

УДК 664-404.8

## Study of rheological properties of gels based on Thorny Skate cartilage broth (*Raja radiata*) in the technology of natural fish culinary products – aspic fish, in jelly, fish jellies and potions

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### Article info

Received  
26.08.2020;

received in revised  
18.09.2020

### Key words:

Thorny skate,  
chondroitin sulfate,  
gelatin gels,  
rheological properties

### Abstract

The paper deals with methodological approaches to study the rheological properties of gelatin gels based on the thorny skate cartilage broth (*Raja radiata*), which is an aqueous extract of chondroitin sulfate, depending on the manufacturing technology. It is shown that the use of modern instrumental methods for studying the rheological properties of gels and methodological approaches typical of physical and colloidal chemistry allows to obtain the most complete and objective information about the mechanisms of formation of key consumer properties of fish products and the main directions of improving the technology of its manufacture. The influence of gelatin concentration in the range from 0.5 to 5.0 % in chondroitin sulfate-containing broths from the thorny skate cartilage on kinematic and dynamic viscosity of broths, as well as on the strength, melting point and shear stress indices of gels based on these broths was studied. As control samples, aqueous solutions of gelatin in concentrations from 0.5 to 5.0 % and gels based on them were used in the studies. The analysis of the results of the study showed that the broths prepared on the basis of the thorny skate cartilage have increased relative to the control samples the values of kinematic and dynamic viscosity, the increase in indicators is characterized by a directly proportional dependence on the concentration of gelatin in the broth. Gelatin gels based on chondroitin sulfate-containing broths made from the thorny skate cartilage have increased strength, which has a positive effect on the organoleptic evaluation and consumer properties of the finished culinary products.

### For citation

Shokina, Yu. V. et al. 2020. Study of rheological properties of gels based on Thorny Skate cartilage broth (*Raja radiata*) in the technology of natural fish culinary products – aspic fish, in jelly, fish jellies and potions. *Vestnik of MSTU*, 23(3), pp. 302–312. (In Russ.) DOI: 10.21443/1560-9278-2020-23-3-302-312.

## Изучение реологических свойств гелей на основе бульона из хрящей ската звездчатого (*Raja radiata*) в технологии натуральных рыбных кулинарных изделий – рыбы заливной, в желе, рыбных студней и зельцев

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### Информация о статье

Поступила в редакцию  
26.08.2020;

получена  
после доработки  
18.09.2020

### Реферат

Рассмотрены методические подходы к изучению реологических свойств желатиновых гелей на основе бульона из хрящей ската звездчатого (*Raja radiata*). Бульон представляет собой водную вытяжку хондроитинсульфата, массовая концентрация которого зависит от технологии изготовления. Показано, что использование современных инструментальных методов исследования реологических свойств гелей и методических подходов, характерных для физической и коллоидной химии, позволяет получить наиболее полную и объективную информацию о механизмах формирования ключевых потребительских свойств желированных рыбных продуктов. Собранная информация позволяет эффективно определить основные направления совершенствования технологии изготовления желированных рыбных кулинарных продуктов. В работе исследовано влияние концентрации желатина в диапазоне от 0,5 до 5,0 % в хондроитинсульфат содержащих бульонах (ХСБ) из хрящей ската звездчатого с добавлением желатина на их кинематическую и динамическую вязкость. Также исследовано влияние концентрации желатина в ХСБ на прочность, температуру плавления и показатели предельных деформаций сдвига гелей на основе бульонов. В качестве контрольных образцов в исследованиях использовались водные растворы желатина в концентрации от 0,5 до 5,0 % и гели на их основе. Анализ результатов исследования показал, что бульоны, приготовленные на основе хрящей ската звездчатого, обладают повышенными относительно контрольных образцов значениями кинематической и динамической вязкости, нарастание показателей характеризуется прямо пропорциональной зависимостью от концентрации желатина в бульоне. Желатиновые гели на основе ХСБ, приготовленных из хрящей ската звездчатого, обладают повышенной прочностью, что положительно сказывается на органолептической оценке и потребительских свойствах желированных рыбных продуктов.

### Ключевые слова:

скат звездчатый,  
хондроитинсульфат,  
желатиновые гели,  
реологические свойства

### Для цитирования

Шокина Ю. В. и др. Изучение реологических свойств гелей на основе бульона из хрящей ската звездчатого (*Raja radiata*) в технологии натуральных рыбных кулинарных изделий – рыбы заливной, в желе, рыбных студней и зельцев. *Вестник МГТУ*. 2020. Т. 23, № 3. С. 302–312. DOI: 10.21443/1560-9278-2020-23-3-302-312.

## Introduction

As it was stated in the report of the Deputy Minister of Agriculture and the head of Federal Fishery Agency of Russian Federation, I. V. Shestakov, titled "Results of the FFA Activity and subsequent tasks for 2018", the resources for which the total allowable catch is not set are at a low loss of fishing. The industrial fishing of the non-quota objects in the Russian Federation is now at a rate very far from the recommended level, which is 1.7 million tons, taking into account the total catch of 500 thousand tons. The situation in the current year 2020 changes little, despite the key provisions of the Decree of the government of the Russian Federation of March 30, 2018 "On amendments to the State Program of the Russian Federation for the Development of the Fisheries". Thus, sub-program no. 7 "On improving the efficiency of use and development of the fish farming" calls one of its main tasks the expansion of the resource base of fishing by expanding its species composition. Thorny skate belongs to the category of fishing objects without a total allowable catch; these types of marine resources have a significant reserve potential for development, since 2/3 of the recommended catch level cannot be reached within the last five years<sup>1</sup> (Pauly *et al.*, 2001; Krichen *et al.*, 2018; Raibulov *et al.*, 2016).

In view of the state support for the fishing of undeveloped resources, it is advisable to use these industrial facilities in the manufacture of culinary products that are in rapidly growing, stable demand among the population.

Due to the peculiarities of the morphological structure of cartilaginous fish, their body retains salts and urea in tissue fluids. During processing, it is necessary to ensure effective removal of urea, since its high content negatively affects the organoleptic characteristics of the product. The threshold of human sensitivity to this compound corresponds to a mass fraction of 1.2 % (Skachkov, 1975). In view of the above, it became necessary to apply various technological methods to reduce the urea content in the product to values below the threshold of human sensitivity.

Products with functional properties, as well as the use of fish with a cartilaginous skeleton, were comprehensively studied by Korchunov V. V., Petrov B. F., Ershov A. M., Shokina Y. V., Sayenkova I. V., Pavlova V. V., Shchetinsky V. V., and Raibulov S. P. (Shokina *et al.*, 2014; Shchetinsky *et al.*, 2012; Grekov *et al.*, 2011; Raibulov *et al.*, 2016). Scientists have developed a range of technologies for removing urea of various effectiveness from the most culinary valuable skate wings, which make up 75 % of edible part. Due to the high content of chondroitin sulfate (CS), from 220 to 280 mg% (Shokina *et al.*, 2014; Miraglia *et al.*, 2016), skate wings can be considered as sources of CS in food. Research of many scientists (Fedorovykh *et al.*, 2014) in relation to cholesterol proven chondroprotective and angioprotective action (Fedorovykh *et al.*, 2014; Lago *et al.*, 2012; Krichen *et al.*, 2017; Korchunov, 2004). The nutritional and biological value of CS and glucosamines is also confirmed by the fact that in 2013 these compounds were included in the list of minor food substances, for which the daily consumption rate was set at 700 mg<sup>2</sup>.

Taking into account the above, the actual research task is to develop a technology for fish culinary products of mass consumption based on cartilage and meat of skate wings. The main requirement for the developed technology is to ensure the content of CS in finished products from 105 to 350 mg%, which will allow classifying them as functional nutrition products aimed at preventing socially significant diseases of the human musculoskeletal system.

The purpose of the research is to provide scientific justification and develop the technology of natural fish products – fish jellies and the wings of thorny skate.

These are tasks formulated to achieve this goal at the first stage of research:

- development of aspic technology – a semi-finished product for natural fish culinary products of a wide range;
- scientific justification of the use thorny skate wings for making aspic in the technology of natural fish culinary products of a wide range;
- study of the dependence of the rheological properties of chondroitin sulfate-containing broths (CSB) based on thorny skate wings on the concentration of food – grade industrial gelatin added to the broth according to GOST 11293-89 "Gelatin. Technical specifications (with change no. 1)"<sup>3</sup>;
- study of rheological properties of gelatin gels based on CSB of thorny skate wings;
- formulation of the direction of further research.

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<sup>1</sup> Federal Agency for fisheries [Electronic resource]. Report of the Deputy Minister of agriculture of the Russian Federation – head of the Federal Agency for fisheries Ilya Shestakov at an expanded meeting of the Board of the Federal Agency for fisheries "Results of the Federal Agency for fisheries in 2015 and tasks for 2016". URL: <http://fish.gov.ru/press-tsentr/vystupleniya-i-intervyu-rukovodstva/12872-doklad-zamestitelya-ministra-selskogo-khozyajstva-rossijskoj-federatsii-rukovoditelya-federalnogo-agentstva-po-rybolovstvu-ili-vasilevicha-shestakova-na-rasshirennom-zasedanii-kollegii-rosrybolovstva-itogi-deyatelnosti-federalnogo-agentstva-po-rybolovstvu>.

<sup>2</sup> Norms of physiological needs in energy and food substances for various groups of the population of the Russian Federation. Methodological recommendations: Rational nutrition [Electronic resource]: MR 2.3.1.2438-08. Approved by the Feder. services for supervision of consumer rights protection and human welfare, 18 Dec. 2008. URL: <http://docs.cntd.ru/document/1200076084>.

<sup>3</sup> GOST 11293-89. Gelatin. Technical specifications (with change no. 1). Moscow, 2001.

### Objects and methods of the study

The objects of research were experimental samples of fish broths based on the cartilage of thorny skate prepared with the addition of food gelatin of industrial production according to GOST 11293-89 in concentrations from 0.54 to 5.0 %, as well as gels based on them; control samples are aqueous solutions of gelatin concentrations from 0.5 to 5.0 % and gels based on them; rheological properties of CSB are kinematic and dynamic viscosities; rheological properties of gelatin gels based on CSB are gel strength, gel melting point and the indicators of ultimate shear deformations of gels.

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The used research methods are the following:

- organoleptic (evaluation of prototypes at a scale developed by taking into account the weight coefficients of indicators) according to GOST 7631-2013<sup>4</sup>;
- physical – density determination of the broth in the "Laktan 1–4" device by the developed technique, considering the coefficient of correlation established by the calibration methods;
- physical-chemical determination of the mass fraction of protein (P) and fat (F) on the "Laktan 1–4" device by the developed technique considering the coefficient of correlation established by the calibration methods (GOST 7636-85<sup>5</sup>);
- determination of kinematic viscosity using a viscometer according to GOST 10028-81<sup>6</sup>;
- determination of dynamic viscosity of broths by calculation;
- determination of gel strength via the Valenta device (*Antipova et al., 2004*);
- determination of the melting temperature of the gel by determining the limit shear deformations on the rheometer "Physica MCR 302" with a measuring cone-plate cell with a fixed value of the deformation amplitude and frequency (*Panagos et al., 2014*);
- determination of emulsifying capacity (EC) by centrifuging a mixture of broth with sunflower oil in a calibrated test tube at a centrifuge speed of 500 rpm, followed by determining the volume of emulsified oil;
- determination of emulsion stability (ES) by heating a mixture of broth with sunflower oil at a temperature of 80 °C for 30 minutes, cooling with water for 15 minutes, and then centrifuging the mixture in a calibrated test tube to determine the volume of the emulsified layer (*Antipova et al., 2004*).

### Results and discussions

For the production of semi-finished products of natural fish culinary products, a technological scheme for preparing aspic from the cartilage of the thorny skate, shown in Fig. 1, has been developed.

The main stages of processing fish raw materials in the proposed technological scheme correspond to the basic generally accepted technology for preparing fish aspic. An innovative stage is the primary treatment of thorny skate wings, which includes blanching, designed to reduce the mass fraction of urea in the raw material as a result of its thermal decomposition at temperatures above 60 °C (*Shokina et al., 2014; Shchetinsky et al., 2012*).

New technological operations that distinguish the developed technology from the basic one are highlighted in Fig. 1 with a colored dotted line.

The skate was cut up manually, and the fish's head, fins, wings, and tail fin were removed, as well as their insides. The wings of the skate were thoroughly washed in running water, after which they were immersed in water with a temperature of 96 to 98 °C for 1 minute. Then the blanched wings were cooled to a temperature no higher than 45 °C at a room temperature via air. The skin (waste) and meat (food production material) were separated from the cartilage, which was then sent to the preparation of CSB. To prepare the broth, cartilage was filled with cold water in the ratio of cartilage and water mass 1 : 1 and cooked at a low boil (at a temperature of 90 to 95 °C) for 1.5 hours with a single top-up of water to the initial volume. Then the broth was cooled to a temperature no higher than 60 °C, filtered, degreased, added salt and spices according to the recipe, brought to a boil and boiled for 5 minutes. Part of the broth was cooled to room temperature and used for soaking gelatin. Soaked gelatin in the amount set by the recipe (experimental scheme) was combined with the rest of the broth, then 80 % acetic acid was added according to the recipe and dissolved when heated to a temperature of 80 to

<sup>4</sup> GOST 7631-2013. Fish, non-fish objects and products from them. Methods of sensory and physical characteristics identification. Moscow, 2010.

<sup>5</sup> GOST 7636-85. Fish, marine mammals, invertebrates and products of their processing. Methods of analysis. Moscow, 2010.

<sup>6</sup> GOST 10028-81. Glass capillary viscosimeters. Specifications. Moscow, 2005.

85 °С. Then the broth was cooled to a temperature no higher than 20 °С, after which it was sent to solidify in a medium-temperature refrigerator at a temperature of 4 to 6 °С.

Henceforth, prototypes of aspic were subjected to investigations in accordance with the developed plan.

At the first stage of research, the greatest interest is to study the functional and technological properties (FTP) of broth prepared according to the technology (Fig. 1), which is an aqueous extract of CS contained in large quantities in the cartilage. Among the FTP of fish broth are the most important rheological indicators that will determine in the future such important consumer properties of ready-made culinary products as consistency and melting point.

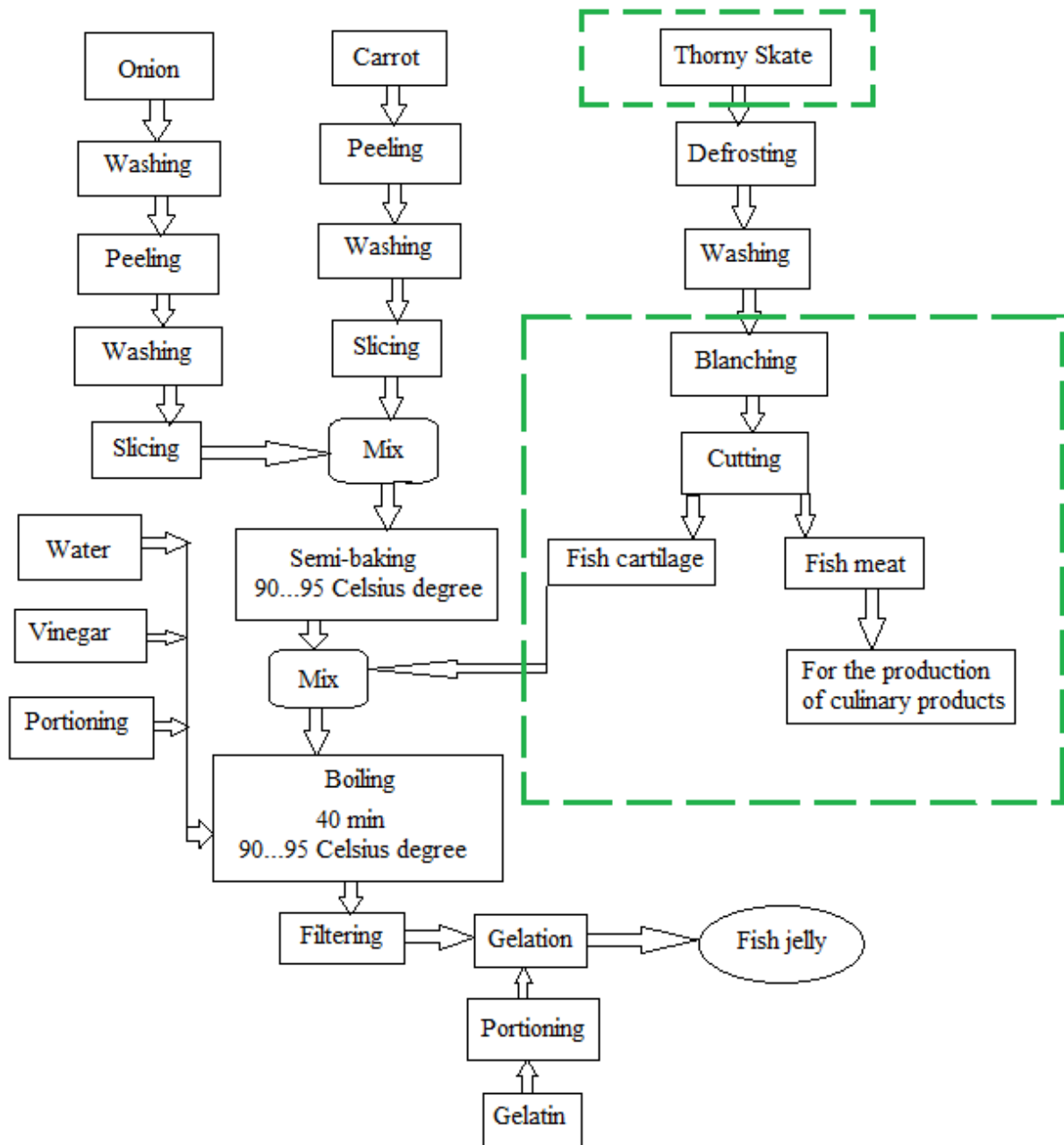


Fig. 1. Technological scheme of production of semi-finished product (aspic) for natural fish product from Thorny Skate (fish jelly)

Рис. 1. Технологическая схема изготовления полуфабриката (заливного) для натурального рыбного продукта из ската звездчатого (рыбное желе)

Rheological properties of experimental CSB broth from thorny skate wing cartilage were determined by kinematic and dynamic viscosity, emulsion stability, and emulsifying ability. In parallel, the chemical composition, the hydrogen index, and the density of the broth were studied using an instrumental method (using the "Lactane 1-4" device). The results of the definitions are shown in Figs 2-6.

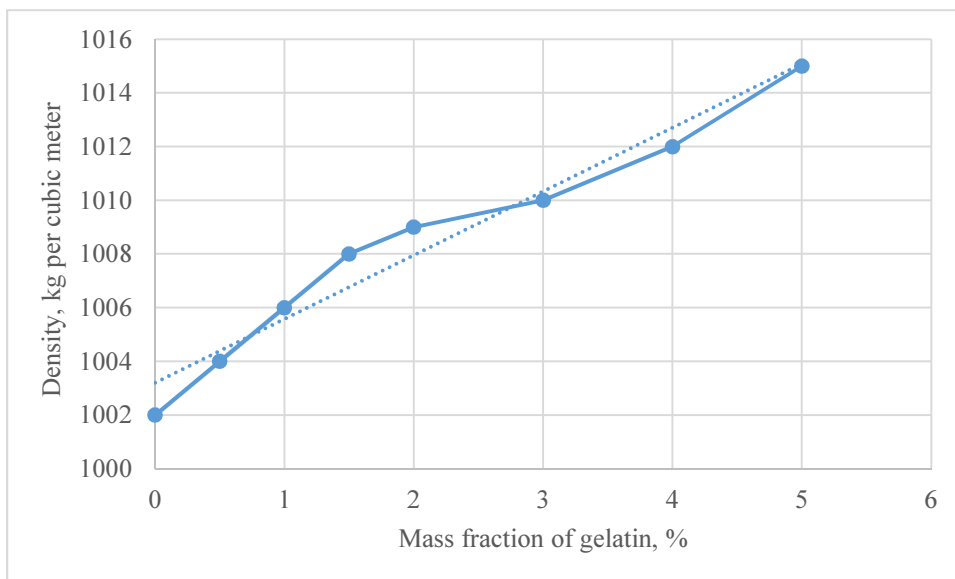


Fig. 2. Results of determining the density of CSB from Thorny Skate cartilage with the addition of food gelatin on the "Lactane 1-4" device (the density value is obtained by multiplying the measurement result on the device by the correlation coefficient  $r = 0.8312$ , established experimentally using a verification technique)

Рис. 2. Результаты определения плотности ХСБ из хрящей ската звездчатого с добавлением пищевого желатина на приборе "Лактан 1-4" (значение плотности получено умножением результата измерения на приборе на коэффициент корреляции  $r = 0,8312$ , установленный экспериментально с использованием поверочной методики)

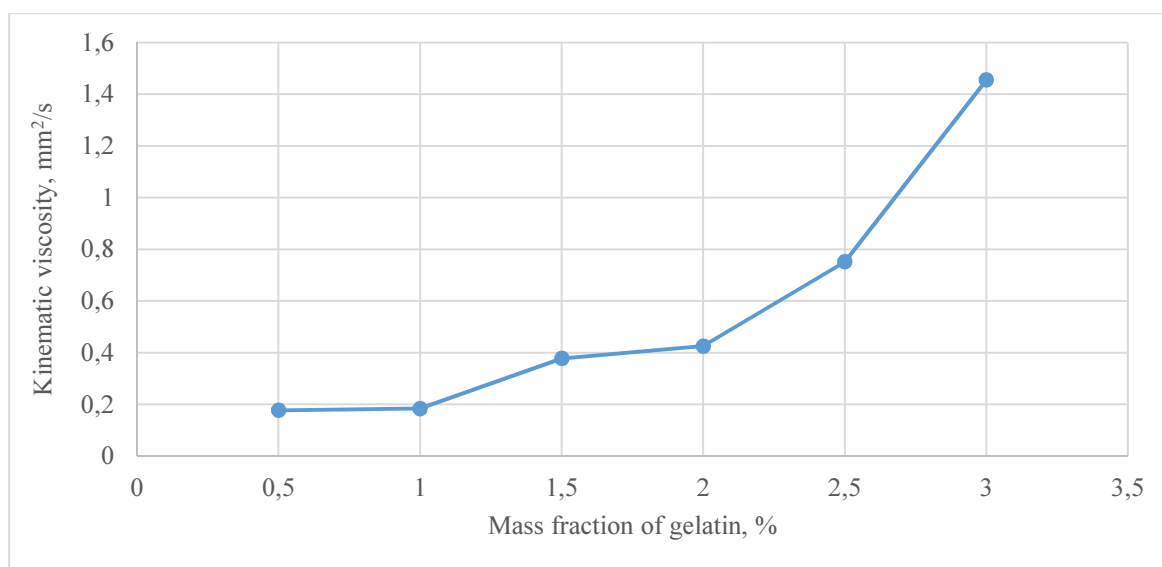


Fig. 3. Results of determining the kinematic viscosity of CSB from the cartilage of Thorny Skate with the addition of food gelatin using a viscometer

Рис. 3. Результаты определения кинематической вязкости ХСБ из хрящей ската звездчатого с добавлением пищевого желатина при помощи вискозиметра

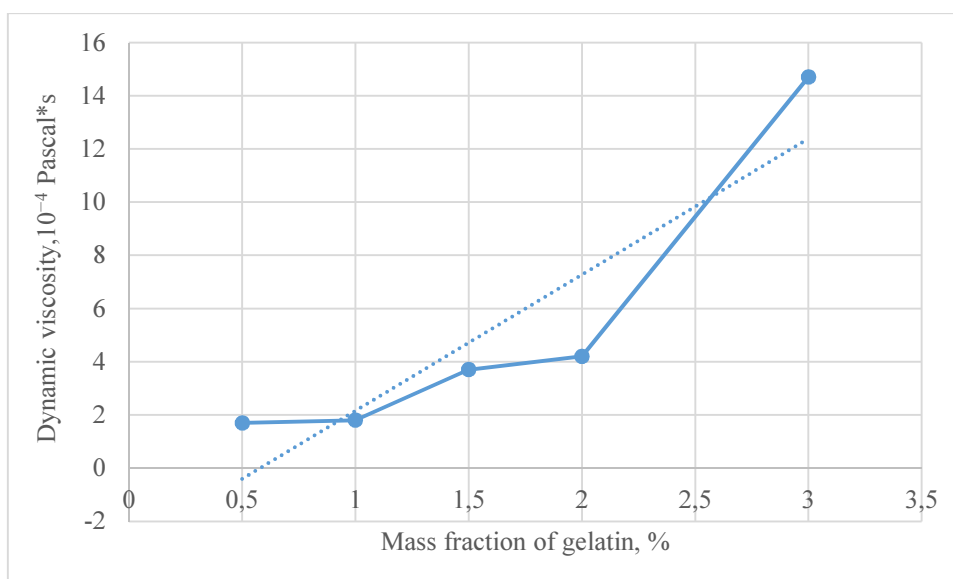


Fig. 4. Results of determining the dynamic viscosity of CSB from Thorny Skate cartilage with the addition of food gelatin using the calculation method (using the formula  $\mu = \nu \cdot \rho$ , where  $\nu$  is the kinematic viscosity,  $m^2/s$ ;  $\rho$  is the density,  $kg/m^3$ ;  $\mu$  is the dynamic viscosity, Pascal·s)

Рис. 4. Результаты определения динамической вязкости ХСБ из хрящей ската звездчатого с добавлением пищевого желатина расчетным методом (по формуле  $\mu = \nu \cdot \rho$ , где  $\nu$  – кинематическая вязкость,  $m^2/c$ ;  $\rho$  – плотность,  $кг/м^3$ ;  $\mu$  – динамическая вязкость, Па·с)

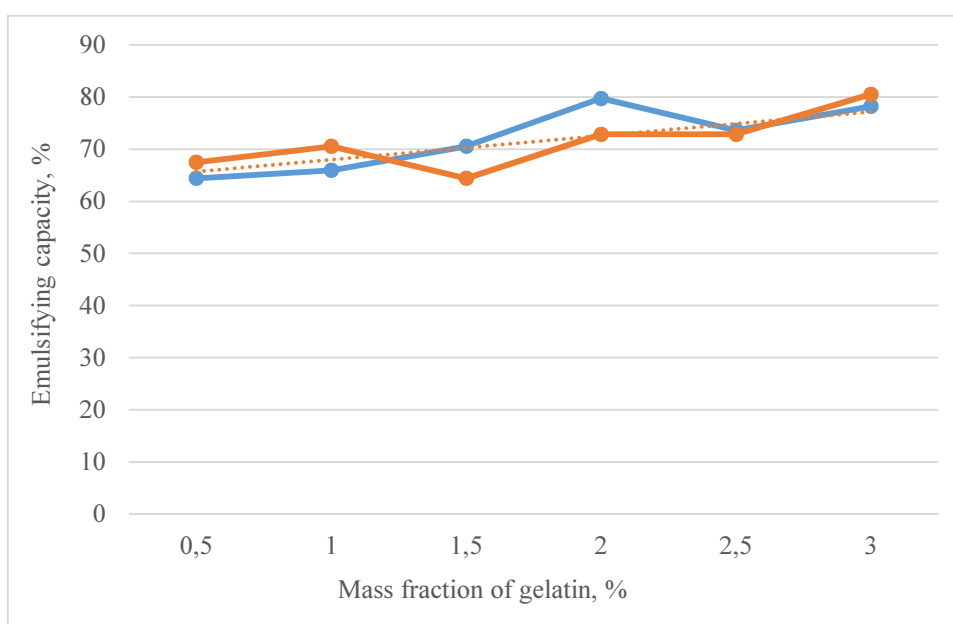


Fig. 5. Results of EC CSB determination from Thorny Skate cartilage with the addition of food gelatin: blue – control; red – broth with gelatin

Рис. 5. Результаты определения ЭС ХСБ из хрящей ската звездчатого с добавлением пищевого желатина: синяя линия – контроль, красная линия – бульон с желатином

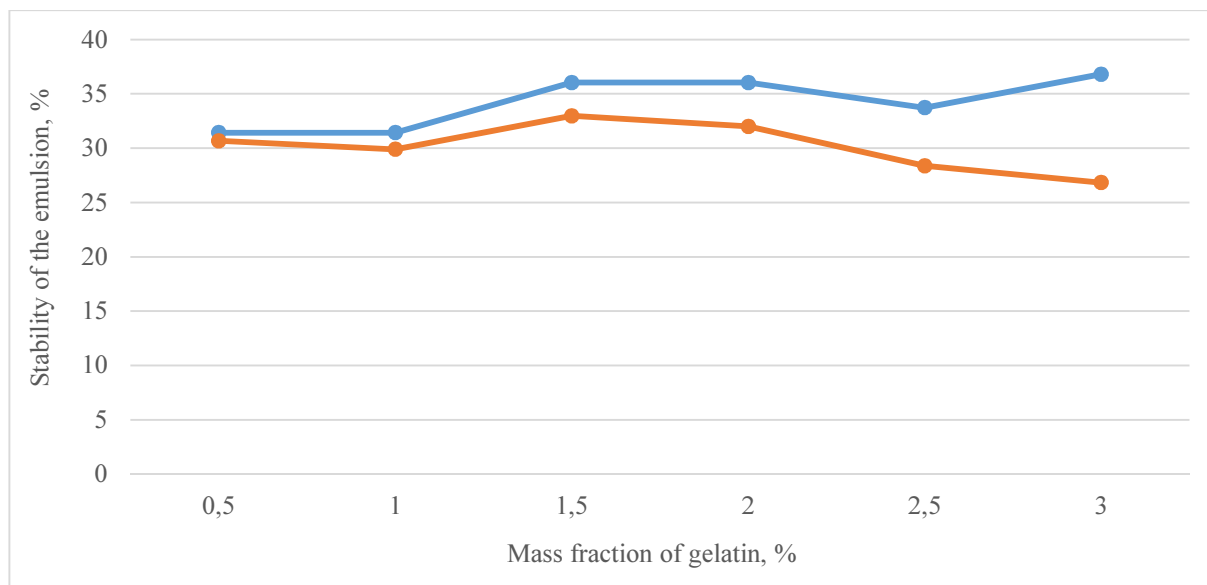


Fig. 6. Results of determination of SE CSB from Thorny Skate cartilage with the addition of food gelatin: blue – control; red – broth with gelatin

Рис. 6. Результаты определения СЭ ХСБ из хрящей ската звездчатого с добавлением пищевого желатина: синяя линия – контроль, красная линия – бульон с желатином

Analysis of the data presented in the figures showed that the addition of food gelatin in concentrations from 0.5 to 5.0 % increases the density, kinematic and dynamic viscosity of CSB (in the figures up to 3.5 % MD gelatin) from the cartilage, which should have a positive effect on the consumer properties of gelled fish culinary products.

Thus, the kinematic viscosity of broth with a maximum concentration of food gelatin of 3.0 % in the studied range is more than 8 times higher than that of broth with a minimum concentration of gelatin of 0.5 %. At the same time, the increase in the broth density in the specified range is only slightly more than 1 % (1.09 %).

The calculated dynamic viscosity increases by more than an order of magnitude in the range from the minimum (0.5 %) to the maximum (3.0 %) concentration of food gelatin in the broth.

However, as the experiment showed, the addition of food gelatin in various concentrations to CSB practically does not affect their emulsifying ability and the index of emulsion resistance. Figs 5 and 6 show that changes in the EC and ES parameters of broths fluctuate slightly with changes in the concentration of food gelatin – within the margin of error of the experiment.

The obtained data on changes in the rheological properties of CSB when adding food gelatin should be correlated with data on the properties of gels based on them.

Table 1 shows the results of determining the strength of gels based on CSB from thorny skate cartilage with different concentrations of added food gelatin. The study was conducted on the valent device. This technique is based on pushing the gel layer with a mushroom-shaped nozzle of a known contact area until the surface layer breaks, and determining the final load mass (Antipova et al., 2004).

Table 1. The results determine the strength of gelatin gels based on HSB of Thorny Skate cartilage

Таблица 1. Результаты определения прочности желатиновых гелей на основе ХСБ из хрящей ската звездчатого

Concentration of gelatin, %	Gel strength, g/sm <sup>2</sup>	
	gelatin gels based on CSB	control (gelatin gels water-based)
0	no gelation	–
0,5	less than 101.5	less than 101.5
1	less than 101.5	less than 101.5
1,5	less than 101.5	less than 101.5
2	101.5	less than 101.5
3	117.9	132.0
4	137.7	143.8
5	167.7	160.6

As it can be seen from the graph in Fig. 7, with increasing gelatin concentration, the value of the "gel strength" indicator gradually increases, reaching a maximum value of 167.7 g/cm<sup>2</sup> with a mass fraction of 5 % gelatin. It is important to note that in the absence of gelatin, there are no gelation processes in CSB.

The melting temperature of the gel was determined by studying the rheological indicator-limit shear deformations. The melting point was determined at the intersection of the loss modulus and accumulation modulus curves.

Fig. 7 shows the curves of the conservation and loss modulus for gels with a mass fraction of the gelatin 1 %, and Table 2 shows the results of determining the melting point of gels for all the studied concentrations of gelatin in CSB.

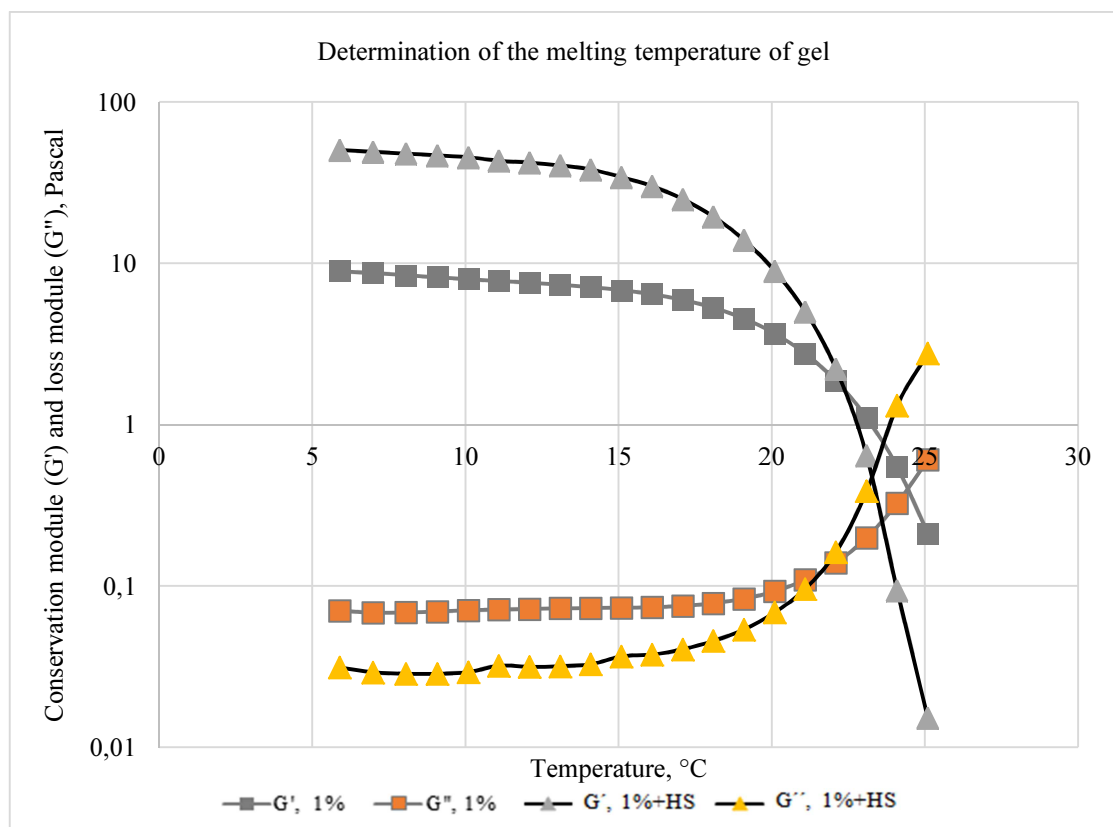


Fig. 7. Indicators of limiting shear deformations of jellies with a mass fraction of gelatin 1 %  
 Рис. 7. Показатели предельных деформаций сдвига студней с массовой долей желатина 1 %

Table 2. Melting temperature of gelatin gels  
 Таблица 2. Температура плавления гелей желатина

Concentration of gelatin, %	Melting temperature, °C	
	control (gelatin gels water-based)	gelatin gels based on CSB
0.5	24.9	24.6
1.0	25.8	25.5
1.5	27.7	26.3
2.0	28.5	27.8
3.0	29.3	28.6
4.0	29.9	29.7
5.0	31.9	30.0

After analyzing the table, it can be concluded that the melting temperatures of CSB-based and water-based gels practically coincide with a slight deviation of the indicator in the lower direction for CSB-based gels.

Based on the results of comprehensive research, it is possible to make a reasonable conclusion about the feasibility and possibility of using secondary food waste from processing thorny skate wings such as cartilage for making aspic.



Aspic based on CSB can be used as a semi-finished product in the technology of production of natural functional fish culinary products of a wide range, aimed at preventing diseases of the human musculoskeletal system.

Further research should clarify the mechanism of interaction of the polysaccharide chondroitin sulfate with the protein gelatin to determine the optimal concentrations of the latter, providing the maximum organoleptic assessment of fish gelled culinary products and its best consumer properties.

### Conclusion

Based on the results of the study, the following conclusions are made:

1. Aspic technology has been improved taking into account the specific techno-chemical properties of raw materials from thorny skate.

2. The use of thorny skate wings for the production of aspic in the technology of natural fish culinary products is scientifically justified.

3. The comprehensive innovative study was conducted of the dependence of the rheological properties of CSB from thorny skate wings on the concentration of mass-produced (industrial) gelatin added to the broth (GOST 11293-89<sup>7</sup>). According to the results of the study, an increase in the kinematic and dynamic viscosity indicators was bound with an increase in the gelatin concentration in the range from 0.5 to 5.0 %, the maximum values are reached when the gelatin concentration in the broth is 5.0 %.

4. The rheological properties of gelatin gels based on CSB were studied for the first time. According to the results of research, an increase in the "gel strength" indicator was found with an increase in the concentration of food gelatin added to the broth in the range from 0.5 to 5.0 %, which positively affects the consistency of the finished fish gelled culinary product.

5. The optimal concentration of food gelatin 5.0 % can be recommended for the preparation of broth (which is the basis of aspic) in the technology of natural fish culinary products enriched with chondroitin sulfate.

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