The object of this study is hot sauces with an emulsion structure, namely their microstructural and rheological indicators. The principal task addressed in the study is to identify the possibility of two-cycle heat treatment of sauces with maximum preservation of their properties. This could significantly expand their technological use, in particular, as part of ready-to-eat meals of industrial production. This approach contributes to the justification of the technological use of sauces while simultaneously determining the indicators that are control points in the technological flow of their production.

Studies on the microstructure of sauces have demonstrated a bimodal nature of the particle distribution with two pronounced peaks, which are in the range of 2.42–4.03 µm and 31.10–36.30 µm. The SPAN index is in the range of 1.62–1.72, reflecting moderate polydispersity. One- and two-cycle heat treatment of sauces contributes to a decrease in the specific surface area and an increase in the values of the volume-surface diameter of particles, which indicates a change in the microstructural characteristics of sauces. Sauces are structured food systems that demonstrate typical non-Newtonian behavior. During cyclic heat treatment of sauces, there is an increase in viscosity, storage moduli, and losses in the series "freshly made sauce→sauce after 1 heat treatment cycle→sauce after 2 heat treatment cycles".

A distinctive feature of the experimental results relates to studying sauces that are consumed hot, which determines additional requirements for their stability. The technological solutions proposed in this paper are aimed at their production in the form of semi-finished products with a high degree of readiness

Keywords: sauces with an emulsion structure, heat treatment, microstructural indicators, rheological indicators

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DETERMINING THE INFLUENCE OF TECHNOLOGICAL FACTORS ON THE MICROSTRUCTURAL AND RHEOLOGICAL PARAMETERS OF SAUCES WITH EMULSION STRUCTURE

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1. Introduction

The development of the food industry under the current conditions is an important component of the implementation of the national food security strategy. The practice of adapting the industry enterprises to the changing conditions of the business environment during the military conflict, in particular, entering new markets, as well as assessing the investment attractiveness of new technologies, contributes to the diversification of production, which results in the expansion of the range of products produced [1]. In the light of modern trends in the development of food technologies [2], the main of which are the focus on safety, functionality, personalization of nutrition, the production of semi-finished products and ready-to-eat meals is gaining momentum.

The evolution of modern lifestyles and the growing demand for convenience have become important driving forces in the sauces market. In a report [3], the global sauces market in 2023 was estimated at USD 46.48 billion, and by 2032 it is forecasted to reach USD 68.48 billion. As for the Ukrainian market, there are no sauce production volumes in value terms. However, the growth rates of their production volumes in Ukraine in 2023 and 2024 amounted to 11 % and 10 %, respectively.

The sauce industry is experiencing significant transformations, which are due to changes in consumer preferences, the development of the restaurant business and retail trade. Ready-to-eat sauces are increasingly in demand as they simplify the cooking process and diversify the taste and nutritional value of meals.

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Along with cold sauces, which are mainly represented by ketchups, mayonnaises, dressing sauces, there is a demand for hot sauces, which are consumed heated. Hot sauces do not require additional preparation, can be sold as a sauce or as part of ready-to-eat meals. The range of meals in which sauces can be used is quite wide. These are baked and stewed meals from vegetables, meat, poultry, fish, pasta, hot snacks. Understanding that in the technological flow of their production and use the sauce is repeatedly subjected to heat treatment predetermines additional requirements for ensuring their stability.

Sauces are considered structured systems, which, depending on the composition and technology, are suspensions, emulsions, or are polyphase dispersed systems. Despite the accumulated experience in their production, the technological aspects of ensuring the stability of the emulsion structure of hot sauces have not received sufficient coverage. Therefore, it is relevant to study the influence of technological factors on the microstructural and rheological indicators of hot sauces with an emulsion structure. The result of such an approach will be the expansion of the technological use of sauces while simultaneously determining the indicators that are control points in the technological flow of their production.

2. Literature review and problem statement

In [4] it is shown that ensuring the stability of the emulsion structure of food products is determined by many factors. Among the main ones are the nature and concentration of the emulsifier, the magnitude of its hydrophilic-lipophilic balance, the rate of adsorption at the interface of two phases, pH, ionic strength, mass fraction of the fat phase, and others. But the issues related to the heat treatment of recipe mixtures as a component of the technological process remained unresolved. All this gives grounds to argue that it is advisable to conduct a study on the influence of technological factors on the microstructural and rheological parameters of both emulsion systems and sauces with an emulsion structure.

In [5], the results of a study on the properties of "oil in water" emulsions with a mass fraction of fat of 5–50 % were reported. Starch octenyl succinic anhydride (OSA) with a concentration of 10 % was used as an emulsifier. It was shown that increasing the concentration of xanthan gum led to a decrease in the average droplet diameter and a more uniform droplet size distribution. All determined emulsions showed shear-thinning flow behavior. However, issues related to the use of xanthan gum in highly concentrated emulsions remained unresolved.

In [6], the prospects for using modified starches as stabilizers of emulsion systems were presented. Modification of starch OSA with the formation of an amphiphilic polysaccharide allows it to be used as an emulsion stabilizer. Additional processing of starch grains by acid hydrolysis and ultrasound makes it possible to obtain small starch granules that have better characteristics for stabilizing emulsions. However, unresolved issues regarding the influence of pH and ionic strength on the stabilization of emulsion systems are a limiting factor for its practical application.

In [7], the stability, color, textural, rheological properties, surface, and interfacial tension of oil-in-water emulsions stabilized with pregelatinized (PG) and granulated cold water swelling (GCWS) starches were investigated and compared. The emulsions demonstrated pseudoplastic behavior in the studied range of shear rates. It was shown that the pseudoplasticity of the emulsions increased due to an increase in starch concentration. The values of viscosity, consistency coefficient, and flow index were higher for emulsions with GCWS starch compared to emulsions made with PG. However, there is an inexpediency in using starch in technological processes that involve heat treatment. An option to overcome these difficulties is to search for new types of starches that increase the viscosity of the systems during heat treatment. This approach would allow them to be used as emulsion stabilizers.

The study of the microstructure and rheological parameters of emulsion systems based on pea aquafaba, which are stabilized by the introduction of pregelatinized starch, is reported in [8]. It was determined that for such parameters as the average diameter of the fat phase droplets ($D_{4,3}$=13.7–16.8 μm), viscosity (7.91–8.52 Pa·s at γ=40.0 1/s) the stability of the emulsions is 98–99 %. The assessment of the rheological parameters of the samples showed that they are food systems with high structural stability, they are characterized by the dominance of elastic properties over viscous ones with physical stability. However, the implementation of the obtained data is considered within the framework of a specific technological process – the production of vegetable snacks. At the same time, issues related to the use of aquafaba in other technologies, in particular, sauces with an emulsion structure, remained unresolved.

In [9] it is shown that pregelatinized corn starch OSA can be used as an emulsifier and gelling agent for the preparation of emulsions in the absence of additional emulsifiers, gelling agents, or heat treatment. Emulsions obtained with a content of 3–10 % OSA demonstrated shear thinning, they were classified as Herschel-Bulkley liquids. The resulting data make it possible to obtain emulsions with different textural and rheological parameters. All this gives grounds to argue that it is advisable to conduct a study on the use of modified starches in the technology of sauces with an emulsion structure.

In [10], the influence of whey protein (MWP) on the stability of milk emulsions was investigated. It was shown that the influencing factors were the parameters of homogenization and heat treatment of MWP suspensions, the mass fraction of MWP and polysaccharide gums, freezing-thawing. The influence of food ingredients (salt, sugar, gums), freezing rate on the microstructure, stability, and physicochemical properties of emulsion sauces was evaluated. The addition of MWP and guar and xanthate gums in a 1:1 ratio to emulsions increased their stability during freezing-thawing, reduced protein and droplet aggregation. However, issues related to the heat treatment of the formed emulsions remained unresolved, which makes it impossible to use the research findings more widely.

In [11], it is recommended to use whey protein concentrate at a concentration of 3.0…5.0 % and modified waxy corn starch at a concentration of up to 5.0 % to obtain thermostable emulsion systems. It is shown that the overall stability of emulsion systems is 97…98 %, which is a necessary condition for ensuring their stability. However, the work lacks information on the impact of cyclic heat treatment on the stability of emulsion systems. This gives grounds to argue about the feasibility of conducting such studies.

Despite the large body of research on ensuring the stability of emulsions, questions remain open regarding the determination of the parameters of sauces that are subjected to additional heat treatment for technological purposes. The limitations of published data necessitate additional studies.

In [12], the microstructure and dielectric properties of hot white sauces were investigated depending on the type of fat, emulsification rate, and during freezing and thawing. It was found that for freshly prepared samples, increasing the turbine speed from 350 rpm to 1100 rpm significantly reduces the size of fat globules. It was shown that defrosting sauces with subsequent heat treatment does not significantly affect the size and shape of fat globules. However, questions related to the determination of the parameters of heat treatment of sauces in the oven and microwave oven remain unresolved. This may result in objective difficulties in implementing this technology.

In [13], the influence of the viscosity of emulsion sauces on their stability indicators was investigated, which was achieved by introducing 0.2 %, 0.5 %, and 0.7 % of guar or xanthan gum. It was shown that heat treatment of guar gum contributed to its hydration, reducing the values of critical deformation and accumulation modulus. The opposite effect was observed when using xanthan gum, which ensured the formation of a structured matrix. All samples had non-Newtonian pseudoplastic behavior. The consistency index of sauces after heat treatment significantly decreased. However, the available experimental data relate to vegetable-based sauces. Therefore, the issues related to the use of gums in the composition of sauces based on dairy raw materials remained unresolved.

Work [14] reports the results of a study on the stability of white sauces in which starch was replaced by three types of dietary fibers – apple, potato, and microcrystalline cellulose. The stability of white sauces during freezing/thawing was studied by physicochemical rheological and sensory indicators. It was shown that the properties of white sauce with microcrystalline cellulose are as close as possible to the control sample. The sauce was characterized by high indicators of thermal stability, creamy texture, and homogeneity after heat treatment. However, the adopted assumption regarding the possibility of using dietary fibers in the composition of pasteurized meals was not confirmed. This predetermines the feasibility of conducting additional studies.

Our review of the literature [5–14] shows that the issues of forming the emulsion structure of hot sauces and ensuring their stability remain unresolved. Given the stable growth dynamics of the sauces market, the formation of the hot sauces segment as a component of industrially produced ready-to-eat meals requires development.

3. The aim and objectives of the study

The purpose of our study is to identify the features of the formation of microstructural and rheological indicators of sauces with an emulsion structure under the influence of one- and two-cycle heat treatment.

This will make it possible to comprehensively characterize sauces as a seasoning for the main meal (product) or side meal. The technological solutions proposed in the work are aimed at the production of sauces in the form of semi-finished products with a high degree of readiness.

To achieve the goal, the following tasks were set:

– to determine the influence of two-cycle heat treatment on the microstructural indicators of sauces with an emulsion structure;

– to determine the influence of two-cycle heat treatment on the rheological indicators of sauces with an emulsion structure.

4. The study materials and methods

The object of our study is hot sauces with an emulsion structure (hereinafter referred to as sauces), namely their microstructural and rheological parameters.

The main hypothesis of the study is the possibility of two-cycle heat treatment of sauces with maximum preservation of their properties. This could significantly expand their technological use, in particular, as part of ready-to-eat industrially produced meals.

Assumptions adopted in the work: the study assumes that the use of modified waxy corn starch and xanthan gum in hot sauces with an emulsion structure could ensure their stability to the effects of two-cycle heat treatment. It is assumed that this would allow the sauce to be used as part of ready-to-eat industrially produced meals. It is predicted that changes in the microstructural and rheological parameters of sauces would not affect their consumer properties.

Simplifications adopted in the work: the study was conducted on a sauce that is basic, without taking into account the possibility of expanding its range by using fillers of plant or animal origin. The study was also limited to the analysis of key parameters, including the stability of the sauce and its microstructural and rheological indicators. Broader aspects, such as the study of changes in organoleptic and physicochemical indicators, are not included in this study.

The starting materials for the study included the following: whey protein concentrate (manufacturer – Lactelle SP., Poland), refined deodorized sunflower oil (manufacturer – TOV "SAN OIL", Ukraine). Xanthan gum (Ziboxan F-200, manufacturer – Deosen Biochemical LTD, China) and modified waxy corn starch (TERMFLO, manufacturer – Ingredion, Germany) were used as stabilizers of the emulsion structure. The food raw materials from which the sauces were made met the requirements of the current regulatory legal acts of Ukraine regarding the quality and safety indicators of food products, packaging, labeling, transportation, acceptance, and storage.

The sauces were made on a universal kitchen appliance Thermomix (Vorwerk, model TM6, Germany). The technological process was carried out in the following sequence: drinking water, dry recipe components (table salt, white sugar, whey protein concentrate) were added to the thermomix bowl, and the mixture was heated to a temperature of 60–65 °C with constant stirring.

Then, without stopping heating, sunflower oil was introduced and the system was emulsified at a turbine speed of 1500–3000 rpm. To ensure the stability of the emulsions, a fat suspension of polysaccharides (xanthan gum, modified starch) was introduced, the mixture was heated to a temperature of 87–90 °C and pasteurized for 10 minutes. Then the sauce was packaged in consumer containers, cooled to a temperature of 20.0±0.5 °C.

Two-cycle heat treatment of sauces was carried out with the following parameters: heating to a temperature of 87–90 °C with subsequent cooling to a temperature of 20.0±0.5 °C.

ANOVA of sauces was carried out using a Mastersizer 3000 particle size analyzer (Malvern Panalytical Ltd, Great Britain), particle size range – 0.01–3500 μm, measurement accuracy – 0.6 %, measurement convergence better than 0.5 % variations. The obtained experimental data were processed using the Mastersizer v3.17 software. The SPAN index was calculated from the following formula [15]:

$$SPAN=(D_{90}-D_{10})/D_{50}, \quad (1)$$

where D_{10} is the particle diameter at 10 % of the total size distribution, respectively;

D_{50} is the particle diameter at 50 % of the total size distribution, respectively;

D_{90} is the particle diameter at 90 % of the total size distribution, respectively.

The particle sizes in the volume-weighted size distribution $D_{4,3}$ (or De Brouckere mean) were determined from the following formula [15]:

$$D_{4,3}=\frac{\sum n_i D_i^4}{\sum n_i D_i^3}, \quad (2)$$

where D_i is the particle size as the diameter of the particles in each size class,

n_i is the number of particles in each size class per unit volume of emulsion.

The volume-surface diameter of the droplets, the Sauter diameter $D_{3,2}$ (Surface-weighted (or Sauter) mean), was determined from the following formula [15]:

$$D_{3,2}=\frac{\sum n_i D_i^3}{\sum n_i D_i^2}. \quad (3)$$

The stability of emulsions was determined according to DSTU 4560:20.

The rheological characteristics of the studied mixtures were determined using a Kinexus Pro+ rotational rheometer (manufactured by Malvern Instruments Ltd, Great Britain). The studies were carried out using the upper measuring geometry – a C25 DIN L0142 SS cylinder, and the lower measuring geometry PC25 DIN C0350. The viscosity and flow curves were obtained by changing the shear rate within 0.1–100 1/s with 10 measurements per decade. Oscillatory studies were carried out at 0.5 % complex shear strain in the frequency range of 10.0–0.1 Hz [16, 17].

The relaxation characteristics of the studied samples were determined by the dependences of shear stress and shear strain on the relaxation time for 5 minutes [18]. To approximate the obtained experimental data, the power law (4) and Herschel-Bulkley (5) models were chosen, which describe the dependence of the shear stress on shear rate:

$$\tau=K\cdot\gamma^n, \quad (4)$$

$$\tau=\tau_0+K\cdot\gamma^n, \quad (5)$$

where τ is the shear stress, Pa;

τ_0 is the shear yield strength, Pa;

γ is the shear rate, 1/s;

K is the consistency coefficient, Pa·s^n;

n is the flow behavior index.

The angular frequency ω [rad/s] or [1/s] was determined from the following formula [19]:

$$\omega=2\pi\cdot f, \quad (6)$$

where π=3.141…;

f – frequency, f [Hz] or [1/s].

The complex modulus, G^* [Pa], was determined from the following formula [19]:

$$|G^*|=\sqrt{(G')^2+(G'')^2}, \quad (7)$$

where G' is the accumulation modulus, [Pa];

G'' is the loss modulus, [Pa].

The loss coefficient tanδ was determined from the following formula [19]:

$$\tan\delta=G''/G'. \quad (8)$$

The complex viscosity, η*, [Pa·s] was determined from the following formula [19]:

$$|\eta^*|=|G^*|/\omega. \quad (9)$$

Mathematical and statistical processing of the results of rheological studies was carried out using the SigmaPlot 14 and OriginPro software packages. To assess the reliability of the results obtained, the calculation of Student coefficients (t_{ST}) was used for the adopted level of dependence P=0.05 and the corresponding (n^{-1}) number of degrees of freedom.

The error in the studies was σ=3…5 %, the number of repeated experiments was n=3, the probability was $p\geq0.95$.

5. Results of research into the influence of technological factors on the microstructural and rheological parameters of sauces

5. 1. Determining the influence of two-cycle heat treatment on the microstructural parameters of sauces

Conceptually, hot sauces are ready-to-eat. They can be used as a part of second courses of traditional or industrial (long shelf life, which is achieved through pasteurization) production. The above requires a study of their indicators under the influence of heat treatment. Fig. 1 visualizes the technological chain of production and use of sauces with an indication of the predicted heat treatment cycles. It is clear from Fig. 1 that the sauce can be used in two ways. The first is the use of sauces as basic in the composition of culinary products (for example, in baked meals from vegetables, meat, mushrooms, pasta, etc.). Under these conditions, the sauce is subjected to 2 cycles of heat treatment: at the stage of cooking the meal and at the stage of its consumption. The second is using sauces immediately after heating them (1 heat treatment cycle).

The microstructural parameters of freshly prepared sauces, as well as after the first (1) and second (2) cycles of heat treatment, were investigated (Fig. 2, Table 1).

Table 1

Particle size distribution of sauces

Name of sauces	Concentration, %	SPAN	Specific surface, m^2/kg	$D_{3,2}$, μm	$D_{4,3}$, μm	D_{10}, μm	D_{50}, μm	D_{90}, μm
Freshly made sauce	0.0033	1.72	711.6	8.4	24.1	2.8	25.0	45.7
Sauce after 1 heat treatment cycle	0.0035	1.62	695.3	8.63	23.6	2.7	24.7	42.8
Sauce after 2 heat treatment cycles	0.0040	1.62	574.5	10.4	26.7	3.2	27.2	47.2

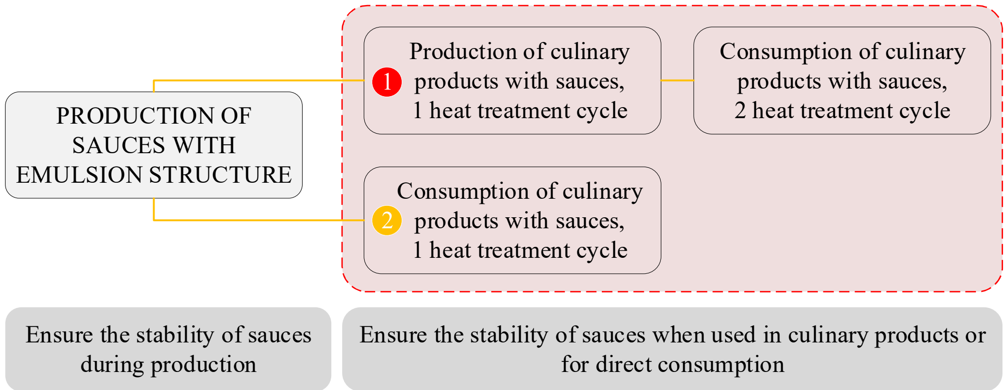

Fig. 1. Technological chain of production and use of sauces with indication of predicted heat treatment cycles

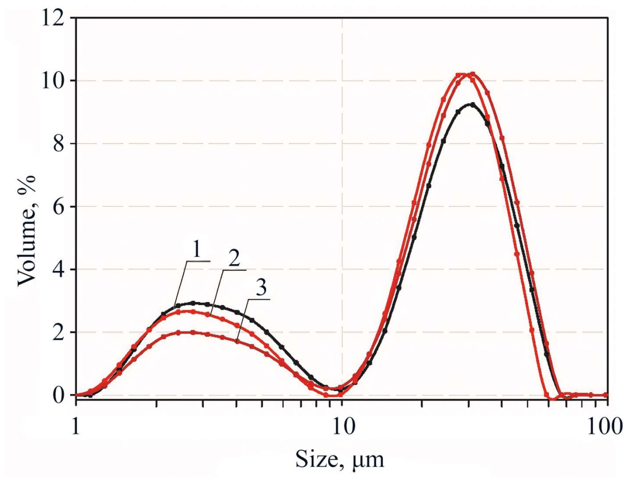

Fig. 2. Particle size distribution: 1 — freshly made sauce; 2 — sauce after 1 heat treatment cycle; 3 — sauce after 2 heat treatment cycles

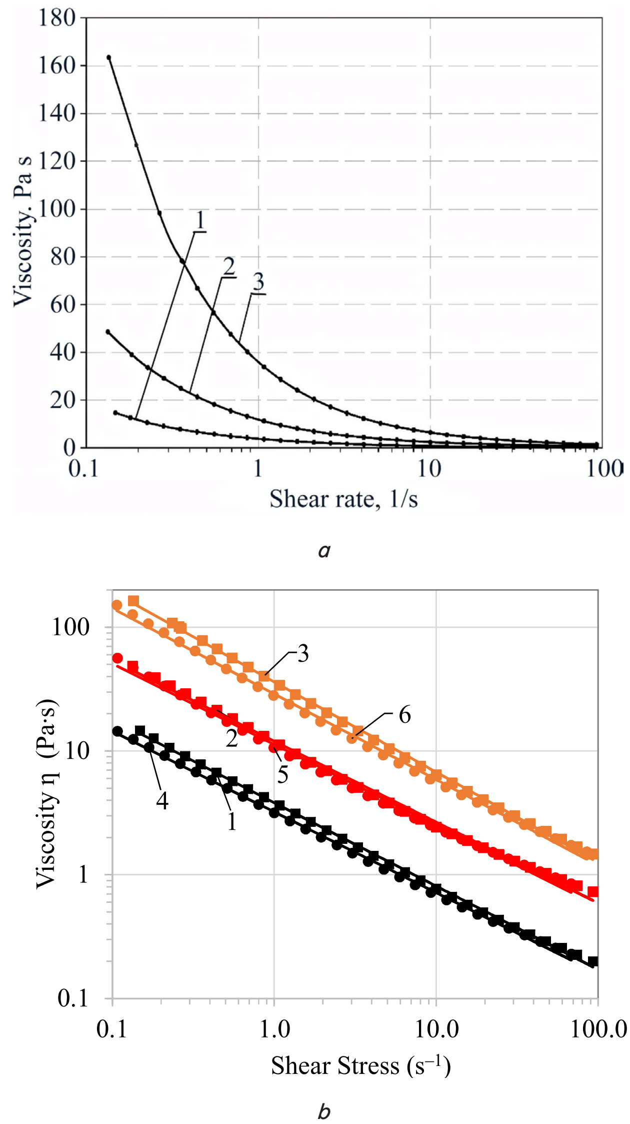

Fig. 3. Dependence of the shear viscosity of sauces on the shear rate under the following mode: *a* — forward movement; *b* — forward and reverse movement (1, 4 — freshly prepared sauce; 2, 5 — after 1 heat treatment cycle; 3, 6 — after 2 heat treatment cycles

This approach models the technological process of their production and further use. This makes it possible to evaluate the microstructural parameters of sauces at specific stages and draw conclusions about the possibility of their use in food technology.

5. 2. Determining the influence of two-cycle heat treatment on the rheological parameters of sauces

Fig. 3 shows the dependences of the shear viscosity of sauces on the shear rate under the forward (Fig. 3, *a*), forward and reverse movement (Fig. 3, *b*) modes. Sauces are structured food systems, which are characterized by the fact that in a certain range of changes in the shear rate, which exceeds the true yield strength, the viscosity of the system drops abruptly to a minimum (Fig. 2, *a*). The studied samples demonstrate the typical behavior of a non-Newtonian fluid, which is characterized by a decrease in effective viscosity with increasing shear rate (pseudoplastic behavior). Under the forward movement mode, the viscosity of sauces in the region of low speed values (up to 10.0 s^{-1}) varies; starting from a shear rate of 10.0 s^{-1}, the difference is significantly smaller.

Table 2 gives the parameters of constants in the mathematical equation of the Herschel-Bulkley and Power Law rheological models, which allowed us to characterize the studied samples.

The changed data, which we changed not due to editorial comments, are highlighted in gray.

The selected rheological models have high correlation coefficients (R^2=0.9997–0.9999), which indicates their high approximation ability. After 1 and 2 cycles of heat treatment, the values of shear stress and consistency coefficients increase, which indicates structural stability. As noted in [20], this may be the result of the interaction of starch polysaccharides, as a result of which a gel-like structure is formed at a temperature of 20.0±0.5 °C. An increase in the consistency coefficient with a simultaneous decrease in the flow behavior index indicates an increase in pseudoplasticity of sauce samples under the influence of heating-cooling.

Fig. 4 shows the dependences of storage (*G*') and loss (*G*'') moduli on the angular frequency for sauces freshly prepared and after two-cycle heat treatment.

The accumulation modulus (*G*') describes the proportion of elastic properties and corresponds to the part of the energy that is stored. The loss modulus (*G*'') corresponds to the viscous properties and determines the part of the energy that is lost during sinusoidal deformation. From the data shown in Fig. 4, it is seen that after the 1st and 2nd heating-cooling cycles, the storage and loss moduli increase. The location of the curves 1', 2', 3' and 1'', 2'', 3'' practically parallel to each other indicates the typical course of the process of increasing elastic properties.

After each heat treatment cycle, the values of the complex viscosity increase. At the same time, the complex viscosity decreases with increasing angular frequency, which is a characteristic feature of the pseudoplastic behavior of the studied samples.

The accumulation (*G*') and loss (*G*'') moduli are fitted to a power model; the linear regression correlation coefficients (R^2) are above 0.99, which confirms the adequacy of the use of the selected model. The resulting parameter values are summarized in Table 3.

During two-cycle heat treatment, the values of the flow behavior index (*n*' and *n*'') decrease, and the values of the consistency coefficients (*K*' and *K*'') increase. At the same time, the values of *n*'' and *K*′ remain dominant in all samples, which indicates the viscoelastic properties of the studied samples.

Table 2

Parameters for fitting the dependence of shear rate and shear stress to the Herschel-Bulkley and Power Law rheological models

Indicator ID	Measurement unit	The value of sauce indicators		
		Freshly made	Sauce after 1 heat treatment cycle	Sauce after 2 heat treatment cycles
Herschel-Bulkley model				
Yield shear stress, τ_0	Pa	0.5794±0.1013	1.1867±0.5459	5.5273±0.9581
Coefficient of consistency, K	Pa·s^n	3.1950±0.1060	10.7235±0.5765	30.3613±0.9936
Flow behavior index, *n*	–	0.3698±0.0156	0.3351±0.0190	0.2883±0.0099
Correlation coefficient, R^2	–	0.9999	0.9998	0.9999
Standard error, SE	–	0.0490	0.1524	0.1744
Power model				
Coefficient of consistency, K	Pa·s^n	3.7912±0.0176	11.9609±0.0464	36.0361±0.0706
Flow behavior index, *n*	–	0.2982±0.0036	0.2986±0.0034	0.2407±0.0018
Correlation coefficient, R^2	–	0.9997	0.9997	0.9999
Standard error, SE	–	0.0695	0.1589	0.2327

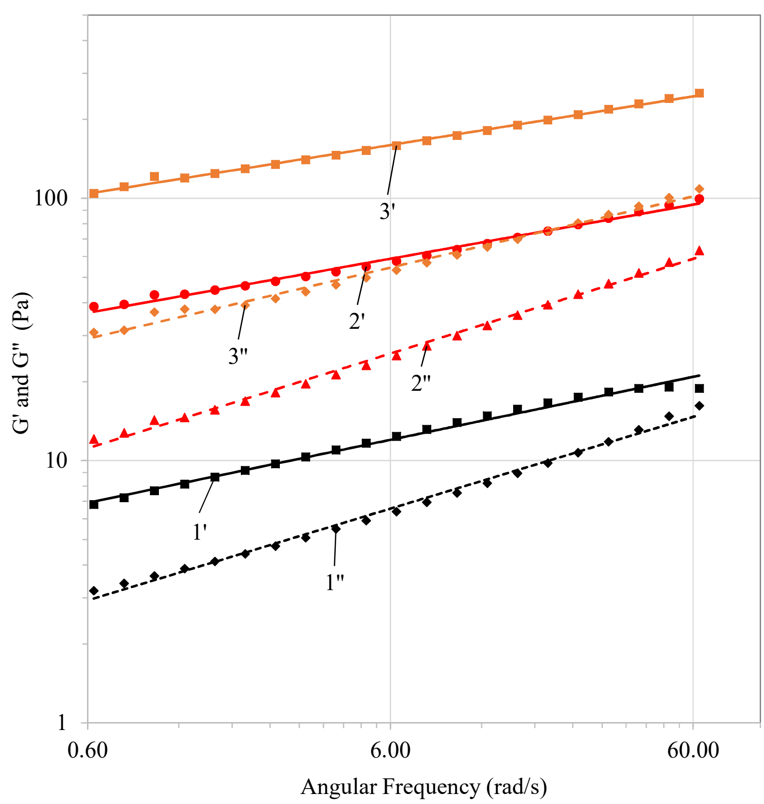

Fig. 4. Dependence of the accumulation modulus (*G*') and loss modulus (*G*'') of sauces on angular frequency [ω, rad/s], where: 1', 1'' – freshly prepared sauce; 2', 2'' – sauce after 1 heat treatment cycle; 3', 3'' – sauce after 2 heat treatment cycles

Table 3

Power law model parameters designed for frequency response curves of the studied sauces

Sample ID	G'			G''		
	K', Pa·s^n	n'	R^2	K'', Pa·s^n	n''	R^2
Freshly made sauce	7.807	0.2402	0.9800	3.5067	0.3497	0.9918
Sauce after 1 heat treatment cycle	40.668	0.2062	0.9927	13.403	0.3612	0.9964
Sauce after 2 heat treatment cycles	114.49	0.1856	0.9971	33.503	0.2711	0.9928

6. Discussion of results based on investigating the influence of technological factors on the microstructural and rheological parameters of sauces

In papers [5–7], which consider the preparation of emulsion systems and ensuring their stability by using food hydrocolloids, the issue of the influence of additional heat treatment is not covered. Unlike [5–7], our studies highlight the influence of two-cycle heat treatment on their parameters. This becomes possible due to the justification of the recipe composition of sauces and technological parameters of their production.

Studies on the microstructure of sauces (Fig. 2), which is described by distribution curves, demonstrate the bimodal nature of the distribution of particles that are tangent to each other. The first, less pronounced peak is observed in the range of 2.42–4.03 µm. It corresponds to the value of D_{10} – the diameter of particles smaller than this value and is 10 % of the total. The second, main pronounced peak, is in the range of 31.10–36.30 µm, with 50 % of the particles in the studied samples having a particle diameter D_{50} smaller than 24.7–27.2 µm. The maximum particle size D_{90} in the system is in the range of 42.8–47.2 µm. The SPAN index is in the range of 1.62–1.72, reflecting the moderate polydispersity of the studied systems (Table 1).

The study of the influence of the 1st and 2nd heat treatment cycles affects the volume-weighted diameter $D_{4,3}$, which increases from 23.6 µm to 26.7 µm after the 2nd heat treatment cycle of the sauce. Similarly, the value of the volume-surface diameter of the droplets $D_{2,3}$ increases – from 8.43 µm to 10.40 µm, which indicates a certain influence of heat treatment on the microstructural characteristics of the sauces (Table 1).

The decrease in the specific surface and dimensional characteristics of the particles after two-cycle heat treatment indicates the presence of a subtle process of coalescence of the dispersed phase. However, the insignificant coalescence of fat droplets does not lead to a loss of sedimentation stability by the system and its stratification. The stability of freshly prepared sauces and after 1 and 2 heating-cooling cycles is 98.5±0.5 %, 99.0±0.5 %, and 99.0±0.5 %, respectively. At the same time, the existing changes may indicate the course of processes that would affect the rheological parameters and the overall stability of sauces, which determines the need to study these parameters.

The presence of an internal structure of sauces, which is formed by starch polysaccharides and xanthan gum, is confirmed by the divergence of the forward and reverse curves (both in freshly prepared samples and those subjected to 1 and 2 heat treatment cycles) (Fig. 3, *a*, *b*). During cyclic heat treatment of sauces, there is an increase in viscosity in the series "freshly prepared sauce®sauce after 1 heat treatment cycle®sauce after 2 heat treatment cycles". This is due, on the one hand, to the emergence of a three-dimensional network of hydrogen bonds, the donors and acceptors of which are starch polysaccharides, and, on the other hand, to the high resistance of gelatinized starch dispersions to thermolysis [21].

All the studied samples of sauces show the ability to thixotropic restoration of the structure. Thus, the degree of restoration of the structure of the freshly prepared sauce is 91.4 %, after 1 cycle of heat treatment – 97.8 %, after 2 cycles of heat treatment – 90.3 %.

Along with viscosity, an important rheological indicator is the accumulation modulus, which characterizes the elastic properties of sauces at small deformations (Fig. 3). In the studied samples, the curves of the accumulation modulus (G') dominate over the curves of the loss modulus (G'') in the entire range of angular frequencies, which indicates the predominance of elastic properties over viscous ones. In the freshly prepared sample of sauce, at the point of angular frequency (ω) 62.832 rad/s, the maximum approximation of the curves is observed, which corresponds to the point of equilibrium of the elastic and viscous components of the studied sample. In addition, the phase shift angle (δ) is within 25.12–40.66°, which indicates the viscoelasticity of the system. The loss tangent (tan δ) does not exceed unity and increases from 0.4689 to 0.8589, respectively $0.1<\tan\delta<1$, which reflects the presence of a weak gel structure of the sample. The nature of the G' and G'' moduli curves, as well as the values of the complex viscosity (η*) indicate the presence of a structural network in the studied sample.

The values of the accumulation modulus (*G*') and the loss modulus (*G*'') increase during two-cycle heat treatment, which correlates with an increase in the viscosity of the studied sauce samples. Thus, for freshly prepared sauce, the values of the accumulation modulus (*G*') are in the range of 6.80–18.85 Pa, the values of the loss modulus (*G*'') are 3.19–16.19 Pa. After 1 cycle of heat treatment, the values of the accumulation modulus (*G*') increase to 38.62–99.55 Pa, while the loss modulus (*G*'') increases to 12.08–63.29 Pa. After 2 cycles of heat treatment, the values of the modules increase to 104.2–251.6 Pa and 30.78–108.5 Pa, respectively. Such dynamics indicate the strengthening of intermolecular bonds, an increase in the density of the gel network and the structural stability of emulsions, which is a characteristic feature of viscoelastic gels with a high degree of molecular interaction.

In addition to the above changes, a decrease in the phase shift angle (δ) values is observed after two-cycle heat treatment, which indicates an increase in the elasticity of the samples. A simultaneous increase in the accumulation modulus (*G*') values may indicate a transition from a viscoelastic to a stiffer structure. After two-cycle heat treatment of the sauce, a decrease in the loss tangent (tanδ) values and an increase in the complex shear modulus *G** values are also observed. This indicates an increase in intermolecular interactions, strengthening of the polymer network and the formation of a gel-like matrix. power-law models designed for the frequency sweep curves of the studied sauces

Fitting the dependence of shear rate on shear stress (Table 2) and frequency sweep (Table 3) to rheological models confirms that two-cycle heat treatment leads to an increase in the values of the shear stress and the consistency coefficient with practically unchanged values of the flow behavior index. The obtained results are explained by the redistribution of moisture in the system and its additional binding by starch polysaccharides and xanthan gum. In addition, the tendency of starch polysaccharides to form hydrogen bonds between starch polysaccharide chains (starch retrogradation) is known, which is enhanced by heat treatment. This results in the formation of a gel-like structure of sauces, which is easily destroyed under the influence of shear stress. The flow behavior index, which is a measure of the degree of non-Newtonian behavior, is less than 1 (n<1) for all samples and describes a system that thins out under shear.

The limitation of this work is the uncertainty of the technological parameters of heat treatment of sauces using different techniques and modes of processing in a microwave unit, combi steamer, and hotplate. In addition, food ingredients (whey protein concentrate, xanthan gum, modified waxy corn starch) with certain functional and technological properties were used, which significantly affect the microstructural and rheological parameters of sauces. This is a drawback of the study since the use of raw materials with other parameters could affect both the formation of emulsion systems and their stability.

It would have been also appropriate to investigate the effect of freezing-thawing on the microstructural and rheological parameters of sauces. For these reasons, the prospect of further research is to expand the techniques and modes of heat treatment of sauces in order to substantiate their technological use.

7. Conclusions

1. Studies on the microstructure of sauces have demonstrated a bimodal nature of the particle distribution with two pronounced peaks, which are in the range of 2.42–4.03 μm and 31.10–36.30 μm. The SPAN index is in the range of 1.62–1.72, reflecting moderate polydispersity of both freshly prepared sauces and after two-cycle heat treatment. Heat treatment of sauces contributes to a decrease in the specific surface area and an increase in the values of the volume-surface diameter of particles, which indicates a change in the microstructural characteristics of sauces. The stability of freshly prepared sauces and after 1 and 2 cycles of heat treatment is 98.5±0.5 %, 99.0±0.5 %, and 99.0±0.5 %, respectively, which indicates the stability of the emulsion structure.

2. Sauces are structured food systems that demonstrate typical non-Newtonian behavior. All studied samples exhibit the ability to thixotropic restoration of the structure, which for freshly prepared sauce and after two-cycle heat treatment is 91.4 %, 97.8 %, and 90.3 %, respectively.

During cyclic heat treatment of sauces, an increase in viscosity occurs. This is due, on the one hand, to the emergence of a three-dimensional network of hydrogen bonds, the donors and acceptors of which are starch polysaccharides, and, on the other hand, to the high resistance of gelatinized starch dispersions to thermolysis.

The values of the accumulation modulus (G') and the loss modulus (G'') increase during the 1st and 2nd heat treatment cycles, which indicates an increase in intermolecular interactions and strengthening of the polymer network. Reorganization of the molecular structure of the samples due to heat treatment leads to the formation of a more elastic and stable gel-like matrix.

Conflicts of interest

The authors declare that they have no conflicts of interest in relation to the current study, including financial, personal, authorship, or any other, that could affect the study, as well as the results reported in this paper.

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Data availability

The data will be provided upon reasonable request.

Use of artificial intelligence

The authors confirm that they did not use artificial intelligence technologies when creating the current work.

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