

A STUDY OF PROPERTIES OF STRUCTURE-STABILIZING AGENTS FOR PRODUCTS BASED ON DAIRY RAW MATERIALS

O. V. Kozlova

Kemerovo Institute of Food Science and Technology,
bul'v. Stroitelei 47, Kemerovo, 650056 Russia,
phone: +7 (3842) 39-68-88, e-mail: kozlovaov79@rambler.ru

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Abstract: An important strategic objective of the food industry is to meet the demands of all categories of the population in high-quality, biologically valuable, and safe food. At present, structured products based on dairy raw materials are commonly used. Food materials used to generate the necessary rheological properties or modify the existing properties for food systems that adjust or form the consistency of food products are referred to as structure-stabilizing agents. Both individual ingredients and complex mixtures thereof can be structure-stabilizing agents. The use of these agents contributes to an increase in the density and to the formation of a specific structure of the food product, which is retained even after heat treatment. In this study, the composition and properties of structure-stabilizing agents for products based on dairy raw materials have been analyzed. The following characteristics have been selected to examine: bulk density, viscosity, the fraction of undissolved residue, microvoid content, specific volume, specific surface area, and characteristic diameter. The dynamics of structural changes that occur during the interaction with the solvent have been analyzed from micrographs. The weight fraction of chemical elements (oxygen, nitrogen, carbon, sodium, chlorine) has been determined via analyzing the spectrophotometric profile.

Keywords: structure-stabilizing agents, modification, structure, dairy products, spectrophotometric analysis

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INTRODUCTION

At present, the production of structured products based on dairy raw materials is one of the most dynamically developing industrial sectors [6]. The quality of dairy products, along with other parameters, is determined by their structure and consistency, which depend on the proper implementation of the production process. The development of modern technological principles of food production is associated with the search for universal methods for impacting multicomponent aqueous systems; one of these methods is the use of food structure-stabilizing agents [7]. The role of structure-stabilizing agents is to provide a good consistency, which does not change during storage and exhibits stability during the process implementation.

The production of stabilized products based on dairy raw materials is characterized by the structure formation owing to the coagulation of casein at the isoelectric point [8]. In this case, the stabilizing agents used in food production must hinder the separation of the product and the isolation of whey; when using fillers and additives, structure-stabilizing agents must provide the uniformity of their distribution over the volume during packing and subsequent storage. The use of structure-stabilizing agents can provide the formation of a gel structure at pH values other than the isoelectric point [1]. Moreover, using structure-stabilizing agents, it is possible to targetedly adjust the structural-mechanical and physicochemical properties, organoleptic parameters, and quality of the end product [3].

Stabilizing systems are commonly used in the dairy industry [9]. Among the currently known structure-stabilizing agents, anionic polysaccharides—both natural (pectin, agar, agaroid, pyrophosphate) and artificial (oxidized starch)—are extensively employed. Alginates, cellulose derivatives, carboxymethyl cellulose (CMC), and various gums enjoy wide application abroad [4]. Stabilizing agents are promising because their composition and properties are constantly being improved. The use of stabilizing agents improves the quality of products (particularly consistency) and increases the yield and the shelf life [2].

Examination of the structure formation laws in dairy systems is of significant theoretical and practical importance because it provides the formation of disperse systems with desired structural-mechanical properties.

The described studies were conducted in conjunction with A.N. Arkhipov and A.V. Pozdnyakova.

OBJECTS AND METHODS OF RESEARCH

To examine the composition of the structure-stabilizing agents, a JEOL JED-2300 analysis station was used; the electron probe microanalysis conducted with this instrument yielded spectrometric profiles that can be used to determine the chemical composition of the structure-stabilizing agents [5].

The objects of research were structure-stabilizing agents with varying bulk density, which are presented in Table 1.

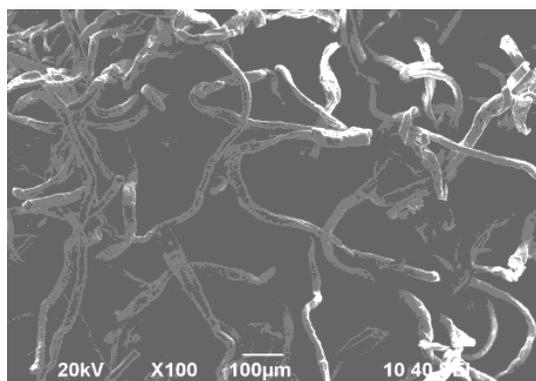
Table 1. Bulk density of the studied structure-stabilizing agents

Structure-stabilizing agent	Bulk density, g/dm ³
CMC (carboxymethyl cellulose) Akucell 3265	450
Konjac gum	540
Pectin ARA 105	580
CMC Akucell 2785	680
Xanthan gum	830

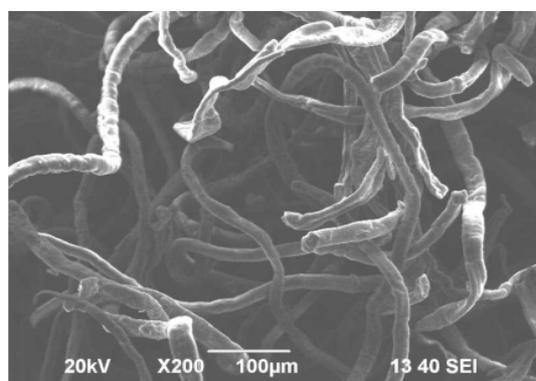
RESULTS AND DISCUSSION

CMC Akucell 3265

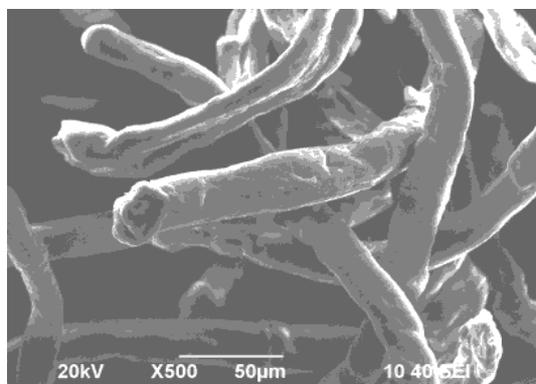
Figure 1 shows micrographs of CMC Akucell 3265 at a magnification of 100, 200, and 500×.



(a)



(b)

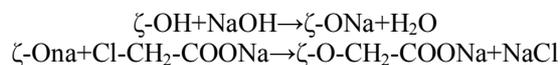


(c)

Fig. 1. Microstructure of CMC Akucell 3265 at a magnification of (a) 100, (b) 200, and (c) 500×.

The micrographs in Fig. 1 show that the structure of CMC Akucell 3265 is composed of dispersed elements in the form of elongated fibers with a rough surface and a diameter of about 20–30 µm. CMC Akucell 3265 has the lowest bulk density of all the studied structure-stabilizing agents; it is 450 g/dm³.

CMC is a salt of a weak carboxylic acid produced through a reaction between sodium monochloroacetate and alkaline cellulose according to the following schemes:



To prepare this structure-stabilizing agent, α -cellulose derived from plant fibers is exposed to a strong alkaline solution and chloroacetic acid; the resulting sodium chloride is washed out. The composition of CMC comprises sodium chloride, sodium glycolate, and unsubstituted cellulose.

Each anhydropryanose unit of the CMC molecule contains three OH groups capable of reacting with sodium monochloroacetate. The substitution of all three OH groups is theoretically possible (the degree of substitution is three); however, the degree of substitution in real CMC samples is 0.4–1.2. At a degree of substitution of 0.5 and 0.8, the pK values of the carboxyl groups are 4.0 and 4.4, respectively. At a pH level of 7.0 and 5.0, about 90 and 10% of the carboxyl groups are in a salt form. CMC is an ionogenic cellulose ester; therefore, its stabilizing effect depends on salt concentration and other properties of the medium.

The highest viscosity of equiconcentrated CMC solutions is observed at a pH of 6–8 (Fig. 2).

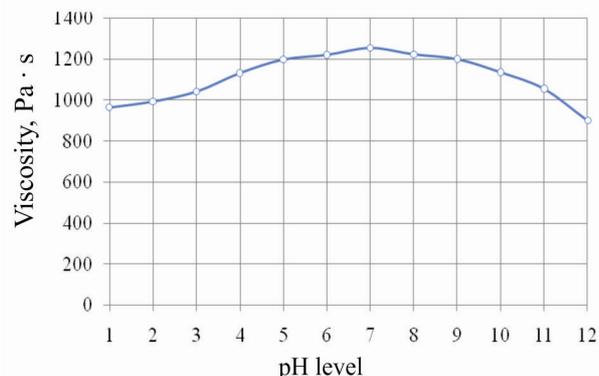


Fig. 2. Dependence of the viscosity of equiconcentrated CMC solutions on pH value.

Most probably, in this pH range, the macromolecules undergo unfolding owing to the electrostatic repulsion of the ionized carboxyl groups. At low pH values, the ionization of the carboxyl groups in an acid form is suppressed, while at high pH values, the repulsive forces decrease because of the presence of a large amount of sodium ions. This fact suggests that the CMC macromolecule is folded at extreme pH levels and unfolded in an optimum pH range of 6–9 (Fig. 3).

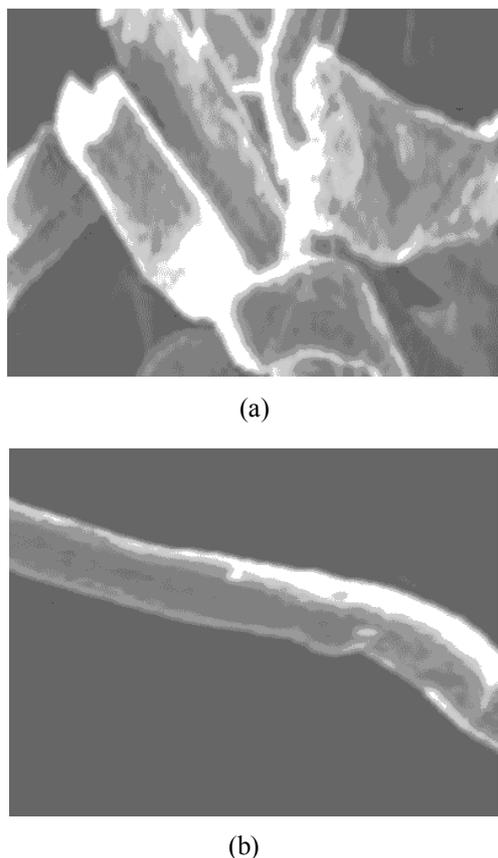


Fig. 3. (a) Folded and (b) unfolded CMC macromolecule at a magnification of 5000 \times .

The solubility and processing properties of CMC are affected by the chain length and the degree of substitution and neutralization. Thus, in the presence of

1% CMC in an aqueous solution, the viscosity varies from 20 to 3500 Pa \cdot s. CMC exhibits high solubility in water and alkalis; it is poorly soluble in glycerol and acids and almost insoluble in organic solvents (Fig. 4).

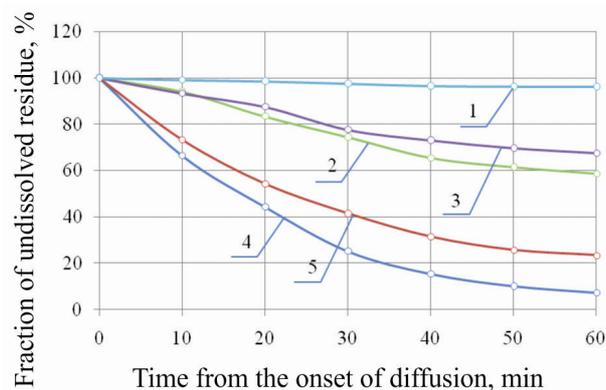


Fig. 4. Dependence of the undissolved residue of CMC on the time of treatment in (1) organic solvents, (2) acids, (3) glycerol, (4) water, and (5) alkali.

The solubility of CMC can be improved by using more active solvents, one of which is a diluted solution of sodium hydroxide, which makes it possible to increase the degree of substitution to values close to 1.2.

Figure 5 shows the spectrometric profile of the composition of CMC Akucell 3265. The derived profile exhibits three characteristic peaks corresponding to carbon, oxygen, and nitrogen and two plateaus attributed to nitrogen and chlorine.

Table 2 lists the component composition of CMC Akucell 3265.

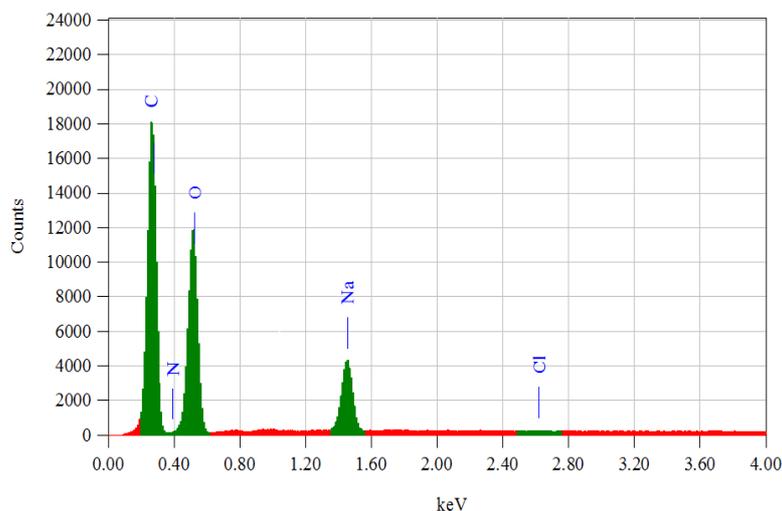


Fig. 5. Spectrometric profile of the composition of CMC Akucell 3265.

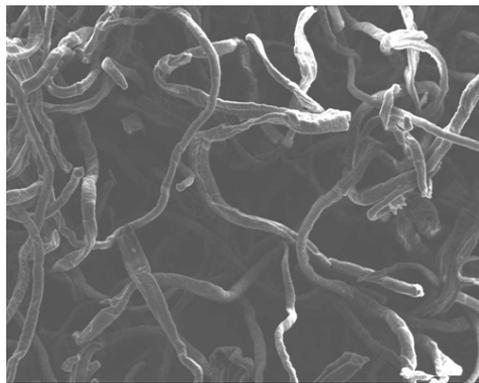
Table 2. Component composition of CMC Akucell 3265

Element	Relative content, wt %
Carbon	31.15 \pm 0.93
Nitrogen	21.90 \pm 0.65
Oxygen	42.78 \pm 1.28
Sodium	4.01 \pm 0.12
Chlorine	0.07 \pm 0.002

The results show that oxygen is the dominant component of CMC Akucell 3265 (42.78%). Chlorine content is the lowest (0.07%).

The microvoid content in CMC Akucell 3265 was determined using the micrograph shown in Fig. 1a. In this case, it was necessary to filter the background elements, which was implemented by enhancing the contrast and manually correcting the mask. The results of determina-

tion of the microvoid content in CMC Akucell 3265 are shown in Fig 6.



(a)



(b)

Fig. 6. Results of determination of the microvoid content in CMC Akucell 3265: (a) a micrograph at a magnification of 100×, (b) a mask of the micrograph shown in panel (a).

According to the histogram, the microvoid content in CMC Akucell 3265 was $51.24 \pm 2.2\%$.

Thus, the microstructure of CMC Akucell 3265 is characterized by a low bulk density of 450 g/dm³; its elements have the form of elongated fibers with a diameter of 20–30 μm. The chemical composition of CMC Akucell 3265 comprises carbon, nitrogen, oxygen, sodium, and chlorine. The microvoid content in the studied structure-stabilizing agent was $51.24 \pm 2.2\%$.

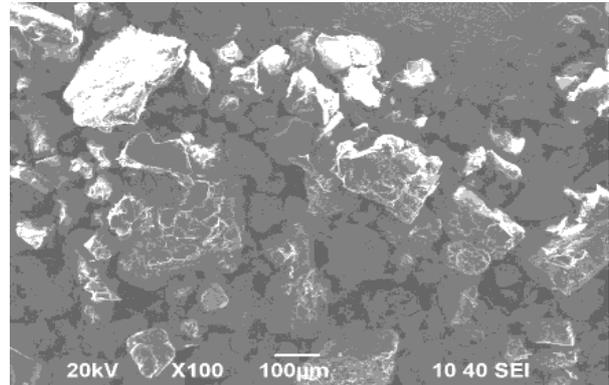
Konjac Gum

Figure 7 shows the microstructure of konjac gum at a magnification of 100, 200, and 500×. The bulk density of this structure-stabilizing agent is 540 g/dm³. It is evident from Fig. 7a that the microstructure of konjac gum is composed of large and small irregularly shaped granules, the size of which varies from 10 to 250–300 μm. The microstructure comprises granules with both a rough crystalline surface and a smooth surface (Fig. 7c).

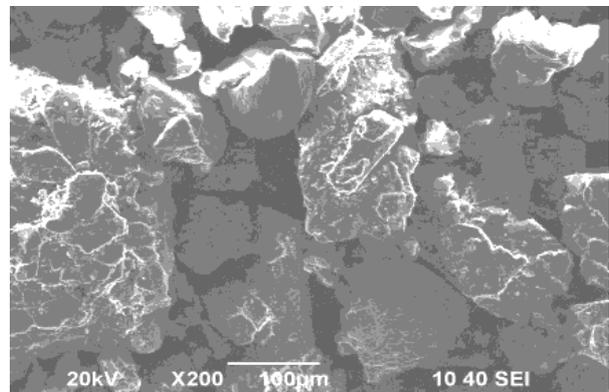
Dispersion of konjac gum in hot and cold water results in the formation of highly viscous solutions with a pH of 4–7 (Table 3).

As the konjac gum concentration increases, the pH

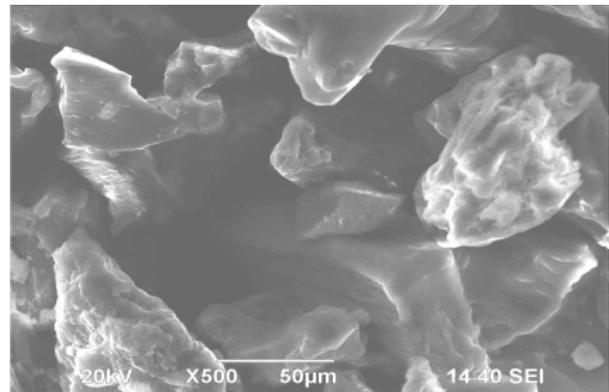
level of the aqueous solution decreases. The solubility increases during stirring and heating. This structure-stabilizing agent is composed of D-mannose and D-glucose monomers linked by a β-glycosidic bond.



(a)



(b)



(c)

Fig. 7. Microstructure of konjac gum at a magnification of (a) 100, (b) 200, and (c) 500×.

Table 3. pH level of an aqueous solution of konjac gum

Weight fraction of konjac gum in the solution	pH
0.5	6.7
1.0	5.9
1.5	5.4
2.0	4.7
2.5	4.1

The solubility of konjac gum in hot water is higher than in cold water, as evident from the dependence of the fraction of dry residue of konjac gum in an aqueous solution derived after 60-min diffusion at different temperatures (Fig. 8).

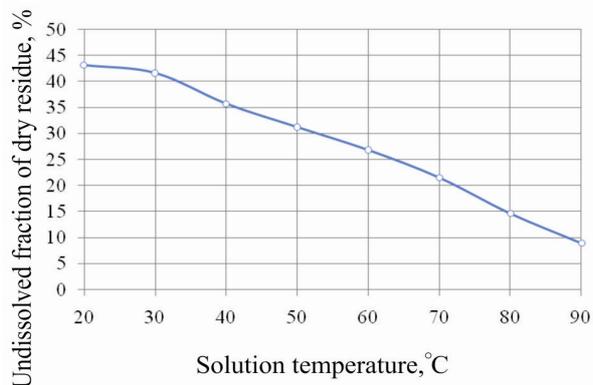


Fig. 8. Undissolved fraction of the dry residue of konjac gum in an aqueous solution in 60 min after the onset of diffusion at varying temperature.

This structure-stabilizing agent is insoluble in organic solvents. A distinctive feature of this structure-stabilizing agent is a high level of viscosity even at low concentrations. The main component in the composition of this material is chitosamin, which is responsible for the dissolution of the structure-stabilizing agent particles in water. In addition, the composition of konjac gum comprises alcohol, which is involved in the precipitation and drying of glucomannan.

In the food industry, konjac gum is commonly used as a fat substitute in the production of fat-free and low-fat meat products. Furthermore, it contributes to the formation of a desired consistency in the production of jellies, puddings, beverages, yogurts, etc.

Figure 9 shows a spectrometric profile for determining the composition of konjac gum; the component composition is listed in Table 4.

Konjac gum contains carbon, nitrogen, oxygen, potassium, and sodium. The content of carbon and oxygen is similar to that observed in CMC 4500-6000. Konjac gum differs from the previously discussed structure-stabilizing agents in the presence of potassium and the absence of sodium.

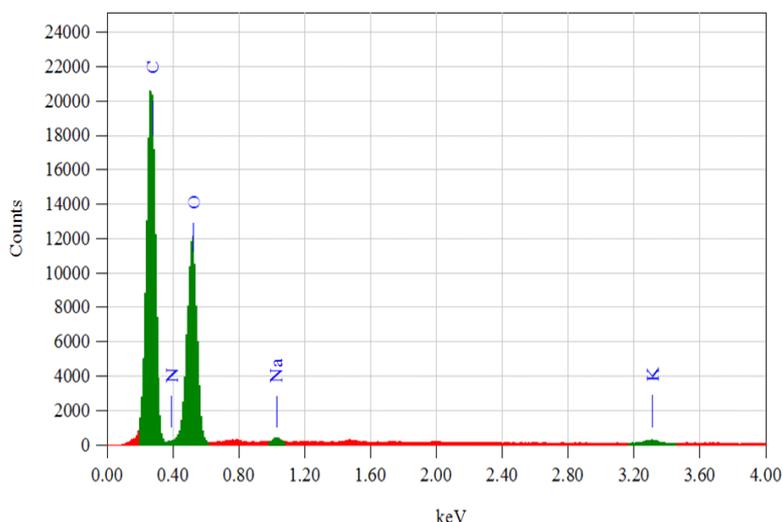


Fig. 9. Spectrometric profile of the component composition of konjac gum.

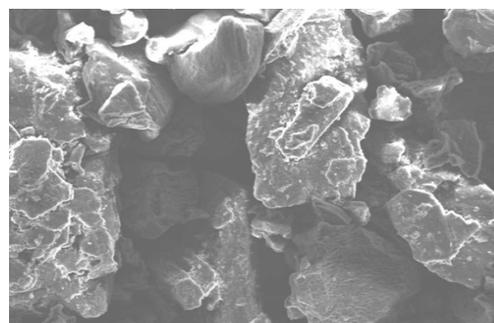
Table 4. Component composition of konjac gum

Element	Relative content, wt %
Carbon	29.79 ± 0.89
Nitrogen	22.29 ± 0.67
Oxygen	47.73 ± 1.43
Potassium	0.17 ± 0.005
Sodium	0.02 ± 0.001

The microvoid content in konjac gum was determined using a micrograph recorded at a magnification of 200 \times (Fig. 10). The microvoid content was $33.87 \pm 1.1\%$. In this case, it was necessary to enhance the image contrast in order to filter the background particles.

Thus, the elements of konjac gum have the form of irregularly shaped granules with a size of 10–300 μm . The component composition of konjac gum comprises carbon, nitrogen, oxygen, potassium, and sodium.

According to the results of mathematical processing, the microvoid content in konjac gum is $33.87 \pm 1.1\%$.



(a)

Fig. 10. *Begin.* Results of determination of the microvoid content in konjac gum: (a) a micrograph at a magnification of 200 \times .

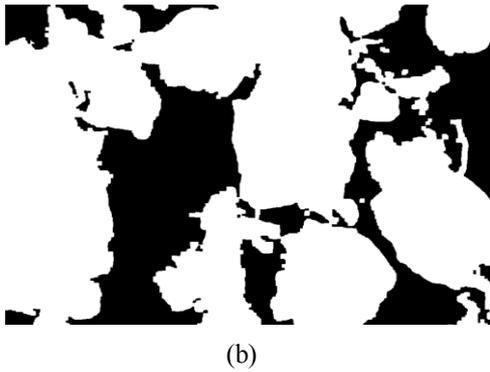
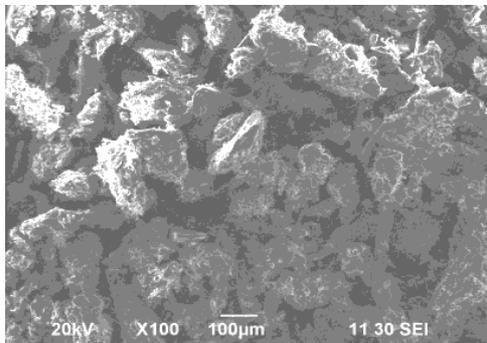


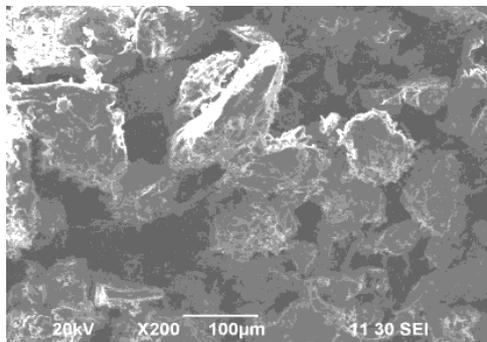
Fig. 10. *End.* Results of determination of the microvoid content in konjac gum: (b) a mask of the micrograph shown in panel (a).

Pectin ARA 105

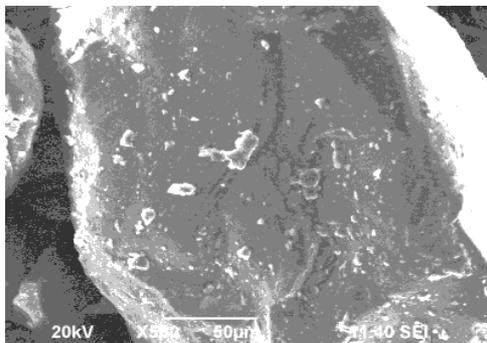
Figure 11 shows micrographs of pectin ARA 105 at a magnification of 100, 200, and 500×.



(a)



(b)



(c)

Fig. 11. Microstructure of pectin ARA 105 at a magnification of (a) 100, (b) 200, and (c) 500×.

The micrographs suggest that the structure of elements of pectin ARA 105 is composed of irregularly shaped dispersed particles with a size of 20–250 µm; in some regions, large granules with a size higher than 300 µm are observed (Fig. 11a). Crystalline clusters are present on the surface of the granules (Fig. 11c). The cluster size can vary from a few micrometers to 30–40 µm (Fig. 11b). The bulk density of pectin ARA 105 is 580 g/dm³.

The main structural element of the macromolecules of pectin as a polysaccharide of plant tissues is galactopyranosyl uronic acid, which comprises L-D-galacturon. Pectin also contains neutral carbohydrates, such as galactose and arabinose. The pattern of distribution of ester groups in the pectin macromolecule has an effect on some physicochemical properties, such as gelling ability, solubility, and surface activity. The thermodynamic flexibility of pectin macromolecules is significantly affected by the degree of esterification. The water solubility of pectin can be improved by decreasing the molecular weight and increasing the degree of esterification (Fig. 12).

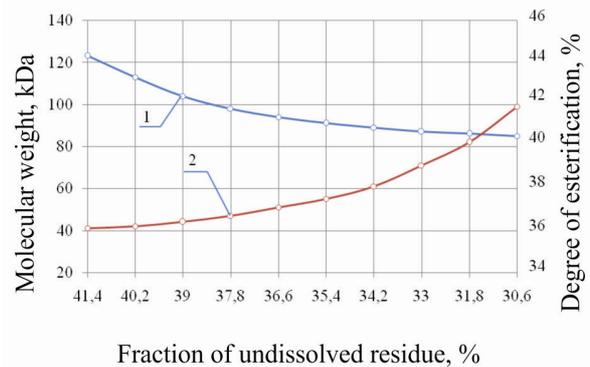


Fig. 12. Effect of (1) the molecular weight and (2) the degree of esterification on the fraction of the undissolved residue of pectin ARA 105 in an aqueous solution in 30 min after the onset of diffusion.

Pectin exhibits the highest stability at a pH of 3–4 (Fig. 13). A deviation of pH to one side from this range leads to a decrease in the charge density of the pectin macromolecules; this phenomenon is most pronounced at temperatures above 50°C.

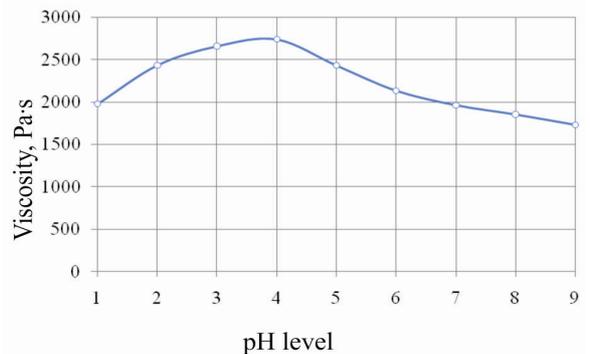


Fig. 13. Dependence of the viscosity of a pectin ARA 105 solution on pH level.

Pectins with a high degree of esterification exhibit gelling properties in an acid medium in the presence of sucrose, whereas pectins with a low degree of esterification are capable of gelling in the presence of salts of polyvalent metals. The widespread use of pectins as structure-stabilizing agents in the confectionery and-

pharmaceutical industries is associated with their physiological inertness and high gelling properties.

Figure 14 shows a spectrometric profile for determining the component composition of pectin ARA 105. The profile exhibits three peaks corresponding to carbon, oxygen, and sodium and a plateau attributed to nitrogen.

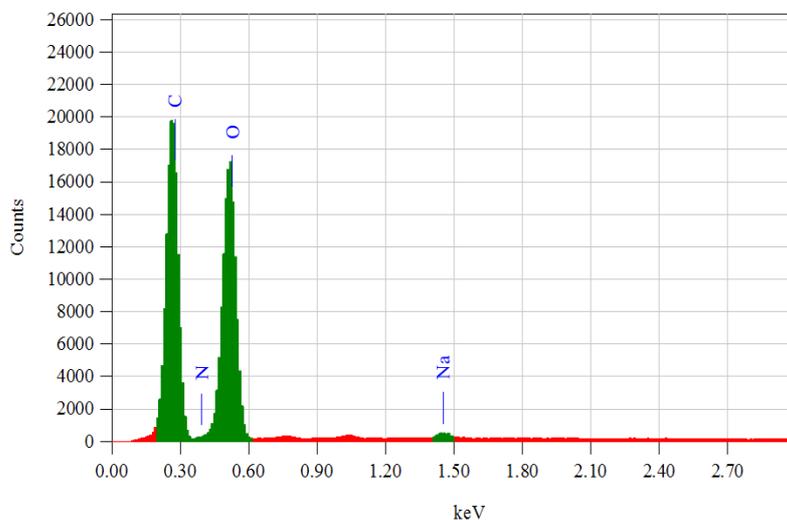


Fig. 14. Spectrometric profile of the component composition of pectin ARA 105.

The component composition of pectin ARA 105 is listed in Table 5. The content of carbon and nitrogen is at almost the same level as in konjac gum. The composition of pectin ARA 105, in common with CMC, comprises sodium and chlorine; the amount of these elements is 3.28 and 0.12%, respectively.

nitrogen, and oxygen, pectin ARA 105 contains sodium and chlorine. The content of microvoids and disperse entities on the surface of the elements is 39.60 and 12.69%, respectively.

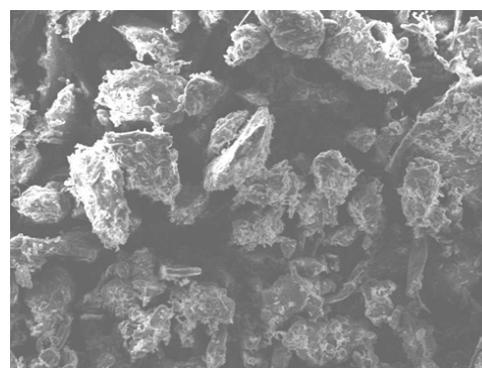
Table 5. Component composition of pectin ARA 105

Element	Relative content, wt %
Carbon	29.04 ± 0.87
Nitrogen	22.70 ± 0.69
Oxygen	44.86 ± 1.34
Sodium	3.28 ± 0.10
Chlorine	0.12 ± 0.004

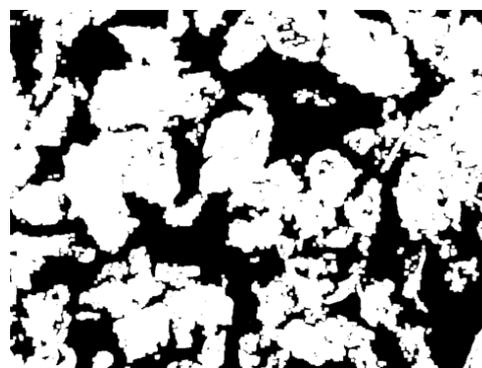
The micrograph of the structure of pectin ARA 105, which is shown in Fig. 15a, was used to determine the microvoid content, while the micrograph shown in Fig. 15c was used to prepare a mask of the disperse entities on the surface of the elements of the structure-stabilizing agent. The respective micrographs and resulting masks are shown in Fig. 15.

The microvoid content in pectin ARA 105 was $39.60 \pm 1.2\%$, while the amount of disperse entities on the surface of the elements of the structure-stabilizing agent was $12.69 \pm 0.7\%$. It should also be noted that, to prepare the mask shown in Fig. 15d, it was necessary to enhance the image contrast and manually correct certain areas of the mask because the required elements in the micrograph had a nonuniform color over the entire surface.

Thus, the microstructure of pectin 105 ARA is characterized by irregularly shaped dispersed particles with a size of 20–250 μm and large granules with a size of higher than 300 μm . Crystalline clusters are present on these elements. In addition to carbon,

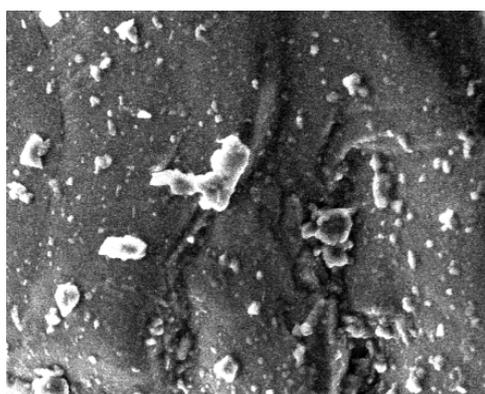


(a)

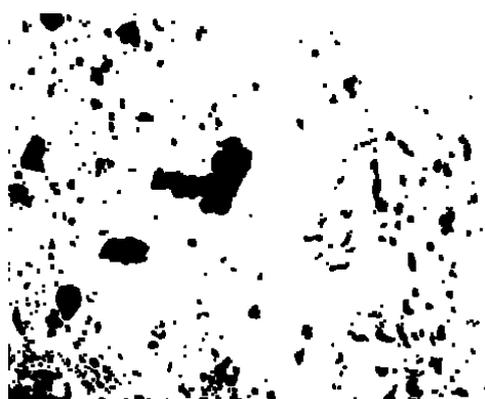


(b)

Fig. 15. *Begin.* Results of determination of (a, b) the microvoid content and (c, d) disperse entities of pectin ARA 105: (a) a micrograph at a magnification of 100 \times , (b) a mask of the micrograph shown in panel (a).



(c)



(d)

Fig. 15. *End.* Results of determination of (a, b) the microvoid content and (c, d) disperse entities of pectin ARA 105: (c) a fragment of the micrograph, and (d) a mask of the micrograph shown in panel (c).

CMC Akucell 2785

Figure 16 shows micrographs of CMC Akucell 2785 at a magnification of 100, 200, and 500 \times .

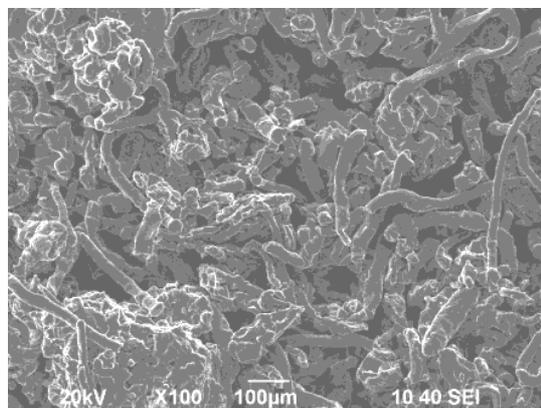
The microstructure of CMC Akucell 2785 is similar to that of structure-stabilizing agents CMC 4500-6000 and CMC 6000-9000. The microstructure of CMC Akucell 2785 is characterized by the closest packing of dispersed elements of all the studied CMC samples; these elements have the form not only of characteristic fibers, but also more rounded irregularly shaped particles with a rough surface (Fig. 16c) and a size of 30–70 μm , while the diameter of the fibers is about 40–60 μm . The structure also comprises large clusters of elements (Fig. 16a). The bulk density of CMC Akucell 2785 is 680 g/dm^3 .

Table 6 lists the component composition of CMC Akucell 2785.

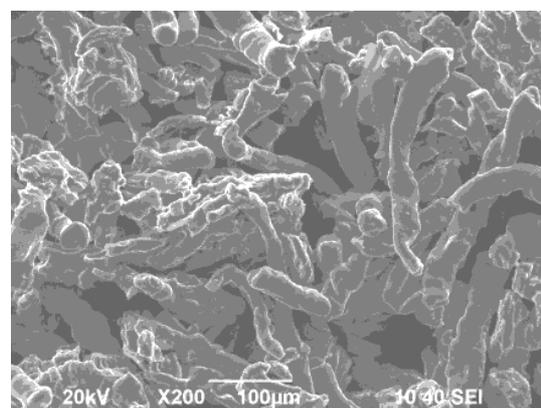
Table 6. Component composition of CMC Akucell 2785

Element	Relative content, wt %
Carbon	30.89 ± 0.93
Nitrogen	22.68 ± 0.68
Oxygen	42.28 ± 1.26
Sodium	4.13 ± 0.12
Chlorine	0.02 ± 0.001

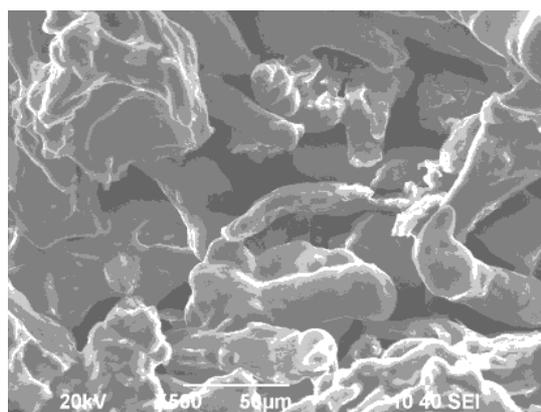
Comparative analysis of the component composition of the CMC Akucell 2785 and CMC Akucell 3265 shows a similarity in chemical composition. Compared to the previous types of CMC, these structure-stabilizing agents are characterized by a higher content of carbon and nitrogen and a lower content of oxygen, sodium, and chlorine.



(a)



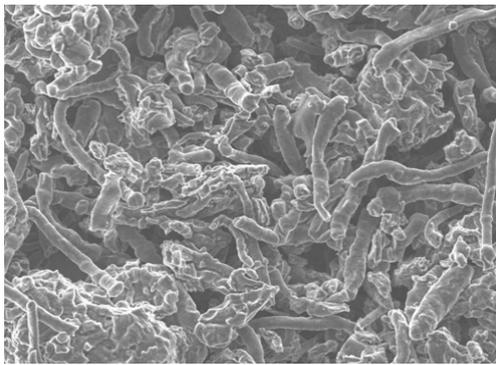
(b)



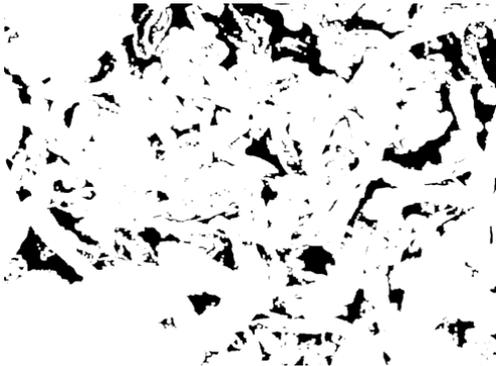
(c)

Fig. 16. Microstructure of CMC Akucell 2785 at a magnification of (a) 100, (b) 200, and (c) 500 \times .

Figure 17 shows photographs of microvoids in CMC Akucell 2785. The mask was prepared using color marking, reducing the boundaries of the marked areas by 1 pixel, and manually editing individual elements. The microvoid content was $11.43 \pm 0.5\%$.



(a)



(b)

Fig. 17. Results of determination of the microvoid content in CMC Akucell 2785: (a) a micrograph at a magnification of 100× and (b) a mask of the micrograph shown in panel (a).

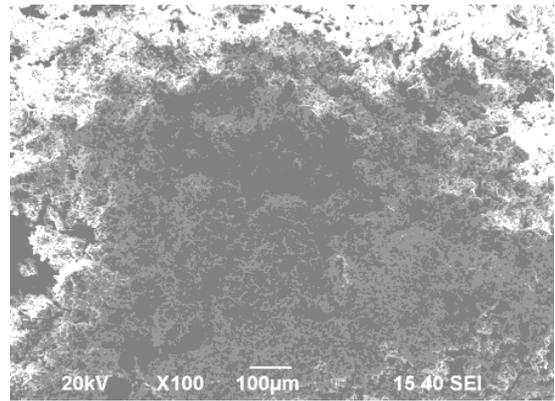
To summarize the data, we can conclude that the structure of CMC Akucell 2785 comprises elements in the form of fibers with a rough surface and a diameter of about 40–60 μm and rounded particles with a size of 30–70 μm . The component composition includes carbon, nitrogen, oxygen, sodium, and chlorine. The microvoid content in CMC Akucell 2785 is $11.43 \pm 0.5\%$.

Xanthan Gum

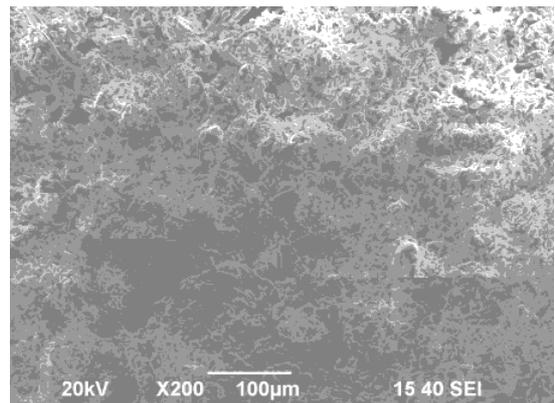
Micrographs of xanthan gum at a magnification of 100, 200, and 500× are shown in Fig. 18.

Xanthan gum is characterized by the highest bulk density of all the studied structure-stabilizing agents: it is 830 g/dm^3 . Xanthan gum has a densely spaced highly dispersed structure. Most of the granules are elongated; their size is 5–40 μm (Fig. 18c). This particle size provides a rapid formation of a highly viscous solution in both hot and cold food systems and contributes to high quality of foods. Some elements of xanthan gum form conglomerates. In addition, the structure contains irregularly shaped microvoids (Fig. 18a).

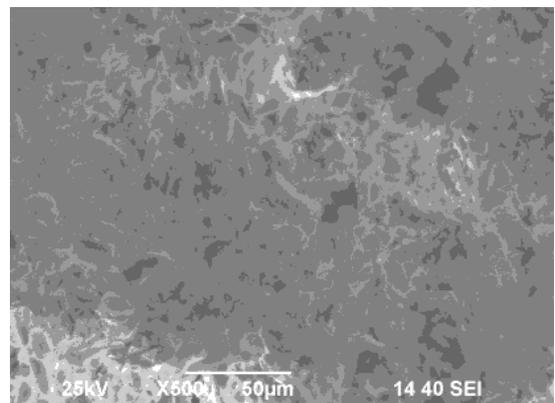
Xanthan gum is produced by the fermentation of sucrose or glucose using the *Xanthomonas campestris* bacteria, which secrete a slimy substance on the outer surface of the cell walls to protect them from viruses and drying up. After fermentation, the culture medium is pasteurized, precipitated with alcohol, and purified by microfiltration to obtain a white powder.



(a)



(b)



(c)

Fig. 18. Microstructure of xanthan gum at a magnification of (a) 100, (b) 200, and (c) 500×.

The stabilizing effect of xanthan gum does not depend on the presence of salts, acids, and heat or mechanical impact. The viscosity of the resulting xanthan gum system does not vary at a pH of 2–12 (Fig. 19).

Xanthan gum is a microbial biopolymer composed of α -D-mannose, β -D-glucose, and α -D-glucuronic acid in an approximate ratio of 3 : 3 : 2. The adsorption of water by xanthan molecules leads to the formation of a three-dimensional network of double helices linked by intermolecular hydrogen bonds. The structure of the resulting substance is similar to that of a gel; however, it exhibits a higher mobility. Under significant mecha-

nical impacts, the networks are destroyed and the double helices elongate in the direction of effort, while the viscosity sharply decreases. After the impact, the three-dimensional network of macromolecules is restored and the viscosity returns to the initial value.

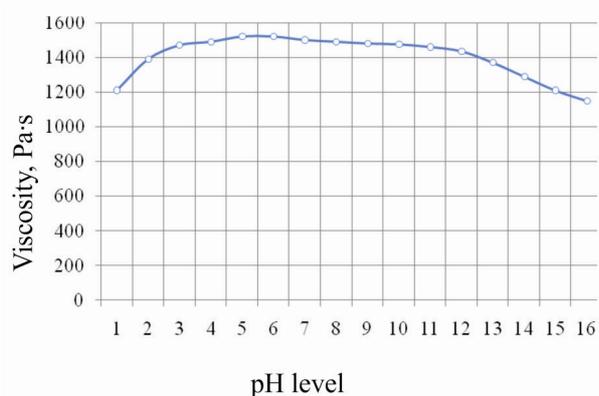
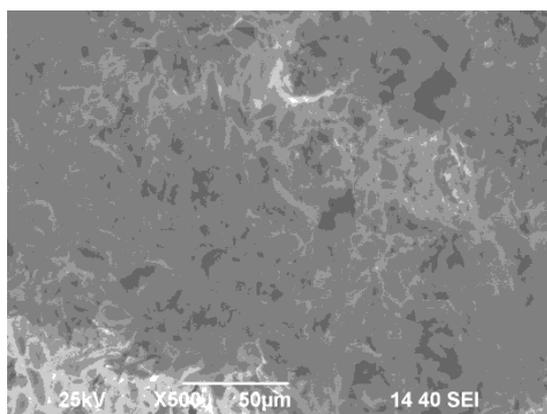


Fig. 19. Dependence of the viscosity of a xanthan gum solution on pH level.

Table 7 lists the component composition of xanthan gum.



(a)

Table 7. Component composition of xanthan gum

Element	Relative content, wt %
Carbon	30.08 ± 0.90
Nitrogen	23.42 ± 0.70
Oxygen	46.38 ± 1.39
Potassium	0.12 ± 0.004

Xanthan gum also comprises carbon, nitrogen, oxygen, and potassium.

The microvoid content in xanthan gum was determined using a micrograph with a magnification of 500× because it provides the highest degree of accuracy. The results are shown in Fig. 20. The microvoid content in the structure of xanthan gum was 6.51 ± 0.3%.

Thus, the microstructure of xanthan gum is composed of closely spaced elongated granules with a size of 5–40 µm. The component composition comprises carbon, nitrogen, oxygen, and potassium; the percentage ratio of these chemical elements is similar to that of konjac gum. According to the results of mathematical processing, the microvoid content in xanthan gum is 6.51 ± 0.3%.



(b)

Fig. 20. Results of determination of the microvoid content in xanthan gum: (a) a micrograph at a magnification of 500× and (b) a mask of the micrograph shown in panel (a).

CONCLUSIONS

The main properties of structure-stabilizing agents with different bulk density have been discussed on the basis of analysis of their micrographs. It has been found that characteristics of the studied structure-stabilizing agents differ in the size, shape, and packing density of the particles. Characteristic particle sizes of each of the structure-stabilizing agents have been shown. The composition of the structure-stabilizing agents has been examined using an electron microscope. Spectrometric profiles of the chemical

composition have been derived; the ratio of the main components in the structure-stabilizing agents has been determined. Some similar features with respect to the presence of particular chemical elements and their ratio in the composition of structure-stabilizing agents of one type (CMC, gum, and pectin) have been revealed. The mathematical processing of micrographs of the structure-stabilizing agents has been conducted; masks for determining the microvoid content in the particle structure of the studied structure-stabilizing agents have been prepared.

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