, juice from sea buckthorn berries and concentrated apple juice, %.

If the number of factors $q = 3$, then the planning is carried out using the right simplex – an equilateral triangle. Each point of the triangle corresponds to a certain formulation of the triple system, and, conversely, each formulation is represented by a certain point.

The values of plastic strength (kPa) of the masses based on pectin y_1 and agar y_2 were taken as the output parameters that characterize the properties of jelly mass.

The experiments were carried out according to the Sheffe's simplex-centroid plan (Table 2). The order of the experiments is randomized through the table of random numbers. The Scheffe's plan provides for experiments both with pure compounding components and experiments at the center of the entire factor simplex and at the centers of all other lower simplexes that form the general simplex [6].

Table 2. Simplex-centroid planning conditions

Experiment No.	Mass fraction, %			Plastic strength of jelly mass, kPa	
	of apple puree z_1	juice from seabuckthorn berries z_2	concentrated apple juice z_3	pectin-based y_1	agar-based y_2
1	1	0	0	28.60	27.78
2	0	1	0	33.07	31.00
3	0	0	1	29.34	28.20
4	0.5	0.5	0	31.10	29.20
5	0.5	0	0.5	29.85	28.60
6	0	0.5	0.5	32.09	30.10
7	0.333	0.333	0.333	30.60	28.92

Having processed the experimental data, we obtained mathematical dependences of the plastic strength of jelly mass on the mass fraction of the studied compounding components in the form of incomplete third-order polynomials:

$$y_1 = 28{,}6z_1 + 33{,}07z_2 + 29{,}34z_3 - 1{,}06z_1z_2 - 3{,}52z_1z_3 - 3{,}54z_2z_3 + 95{,}49z_1z_2z_3, \quad (2)$$

$$y_2 = 27{,}78z_1 + 31{,}0z_2 + 28{,}2z_3 + 0{,}76z_1z_2 - 2{,}44z_1z_3 - 2{,}0z_2z_3 + 39{,}06z_1z_2z_3. \quad (3)$$

Since the Sheffe's simplex centroid plan is full (the number of experiments equals the number of the regression coefficients to be determined), then there is no required number of degrees of freedom to verify the adequacy of the obtained polynomial. In this regard, to check the adequacy, the additional experiments were carried out in 6 control points, the coordinates of which correspond to the combinations of values of the mass fraction of compounding components 0; 0.333 and 0.666. The choice of these conditions for checking the adequacy is explained (if necessary) by the possibility of transition to higher-order planning and the construction of complete polynomials of the third degree [7].

The results of the control experiments confirmed (according to Student's criterion) the adequacy of the equations (2) and (3) to the experimental data.

The graphic interpretation of incomplete third-order polynomials (2) (3) is shown in Fig. 7 and 8 in the form of Rosenbaum concentration triangles.

For a 3-component mixture, each diagram is a factor space in the form of an equilateral triangle. The content of each separate component z_i at the corresponding vertex of the triangle is 1 (or 100%) [8].

Along the side of the triangle z_1 z_2 from the vertex z_2 (in percent of the length of the triangle side taken as 100%), the relative content of the component z_i in the 2-component mixture z_1 z_2 is plotted. The points inside the triangle correspond to the 3-component mixture $z_1z_2z_3$. The percentage of the component z_1 that corresponds to an internal point A (Fig. 7) is determined by the length of the side of an equilateral triangle, similar to the original triangle $z_1z_2z_3$. It is constructed in such a way that the point A is one of the vertices, and one of the sides is the segment of the side z_1z_3 and z_1z_2. The values of the plastic strength (kPa) of the jelly mass prepared based on pectin y_1 and y_2 agar are shown by contour isolines in each of the diagrams.

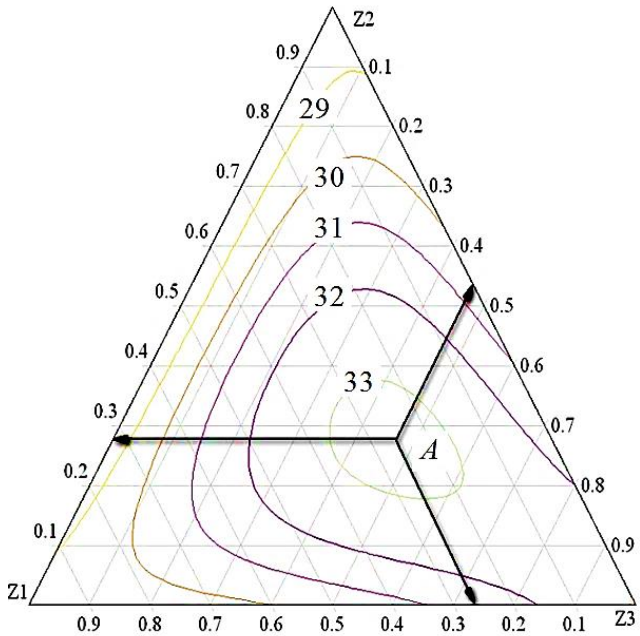

Fig. 7. Isolines of the plastic strength of the jelly mass based on pectin.

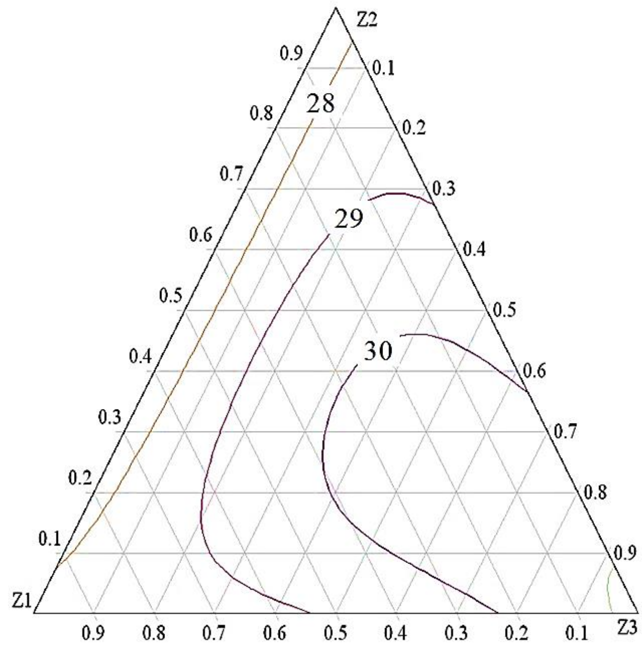

Fig. 8. Isolines of the plastic strength of the jelly mass based on agar.

The required diagrams allow, on the one hand, to predict the values of the plastic strength of jelly masses for the given values of the mass fraction of apple puree z_1, juice from sea buckthorn berries z_2 and concentrated apple juice z_3. On the other hand, it is possible to select the values of the mass fraction of the applied compounding components that make it possible to prepare jelly masses based on pectin or agar with the given plastic strength.

Using the simplex method of optimization made it possible to determine the optimum values of the mass fraction of the applied compounding components that provide the preparation of jelly masses with the maximum plastic strength (Table 3) [2, 3, 9].

With the found optimal ratios of apple puree, juice from sea buckthorn berries and concentrated apple juice, jelly masses have the best jelly-forming ability, do not lose their functional properties and contain the sufficient number of useful substances (fiber, macro- and microelements and vitamin B and C complexes) [10].

A formulation (Table 4) and a method for producing the fruit jelly marmalade "Solnechnoe utro" and "Uslada" have been developed.

Vegetable raw materials (vegetables, fruits and juices) are the main source of antioxidants. The need to estimate the content of antioxidants in food products is caused by striking the oxidant/antioxidant balance in the human body.

The content of antioxidant activity was determined experimentally in the marmalade "Uslada" based on pectin and "Solnechnoe utro" based on agar (Fig. 9).

Figure 9 shows that the highest value of antioxidant activity, compared to the control, in "Uslada" marmalade is 0.35 g quercetin/100 g of product, which is due to a high content of vitamins in the source raw materials.

The nutritional and energy value of new products has been calculated (Tables 4 and 5), the diagram of their comparative estimation has been presented (Fig. 10).

The energy value of "Solnechnoe utro" marmalade is lower than that in the control by 211 kcal (881.98 kJ), and "Uslada" – by 226 kcal (944.68 kJ)

The degree of meeting the daily needs when consuming 100 g of the marmalade "Solnechnoe utro" and "Uslada" increases in comparison with the content control: protein – by 23.64 and 6.5 times; fiber – by 1.34 and 1.26 times; sodium – by 4.03 and 6.72 times; potassium – by 16.06 and 3.41 times; magnesium – by 11.27 and 16.64 times; phosphorus – by 9.74 and 8.70 times; iron – by 4.23 and 4.17 times, A vitamins – by 54.92 and 42.34 times; C – by 17.1 and 25.9 times; E – by 1.23 and 6.13 times; B_1 – by 31 and 29 times; B_2 – by 32 and 14.5 times; B_9 – by 1.35 and 1.56 times, respectively.

The obtained products have been tasted (Table 6).

Table 6 shows that the best example is "Uslada" marmalade. It has the highest total score – 8.5, which indicates the highest organoleptic properties.

Table 3. Optimization results

Type of jelly mass based on	Plastic strength, kPa	Mass fraction, %		
		apple puree z_1	juice from seabuckthorn berries z_2	concentrated apple juice z_3
pectin	33.23	0.28	0.46	0.26
agar	31.0	0	1.0	0

Table 4. Formulation of marmalade

Name of raw materials	Marmelade	
	"Uslada"	"Solnechnoe utro"
Agar	–	+
Pectin	+	–
Stevioside	+	+
Apple puree	+	–
Juice from seabuckthorn berries	+	+
Concentrated apple juice	+	–

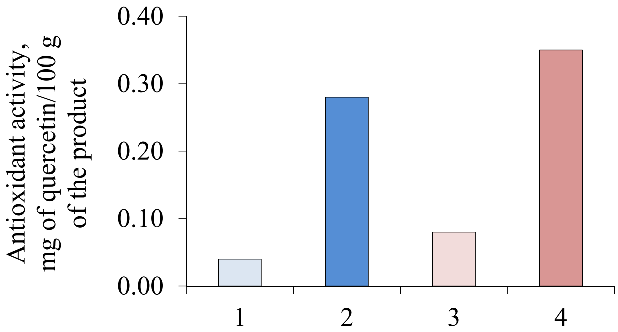

Fig. 9. Change in antioxidant activity in the marmalade samples: (1) agar-based marmalade (control); (2) agar-based marmalade "Solnechnoe utro"; (3) pectin-based marmalade (control); (4) pectin-based marmalade "Uslada".

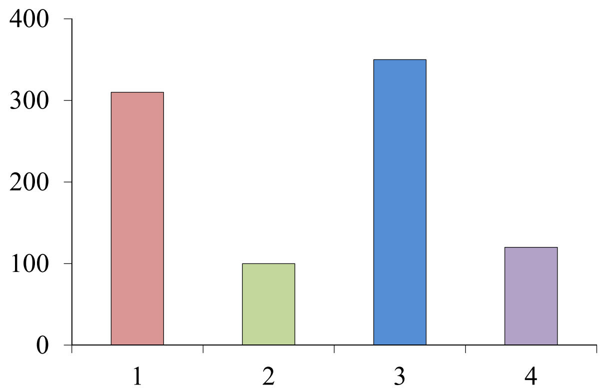

Fig. 10. Diagram of comparison of the energy value of marmalade: (1) "Jelly-fruit" based on agar (control); (2) "Solnechnoe utro" based on agar; (3) "Jelly-fruit" based on pectin (control); (4) "Uslada" based on pecti.

Table 4. Nutritional and energy value of marmalade

Name of nutrients	Content of nutrients in marmalade, g (mg) / 100 g			
	Jelly-fruit agar-based (control)	Jelly-fruit agar-based "Solnechnoe utro"	Jelly-fruit pectin-based (control)	Jelly-fruit pectin-based "Uslada"
Protein, g	0.17	4.02	0.63	4.07
Fat, g	0.06	0.74	–	0.45
Carbohydrates, g	82.4	71.12	72.75	69.93
Organic acids, g	1.17	0.21	0.79	0.28
Fiber, g	7.36	9.65	13.25	16.70
Minerals, mg				
Sodium	20.1	80.99	12.51	84.06
Calcium	30.88	27.23	16.0	26.99
Potassium	23.5	377.46	116.14	396.66
Magnesium	4.6	51.88	3.45	57.42
Phosphorus	10.80	105.17	11.38	99.05
Iron	0.82	3.47	1.01	4.22
Vitamins, mg				
PP	0.013	0.13	0.44	0.06
A, μg	–	54.92	–	42.34
B_1	0.0011	0.31	0.01	0.29
B_2	0.0011	0.32	0.02	0.29
B_5	–	0.59	–	0.66
B_6	–	0.62	–	0.66
B_9		1.35	–	1.56
F	–	1.23	0.22	1.35
C	0.23	39.50	1.72	44.55
Energy value, kcal (kJ)	313 (1308)	102 (426.36)	355 (1484)	129 (539.22)

Table 5. Degree of meeting daily needs

Name of nutrients	Degree of meeting daily needs, %				Norm, g (for minerals and vitamins, mg) in accordance with TR TS 022/2011
	Jelly-fruity agar-based (control)	Jelly-fruit pectin-based (control)	Jelly-fruit based on the agar "Solnechnoe utro"	Jelly-fruit based on the pectin "Uslada"	
Protein, g	0.02	0.7	4.47	4.52	75
Fat, g	0.08	–	0.80	0.50	94
Carbohydrates, g	18.31	16.7	16.54	15.54	530
Organic acids, g	59.0	39.5	11.00	14.00	2
Fiber, g	36.8	48.25	66.25	83.5	20
Minerals, mg					
Sodium	1.55	0.96	6.23	6.47	1300
Calcium	3.1	1.6	2.7	2.7	1000
Potassium	0.94	4.67	15.10	15.87	3500
Magnesium	1.16	0.86	14.36	14.36	400
Phosphorus	1.35	10.1	26.29	24.77	800
Iron	8.30	1.42	34.70	42.2	14
Vitamins, mg					
A, R.E., μg	–	–	16.14	13.45	800
C	0.27	2.22	43.88	49.44	60
B_1	0.08	0.69	20.66	13.33	1.4
B_2	0.07	0.68	17.78	16.11	1.6
B_5	–	–	7.38	8.25	6
B_6	–	–	32.63	34.74	2
B_9		–	67.5	78.00	200
F	–	1.38	72.35	79.41	10
PP (niacin)	0.06	2.44	0.65	0.30	18

1 **Table 6.** Results of a scoring of the organoleptic indicators of jelly-fruit marmalade

Quality indicators	Significance factor	Number of degrees of quality	Number of tasting participants	Estimate, score	
				"Uslada" marmalade pectin-based	Marmelade "Solnechnoe utro" agar-based
Taste, flavor	4	3	20	68	62
Structure and consistency	3	3	20	53	43
Color	2	3	20	35	33
Form	1	3	20	14	14
Overall estimate				172	152
Total score				8.5	7.6

CONCLUSIONS AND RECOMMENDATIONS

Thus, based on the studies carried out, the technology of marmalade has been developed with new types of vegetable raw materials and the optimal ratio of fruit and berry fillers has been chosen: apple puree, concentrated apple juice and juice from sea buckthorn berries.

The experimental and calculated data showed that marmalade has an increased nutritional value. Its use will enrich the diet with natural biologically active ingredients. The developed products can be recommended for dietary and diabetic nutrition, since they contain no sugar and molasses, as well as for all who look after their health.

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