UDC 664.665.94
DOI: 10.15587/1729-4061.2020.214903

DEVELOPMENT OF TECHNOLOGY FOR PREPARING THE THERMOSTABLE MILK-CONTAINING FILLING AND STUDY OF INFRARED SPECTRA OF ITS COMPONENTS

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Solving the problem of detecting counterfeiting in confectionery requires appropriate identification methods and is of paramount importance in the list of measures aimed at achieving food safety and quality.

The following infrared spectra of the samples were studied in this work:

– a model system containing xanthan gum and tare gum;

– a model system containing xanthan gum, tare gum, and sugar;

– a model system containing xanthan gum, tare gum, skimmed milk powder;

– a model system containing xanthan gum, tare gum, maltodextrin.

Absorption is characteristic of certain groups of atoms, its intensity is directly proportional to their concentration. Thus, the measurement of the absorption intensity makes it possible to calculate the amount of a given component in the sample.

A detailed technological scheme for the production of heat-resistant fillings using gelatin has been developed, which allows obtaining a product with new organoleptic and physical and chemical properties. The novelty of the technological scheme is the use of thermostable fillings of gelatin and the enzyme transglutaminase, and a mixture of gums, in the technology. A special feature in the production of these fillings is the operation of structuring at a temperature of 55±5 °C, which distinguishes it from existing technologies.

This paper reports the results of the infrared spectroscopic analysis of fillings, which made it possible to argue about the positive effect of gelatin usage in the technology of confectionery. The developed heat-resistant milk-containing filling has a high biological and nutritional value in comparison with the existing technologies of fillings for confectionery.

It has been analyzed that polysaccharides, gelatin, skimmed milk powder, and powdered sugar are rich, first of all, in reactive groups. Therefore, the physical and chemical properties of the fillings will be enhanced (adhesion, moisture-binding forms, etc.)

Keywords: thermostable fillings, xanthan gum, tare gum, confectionery, transglutaminase, IR-spectroscopy analysis

Received date 09.08.2020
Accepted date 01.10.2020
Published date 30.10.2020

1. Introduction

The use of various thermostable fillings [1] is a source of expanding the range of gingerbread, various types of cookies, cakes, rolls, and other flour-based confectionery [2]. During thermal treatment, conventional fruit products (jam, marmalade, preserves) boil, flow, burn, absorb into the dough. Thermostable fillings do not have these shortcomings. They retain their properties under normal conditions – when baking. Thermostable fillings are a typical semi-finished product that confectionery enterprises usually buy ready-made. It can be made directly at a confectionery enterprise; it is necessary to mix a standard fruit filling with a heat-stabilizing additive (pectin or a special mixture of hydrocolloids).

Infrared (IR) spectroscopy is one of the most common methods of molecular spectroscopy that studies the fluctuating spectra of molecules. These spectra are determined by the structure of the molecule and are associated with transitions between the oscillatory energy states or, in the classical interpretation, with the oscillations of atomic nuclei relative to equilibrium positions. The number and frequency of absorption bands are determined, first, by the number of atoms that make up the molecules, the masses of atomic nuclei, the geometry and symmetry of the equilibrium nuclear configuration, and, second, by the potential field of intramolecular forces. Thus, fluctuating spectra are the extremely specific and sensitive characteristics of molecules, which explains their widespread application in chemical research.

Infrared absorption spectra are a unique physical property. There are no two compounds, except optical isomers, with structures that differ but have the same IR spectra. In some cases, for example, polymers with a close molecular weight, the differences may be almost invisible but they are always there. In most cases, the IR spectrum is the "fingerprint" of a molecule, which is easy to distinguish from the spectra of other molecules.

Specialists in the confectionery industry must be aware of methods for assessing product quality [3]. Evaluation methods are based on the principle of modeling the temperature effect, as well as its duration, on the examined product under certain conditions (product shape, temperature, the duration of exposure, heat treatment time).

The use of heat-resistant fillings in the confectionery and baking industries is very important. The introduction of the improved technology of fillings for flour-based confectionery and culinary products would allow the use of raw materials of low quality, damaged during harvesting and transportation, or substandard raw materials. A thermostable filling will have high organoleptic characteristics, technological properties, biological value, as well as predictability in the technological process [4].

Various types of thickeners, gelling agents, or their mixtures of gelatin, pectin, agar, carrageenan, gums, native and modified starches, etc. are used in the production of fillings. When two or more thickeners are used together, a synergistic effect may occur: the mixtures thicken stronger, for example, xanthan with guar gum or locust bean gum. In the latter case, even gelation is possible [5]. The authors of [6] developed a heat-resistant filling using gelatin; they considered the effect of each formulation component on the experimental system by identifying interactions with their sequential application.

2. Literature analysis and problem statement

Work [7] reports the results of studying the infrared spectroscopic analysis of fillings. The positive effect of the use of concentrates in the technology of fruit and fat fillings for waffle products is shown. However, the work does not specify the ratio of formulation components of the filling and how the results affect the production technology.

Paper [8] describes the results of a study showing the effect of mechanolysis on the activation of sparingly soluble nanocomplexes of heteropolysaccharides in the development of nanotechnologies of plant additives. The authors showed the effect of the comprehensive action of cryogenic freezing or steam thermal processing and non-enzymatic catalysis, mechanolysis involving fine grinding, on the activation of inactive latent forms of sparingly soluble nanocomplexes of heteropolysaccharides with biopolymers. They also studied their transformations into a soluble nanoform during the processing of fruits, berries, and vegetables, as well as the development of nanotechnology of additives in the form of purees and powders. The comparison of the spectra of frozen fine puree made from fruit and berry raw materials and fresh raw materials is characteristic of the valence bonds of OH functional groups. Despite all the advantages, the condensate formed during the shock thawing of the raw materials has a negative impact on product quality while a given technology is complicated by the high cost of equipment.

Valence fluctuations are involved in the formation of intramolecular and intermolecular hydrogen bonds. The frequency domain is in the range from 3,200 to 3,650 cm^{-1}s. They are part of the free and bound moisture, the complexes of biopolymer BAS, such as phenolic compounds, tannins, sugars, and other BAS, there is a decrease in the intensity of the spectra, this is the approach used in [9]. All this suggests the destruction of the intermolecular and intramolecular hydrogen bonds, the destruction of nanocomplexes of biopolymers, including pectin, with other biopolymers and low molecular weight BAS, the disaggregation and mechanolysis of biopolymers or their associates and nanocomplexes.

Work [10] reports the results of research into the mechanism of interaction of vegetable cryopastes with the components of pasta dough. The work developed the technology of pasta of high nutritional and biological value with the addition of cryopastes from carrot and pumpkin. The effect of the selected additives on the components of pasta dough, gluten proteins, starch, water, was studied. The authors proved the strengthening effect of cryopastes on the gluten proteins of wheat flour, a reduction in the viscosity of starch paste in the presence of additives. The infrared spectroscopy of pasta dough samples revealed the formation of additional intermolecular bonds and structural complexes between the components of vegetable cryopastes and flour. However, the work does not specify how the measurement of absorption intensity makes it possible to calculate the amount of these components in the sample.

Paper [11] considered the regularities of interaction between collagen-containing proteins in the presence of transglutaminase and the protein substances of gluten-free flour and dough, which are manifested by an increase in the intensity of oscillations of groups CH- and COO-, skeletal oscillations of the side chains of protein macromolecule; changes in the native conformation of protein macromolecules. This is confirmed by the capability of proteins to bind the H+ and OH-ions and the formation of a structure with significant heterogeneity in terms of molecular weight in the technology of gluten-free bakery products using collagen-containing proteins and transglutaminase [12]. However, the paper does not determine the effect of each formulation component on the experimental system by identifying interactions with their sequential introduction.

Given the above, the cited works did not analyze the ratio of formulation components to obtain a thermostable filling. In addition, there are no studies into the infrared spectra of the qualitative composition of gels in the technologies of confectionery products. Therefore, our work addresses this research area.

3. The aim and the objectives of the study

The aim of this study is to develop a technology for the manufacture of a thermostable milk-based filling and to investigate the infrared spectra of the qualitative composition of gels in the technology of confectionery. This would make it possible to choose the optimal temperature for the technological operation of dispersing the mixture during the production of the developed filling. In addition to the fact that the absorption is characteristic of certain groups of atoms, its intensity is directly proportional to their concentration. Thus, the measurement of the absorption intensity makes it possible to calculate the amount of a given component in the sample.

To achieve this aim, the following tasks were set:

– to determine the ratio of the formulation components of a thermostable milk-containing filling;

– to develop the technological scheme for producing a thermostable milk-containing filling with the use of gelatin;

– to study the infrared spectra of model systems that contain: xanthan gum and tare gum; xanthan gum, tare gum, and sugar; xanthan gum, tare gum, skimmed milk powder; xanthan gum, tare gum, maltodextrin.

4. Materials and methods to study the use of infrared spectroscopy analysis

The spectroscopic studies were performed at the IR-Fourier spectrophotometer "Nicolet 380" made by Thermo electron corporation (USA). All the spectra from samples were acquired using a light-reflecting attached device; the operating wavelength range was 650–4,000 cm^{-1}; the spectra were recorded in 4 cm^{-1} increments. The obtained absorption spectra were processed using specially developed programs [13]. Each absorption band is characterized by a position in the spectrum, that is, a well-defined maximum absorption frequency and intensity – the width and height. For the quantitative characteristics of the spectra, a second-order derivative was calculated, as well as the integrated intensity, which is the area under the absorption curve. The quantitative content of different types of secondary structure was calculated by decomposing the total absorption spectra into individual components and determining the percentage (%) of the area of each component relative to the area under the cumulative curve. For statistical data treatment, we used a method of the spectrum component identification in the class of Gaussian and Lorentz curves using the differentiation algorithm. In addition, the law of the normal or Gaussian distribution was applied, that is, the original complex spectrum was decomposed into the sum of Gaussian curves. The Lorentz distribution corresponds to physical distribution. The asymmetry coefficient was also determined, which shows the deviation from the symmetry, that is, the offset relative to the average values, and the characteristic of congestion or scattering of data.

5. The results of developing a production technology for the thermostable milk-containing filling

5. 1. Determining the ratio of the formulation components for obtaining a thermostable milk-containing filling

In general, obtaining a heat-resistant milk filling implies the following. The dry components are mixed: a mixture of xanthan gum and tare gum at a ratio of 60±5 %: 40±5 % in their total concentration from 0.4 % to 1.2 %. Gelatin is mixed in an amount from 0.4 % to 1.0 %, transglutaminase – in an amount from 0.05 % to 0.3 %, skimmed milk powder – in an amount from 5.0 to 10.0 g. Powdered sugar is mixed in an amount from 10.0 to 35.0 g, maltodextrin – in an amount from 10.0 to 15.0 g; all dry components are mixed. The resulting mixture is hydrated in drinking water at a temperature of 55±5 °C for 7.5±2.5 minutes with constant stirring at a speed that ensures the even distribution of the components throughout the volume. Confectionery fat, pre-melted at a temperature of 55±5 °C and emulsified for 10±2 minutes, is added to the resulting system, in an amount from 10.0 to 20.0 g. The resulting mixture is poured into a production container and thermostated at a temperature of 55±5 °C, over 30 to 120 minutes. After thermostating, the filling is cooled to a temperature of 4±2 °C, packed, and stored.

Based on the results of our study, the following ratio of the formulation components of the thermostable milk-containing filling "Thermofilling" is proposed, given in Table 1.

Table 1

The ratio of formulation components for obtaining the thermostable milk-containing filling "Thermofilling"

Ratio of formulation components	(%)
Dry skimmed milk	5.0...10.0
Powdered sugar	10.0...35.0
Maltodextrin	10.0...15.0
Gelatin	0.4...1.0
Transglutaminase	0.05...0.3
Xanthan gum	0.24...0.72
Tare gum	0.16...0.48
Confectionery fat, or margarine, or spread, or butter	10.0...20.0
Water	60.0...20.0

The sequence of connecting the subsystems has a specific purpose and function (Table 2).

Table 2

The structure of the technological system for producing the thermostable milk-containing filling "Thermofilling"

Subsystem (stage) title	Operation	Characteristic of the subsystem (stage) functioning
E «Preparation of the formulation components 1»	Obtaining a dry mixture: – sifting; – mixing; – stirring	Quantity and quality control Providing dilution
D «Preparation of the formulation components 2»	– Sifting; – dispersion; heating; stirring	Quantity and quality control Providing dilution and swelling
C «Preparation of the semi-finished filling»	– Dispersion; – emulsification	Connecting the formulation components, the formation of structural-mechanical, physical- chemical properties
B «Structure formation and cooling of the semi-finished filling»	– Structure formation; – cooling	Providing the shape, structure, and the required quality characteristics of the filling
A «Obtaining the thermostable milk-containing filling»	– packing; – packaging; – storing	Obtaining heat-resistant milk-filling with specified organoleptic, physical-chemical, structural-mechanical, and microbiological characteristics

5. 2. Development of the technological scheme for producing a thermostable milk-containing filling using gelatin

Fig. 1 shows the general technological scheme of the production of a thermostable milk-containing filling using gelatin.

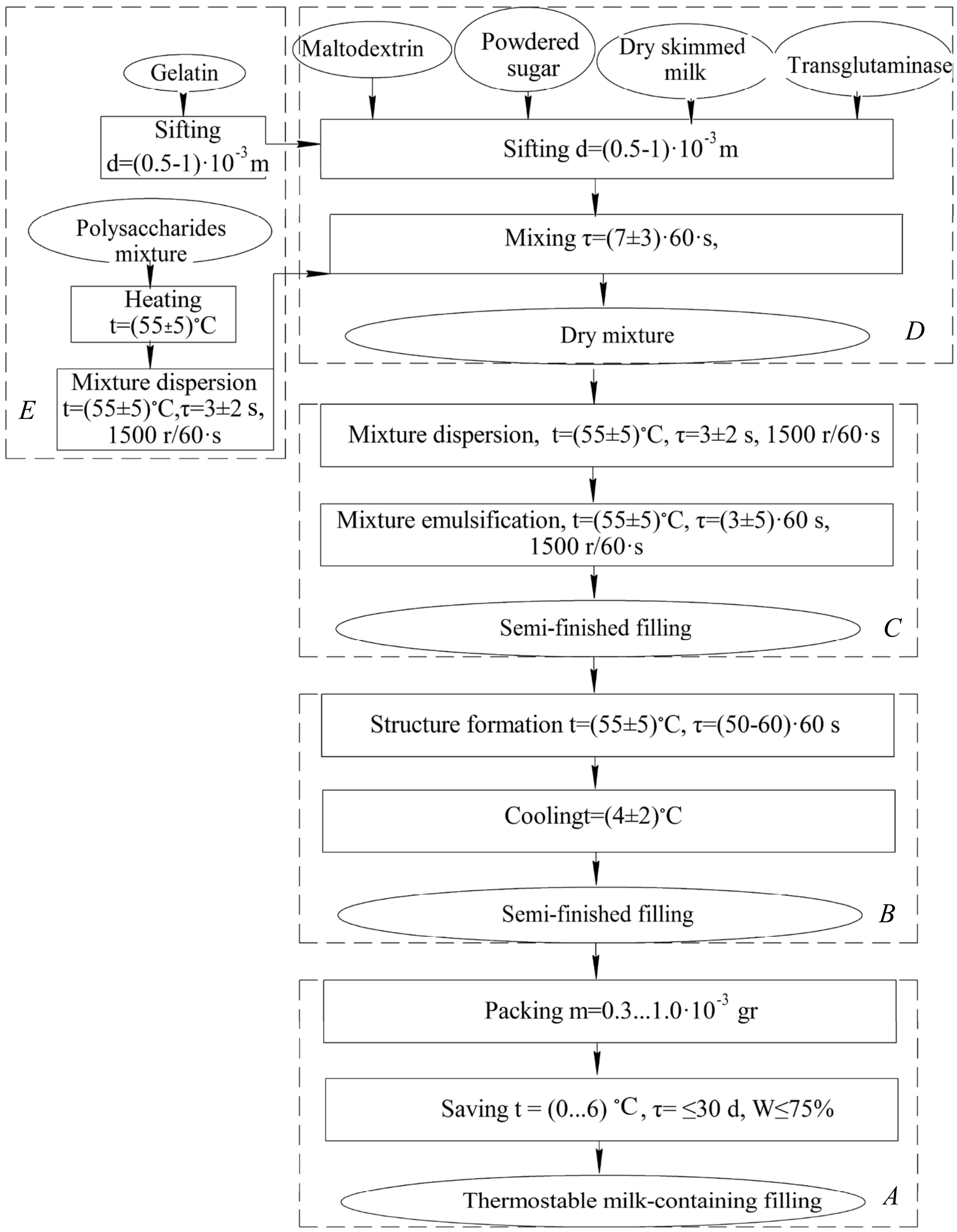

Fig. 1. Technological scheme of the production of a thermostable milk-containing filling using gelatin

The qualitative properties of a dry thermostable milk-containing filling are formed during its drying [14]. The physical, taste, and aromatic properties that are formed as a result of drying are due to the significant changes in the composition of the raw materials that occur as a result of biochemical reactions.

The physical-chemical parameters of the dry thermostable filling are given in Table 3.

Table 3

The physical-chemical parameters of the dry thermostable milk-containing filling

Indicator title	Standard	Control method
	Dry mixture	Requirements of regulatory documents
Mass fraction of dry matter, % not less	90–95	According to DSTU 4910
Titrated acidity, %	19	According to GOST 3624-92
Mass fraction of total sugar, % not exceeding	20–40	According to DSTU 5059
Dispersion (passage through a sieve), *d*, mm	Not ≥0.5	According to GOST 15113.2
Impurities	Not allowed	Visually
Mineral impurities	Not allowed	According to DSTU 4913

By studying the physical-chemical parameters, it was found that the dry thermostable milk-containing filling has high consumer properties, is an environmentally friendly product, and has a high nutritional value. Therefore, this technology has great prospects for development.

5. 3. The results of studying the infrared spectra of model systems in the production technologies of confectionery

Due to the combination and introduction of the established concentrations of protein-polysaccharide components (skimmed milk powder, maltodextrin, gelatin, xanthan gum, and tare gum), the filling acquires heat-resistant properties. IR spectroscopy was used to confirm these necessary effects and to identify the links between the formulation components of the experimental system of the milk-containing heat-resistant filling.

It is known that the radiation of IR spectra depends on the general structure of the molecule and its environment, on the group of atoms that are part of it, and the individual bonds between them (electron polarization). The molecule can only be in certain energy relations. Each energy level corresponds to its total energy, which characterizes the transition of the molecule from one level to another. In this case, only the frequency of radiation is absorbed, the energy of which is equal to the energy difference of any two levels, that is, those wavelengths that are able to change their energy state can be absorbed. The release of energy, which causes the oscillations of atoms in the molecule, is accompanied by the absorption of IR spectra due to changes in the dipole molecule moment. The effect of infrared radiation at a constantly variable frequency on the experimental system causes the absorption of certain radiation units by the molecule. This causes the deformation of the corresponding bonds (Table 4), which are registered in the form of bands with a minimum and maximum oscillation frequency (Fig. 2–5).

Table 4

Characteristic bands of valence oscillations of experimental systems

Sample No.	Peak region, cm^{-1}	Band assignment	Conclusion
1–4	3,600...3,100	Valence oscillations of OH group	Characterized by the construction of intermolecular hydrogen bonds
4–2	3,350...2,850	Valence oscillations of CH group	Characterized by the structure of alkanes
4–2	2,440...2,350	Valence oscillations of C=C group	Characterized by the structure of alkenes
1–2	2,140...2,100	Oscillations of organophosphorus P-H group	Due to the structure of the bonds in the presence of a milk base
1–4	1,680...1,620	Oscillations of C=C group	Characterized by the structure of alkenes
1–4	1,470...1,355	Symmetric deformative oscillations of CH_3 group	Characterized by the structure of alkanes
1–4	1,350...1,280	Valence oscillations of C-N group	Characterize peptide bonds
1–4	1,280...1,150	Oscillations of C-O-C ether group	Characterized by the structure of ether bonds
1–4	1,100...900	Valence oscillations of CH group	Characterized by the structure of alkanes
1, 3–2, 4	550...450	Oscillations of S=S group	Characterized by the structure of proteins

It should be noted that in the model systems in Fig. 2–5, all the shown spectra contain clearly expressed oscillation bands in the region of 1,000...3,100 cm^{-1}.

One should consider that all the studied samples have a band with a maximum near 2,500 cm^{-1}, which reveals the groups C–O–C and C≡C. Moreover, the peak with a frequency of 2,500 cm^{-1} is more pronounced for gel systems.

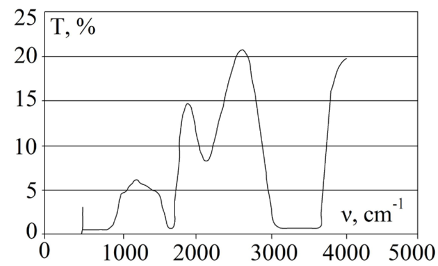

Fig. 2. IR spectra of the model system (a mixture of xanthan and tare gums)

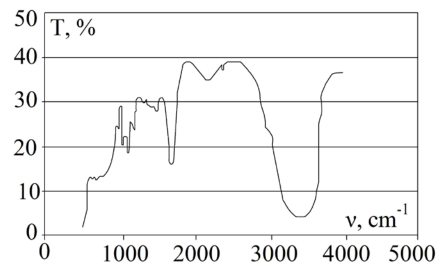

Fig. 3. IR spectra of the model system (a mixture of xanthan and tare gums with powdered sugar)

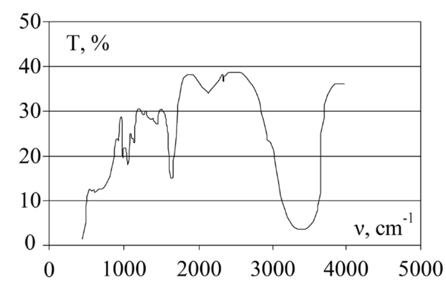

Fig. 4. IR spectra of the model system (a mixture of xanthan and tare gums with dry skimmed milk)

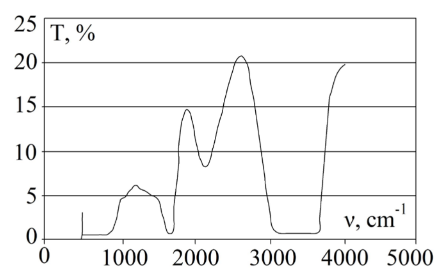

Fig. 5. IR spectra of the model system (a mixture of xanthan and tare gums with maltodextrin)

6. Discussion of results of studying the qualitative content of gels in the confectionery production technologies

All test samples have intense bands of absorption of valence antisymmetric and symmetric oscillations of C-H bonds of aliphatic and aromatic groups at 3,020 cm^{-1} and 2,930 cm^{-1}; these maxima are especially intensely expressed on the spectrum of the model systems made from a mixture of gums, powdered sugar, and skimmed milk powder.

For all studied samples, the C-H bond is manifested in wide absorption bands in the region of 2,950…2,850 cm^{-1} (especially wide bands are characteristic of the sample with polysaccharides in Fig. 1).

Fig. 1 shows that the mixture of gums is characterized by a less pronounced band around 2,500 cm^{-1}. This is due to the difference in the chemical composition of model systems with a fundamentally different base. The gel systems have a high moisture content (80…90 %). Therefore, the presence of highly reactive hydroxyl groups in the systems indicates a high interaction between the formulation components.

In addition, relative to the maximum near 2,500 cm^{-1}, we can note the effect of three different concentrates on the samples of the studied gels and the presence of highly reactive groups C–O–C and C≡C in them.

Fig. 2 shows that it is the model system from a mixture of xanthan and tare gums with powdered sugar that contributes to the formation of a wide absorption band, in which there are valence oscillations of the CH group.

Fig. 3 shows that the model system from a mixture of xanthan and tare gums with skimmed milk powder is formed by a wide group in which there are valence oscillations of group C=C, which are characterized by the structure of alkanes.

The model system in Fig. 4 has a more pronounced content of highly reactive groups C–O–C and C–OH than other presented samples, which does not contradict the study of IR spectra.

The above IR spectra demonstrate that all samples have the same explicit absorption peaks in the frequency range from 1,500 to 4,000 cm^{-1}, namely, cm^{-1}: 1,648; 2,122; 3,406. However, their intensity and amplitude, as well as the width of the absorption line, different. This is especially true of the peak at 3,406 cm^{-1}. For the model system containing xanthan gum and tare gum, the peak width is the widest: the absorption band starts at 3,063 and ends at 3,631. For the model system containing xanthan gum, tare gum, and maltodextrin, the absorption band is reduced by 10…15 %; for the model system containing xanthan gum, tare gum, and sugar – reduced by 50 %; and for the model system containing xanthan gum, tare gum and skimmed milk powder is 30 % of the band for the model system containing only xanthan gum and tare gum.

The presence of absorption bands in the model system containing xanthan gum and tare gum is determined by the composition of a givens system, namely the chemical composition of the components, that is, the chemical composition of xanthan gum and tare gum.

Thus, our study of the infra-red spectra of model systems containing xanthan gum, tare gum, sugar, skimmed milk powder, maltodextrin indicates that the absorption bands, and, therefore, the chemical composition, on these IR spectra, are determined by the IR spectra, and, accordingly, by the chemical composition of the components of a particular model system.

For the IR spectra of the studied model systems, the rule of additivity holds, that is, there are no chemical reactions between the components of these systems. However, the presence of the new, compared to the starting raw materials, bonds in the model systems changes their physical properties, namely thermophysical (heat capacity, melting point, crystallization temperature, or transition to the amorphous state), rheological (effective viscosity, shear stress).

7. Conclusions

1. Based on the results of our study, the ratio of formulation components for obtaining the thermostable milk filling "Thermofilling" has been proposed, namely: skimmed milk powder, 5.0…10.0 %; powdered sugar, 10.0…35.0 %; maltodextrin, 10.0…15.0 %; gelatin, 0.4…1.0 %; transglutaminase, 0.05…0.3 %; xanthan gum, 0.24…0.72 %; tare gum, 0.16…0.48 %; confectionery fat, or margarine, or spread, or butter, 10.0…20.0 %; water, 60.0… 20.0 %.

2. A detailed technological scheme for the production of a heat-resistant milk-containing filling using gelatin has been developed, which would allow obtaining a product with new organoleptic and physical-chemical properties. It has been experimentally confirmed that the organoleptic and physical-chemical parameters of the filling obtained by the developed technology meet the requirements set by TU 10.7-3105011043-001: 2020.

Obtaining a heat-resistant milk-containing filling is conducted as follows: mixing the dry components – a mixture of xanthan gum and tare gum at a ratio of 60±5 %: 40±5 % in a concentration of 0.4 %. Gelatin is in the amount of 0.4 %, transglutaminase – from 0.05 %, skimmed milk powder is in the amount of 5.0 g. Powdered sugar – in the amount of 10.0 g, maltodextrin – in the amount of 10.0 g, all dry components are stirred. The resulting mixture is hydrated in drinking water at a temperature of 55±5 °C for 7.5±2.5 minutes. Confectionery fat is added to the resulting system, in an amount from 10.0 to 20.0 g, pre-melted at a temperature of 55±5 °C, and emulsified for 10±2 minutes. The resulting mixture is poured into a production container and thermostated at a temperature of 55±5 °C over 30 minutes. After thermostating, the filling is cooled to a temperature of 4±2 °C, packed, and stored.

3. The effect of each formulation component on the experimental system was experimentally determined by identifying interactions with their sequential application. According to the results of IR spectroscopic analysis of model systems, there is a tendency to absorb the corresponding groups: OH, CH, PH, C≡C, CH, and S=S, which indicates the emergence of molecular and intermolecular interactions between the formulation components. The occurrence of the physical-chemical interactions between the formulation components of the experimental system of the thermostable milk-containing filling has been established, which contribute to the formation of the filling with stable physical-chemical parameters.

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