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INNOVATIVE ENCAPSULATION TECHNOLOGY OF FOOD SYSTEMS USING A BY-PRODUCT OF DAIRY PRODUCTION

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Abstract: Currently, promising innovative direction in the food production technology is the use of techniques of "molecular gastronomy", allowing to modify the consumer properties of traditional foods. One of the techniques of this direction is the technology of "spherification" – the process of encapsulation of various food masses (sauces, juices, extracts, etc.). This technology will allow not only to use it as a tool of innovative management in the restaurant business, but also to expand the field of application of encapsulation in connection with the use of structure-forming agents of domestic production and by-products of the dairy industry. The purpose of the study was to investigate the influence of various factors on the process of encapsulation of food masses: generation of alginate shell when using curd whey as a capsule-forming medium. Methods of instrumental analysis are used in the work. Determination of the qualitative composition and identification of the test ingredients for encapsulation was carried out by IR-Fourier spectrometry. Capsules were prepared by axially feeding into the curd whey of sodium alginate solutions through a device for producing encapsulated products with a fixed size of the nozzle outlets. The results of determining the qualitative composition of the binary system "sodium alginate – curd whey" presumably indicate the complexation of polymers present in the system. It was found that in order to obtain a capsule shape close to a roundly regular one, the concentration of sodium alginate in the encapsulated solution should be between 0.8% and 1.2%. With an increase in the outlet diameter, and also with an increase in the concentration of the structure-forming agent in the encapsulated solution, an increase in the diameter of capsules was observed. The diameter of capsules obtained under the given conditions ranged from 3.7 to 5.7 mm. Generalization of the experiment results served as the basis for developing the technology of encapsulated food products based on the "spherification" method by diffusion of the cross-linking ion (Ca^{2+}) from an external reservoir (curd whey) into a capsular liquid containing sodium alginate. Process conditions for the production of encapsulated food products with specified consumer properties have been established.

Keywords: Capsulation, sodium alginate, milk whey, calcium alginate

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INTRODUCTION

One of the innovative directions in the technology of catering production is the use of "molecular gastronomy" techniques, based on scientific knowledge of the properties of food products and the possibility of modifying their consumer properties [1].

With the advent of "molecular gastronomy," the technological properties of little-known hydrocolloids became more applicable for catering producers, which influenced the expansion of their use in the development of innovative food products [2, 3]. One of the methods of this direction is encapsulation of various food masses (sauces, juices, extracts, etc.).

In the food industry, the use of encapsulation technology is due to a variety of reasons. This method is an effective tool for delivering biologically active molecules (antioxidants, minerals, vitamins, phytosterols, polyunsaturated fatty acids, etc.) and living cells (probiotics) into food [4–6].

Encapsulation is the process of incorporating one material into a shell made of another material to produce particles ranging in size from several nanometers to several millimeters, in other words, immobilizing solid, liquid or gaseous substances into capsules that release the contents at a controlled rate for a predetermined period of time under certain environment conditions. The substance that is encapsulated is called the active substance, or the main product, or internal phase. The material into which the main product is enclosed is called a shell, a membrane, a wall, an outer phase, or a matrix [7–11].

The following substances are used as membrane material for encapsulation: Hydrogels (agar-agar, k-carrageenan, alginates, chitosan, cellulose, etc.), proteins (collagen, gelatin, chicken egg whites, fibrin, etc.), synthetic polymers (polyacrylamide, polyvinyl alcohol, polyethylene glycol methacrylate, polyisocyanates, polyurethane, etc.), etc. Based on the desired characteristics of the finished capsules, it is possible to select a particular substance for encapsulation [12–14].

Alginates are one of the most preferred ingredients for use in encapsulation technology. They are widely distributed in nature as a structural component of brown algae [15]. The safety of alginic acid and its ammonium, calcium, potassium and sodium salts was established by the Joint Expert Committee on Food Additives (JECFA) in 1992 [16].

Alginates are a family of unbranched binary copolymers formed by the residues of β-D-mannuronic acid (M) and α-L-guluronic acid (D), connected by a (1→4)-bond, which vary greatly in composition and sequence. The composition and structure of the sequence may vary depending on the season and growth conditions, as well as the part of the plant from which the structure-forming agent was obtained. The distribution of monomer residues of these acids along the polymer chain has a block character and forms three types of blocks:

– Homopolymer blocks of monotonic sequences of β-D-mannuronic acid residues (M-blocks);
– Homopolymer blocks of monotonic sequences of α-L-hyaluronic acid residues (H-blocks);
– Heteropolymer blocks with alternation of residues of both acids (M-H-blocks).

Such a structure of polymer molecules leads to formation of crystallinity zones in H-blocks, amorphous regions (flexibility zones) in M-blocks and areas with intermediate rigidity in heteropolymer M-H-blocks [17, 18].

Alginic acid salts are classified as high molecular weight water-soluble compounds used in the food industry as a structure-forming agent [19].

Alginates are thermostable compounds. The viscous properties of alginate remain in a wide range of temperatures. The viscosity of alginate solutions decreases by approximately 12% with temperature increase by every 5.6°C. These changes are reversible, solutions acquire initial viscosity upon cooling again. However, as a result of high thermal effects (above 95°C), destruction of the alginate molecule and the weakening of the van der Waals cohesion forces in the dispersion medium occur, which leads to a complete loss of aggregative gel stability, combination of colloidal particles in large aggregates, and formation of a dense precipitate, coagulate [20, 21].

Alginates have an important ability to bind ions, which is the basis of their gel-forming properties. The affinity of alginates for alkaline earth metals increases in the following order: $Mg^{2+} < Ca^{2+} < Sr^{2+} < Ba^{2+}$. This unique property of alginates distinguishes them from other polyanions. The only polyanion resembling alginates from this point of view is pectic/pectinic acid, whose affinity follows the scheme Mg < Ca, Sr < Ba [22]. In the absence of a bivalent cation (Ca^{2+}, Mg^{2+}), alginates only increase the viscosity of the solutions in proportion to the concentration of the structure-forming agent. However, when a multivalent cation is added, especially calcium and at a low pH (≤ 4), a gel is formed. The molecules in this case will be crosslinked by polyvalent ions (the "egg box" model). As the acidity of the solution decreases, the charge of dispersed particles decreases, and the forces of attraction begin to predominate over the repulsive forces. The residues of guluronic acid form a curved conformation, which ensures effective binding of cations (Fig. 1) [23, 24].

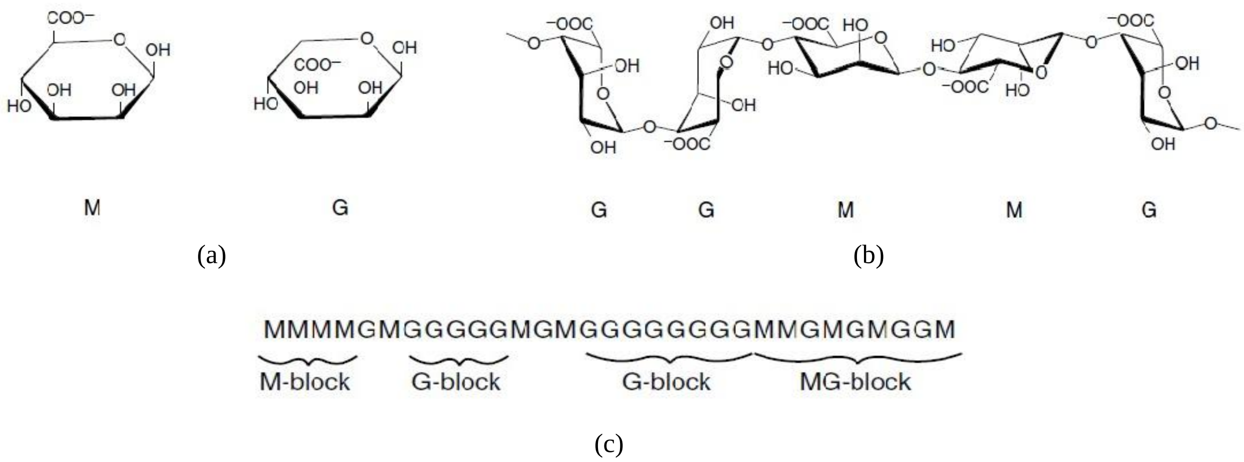

Fig. 1. Structural characteristics of alginates: (a) M: β-D-mannuronate, G: α-L-guluronate; (b) chain conformations; (c) block distribution.

In solving problems associated with gelation of sodium alginate, the determining property is the ability to regulate the introduction of cross-linking ions. This control can be carried out using two basic methods of preparing an alginate gel: method of diffusion and method of internal gel formation. The first method is characterized by diffusion of a cross-linking ion from the external reservoir into the alginate solution. Internal gel formation (sometimes called gelling in the block (*in situ*) differs from the previous method in that Ca^{2+} ions release from an inert calcium source in an alginate solution in a controllable way. Controlled release is usually achieved by changing pH level and/or by a limited source solubility of the calcium salt [25].

Ferran Adrià, chef at the El Bulli restaurant, became the pioneer of encapsulation technology in public catering, using the ability of alginate solutions to form gels by adding Ca^{2+} ions to them. In gastronomic circles, this method was termed "spherification" [26, 27]. The spherification technology consists in applying the diffusion method. The essence of the method consists in the interaction of sodium alginate with a bivalent cation, for example, a calcium ion or other polyvalent metal (zinc, aluminum, copper), to form a gel. Diffusion formation of the gel structure is characterized by a high gel formation rate, and this high-speed curing is used as a method of immobilizing and restructuring food products. Each drop of the alginate solution forms one granule of gel with the inclusion of the active agent. The spatial grid of the gel is formed by the interaction of calcium ions with carboxyl groups and additionally stabilized by coordination links between calcium ions and hydroxyl groups, hyaluronic acid residues (Fig. 2) [22].

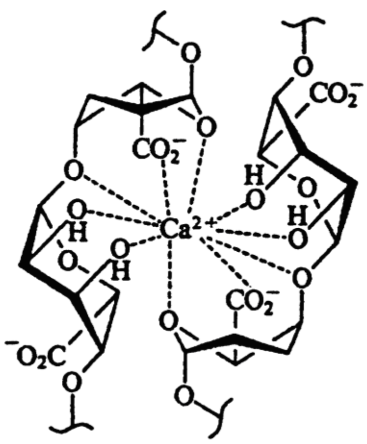

Fig. 2. Formation of a polysaccharide intermolecular complex with calcium ions.

A feature of the diffusion gel formation process is that the resulting gel is characterized by an inhomogeneous distribution of alginate with its high concentration on the surface, which gradually decreases towards the center of the gel. This phenomenon is explained by the fact that during the diffusion process a gelation zone is formed, which moves from the surface to the center of the gel. The alginate activity in this zone is zero, and the alginate molecule diffuses from the internal, unstructured part of the system, passing into the gel, into the zone of zero activity [28, 29].

This process of formation of alginate gels is used to produce spheres of small size with a dense shell and a liquid center. The product obtained in this way has the texture and appearance of natural fish caviar, and at the same time it has a different flavor and taste [30].

For the implementation of the spherification technology, a solution is prepared based on sodium alginate and liquid filler with projected taste. Introduction of the encapsulated mixture is carried out by axial dropping into a prepared solution containing calcium ions, and an instantaneous formation of a spherical shell around the encapsulated substance takes place. One of the striking examples of the use of this technique in the restaurant El Bulli is the production of spherical green "olives", which are served on a special spoon [30].

In the original procedure for the implementation of the spherification method, the concentration of alginate in the encapsulated solution is from 0.6 to 4%, the content of calcium chloride in the forming solution varies from 0.05 to 1.5 mole. Thus, it is possible to obtain spherical capsules with a diameter of 0.2 to 5 mm depending on the instrumentation used (pipette, syringe, oscillating nozzle, spray nozzle, coaxial air flow, electric field, etc.) and viscoelastic characteristics of the encapsulated solution of sodium alginate [5].

In studying this encapsulation technology, scientists have found that the rate of gel formation of sodium alginate solutions is the highest when using calcium chloride as a calcium-containing component. Compositions based on gluconate or calcium lactate have the least gel-forming ability. Alginate capsules formed by keeping them in a solution of calcium chloride acquire the maximum strength characteristics after 100 seconds of droplet retention in the forming solution, while in order to achieve such characteristics, capsules in a calcium lactate solution require a holding time of 500 seconds, calcium gluconate – of 3000 seconds.

Based on the application of the Tate law, a model was developed to predict the shape and size of alginate granules in the range from 2.9 to 3.25 mm produced by the drop method [31].

An alternative to "suppliers" of calcium ions (calcium chloride, lactate and gluconate) is a milk whey, which is a by-product of cheese production, or casein, and is not widely used for commercial purposes, and often simply recyclable.

Milk whey is close to natural cow milk in its composition [32] and is rich in physiologically valuable whey proteins, water-soluble vitamins and lactose. There are two types of milk whey: curd (sour) and cheese (sweet).

The choice of curd whey as a calcium-containing component with the application of encapsulation technology by the method of spherification is due to the high content of calcium in it as compared with the cheese whey [33]. Inclusion of curd whey in the composition of the shell increases the nutritional value of a capsule, and its application in the production of encapsulated food products seems to be a promising direction.

The purpose of the study was to review the process of the formation of sodium alginate when using by-products of dairy production as a calcium-containing component for encapsulation of food masses.

OBJECTS AND METHODS OF STUDY

Sodium alginate (TU 15-544-83 "Sodium alginate food") and curd whey (GOST R 53438-2009 "Dairy whey. Specifications") were used as the objects of the study.

For the preparation of binary systems "sodium alginate – curd whey", a sample of sodium alginate was injected into the curd whey solution, dispersed at a temperature of 20 to 22°C for 60 minutes with a magnetic stirrer. The resulting solution was dried in a Vac Modul vacuum module, with a vacuum pump VP1, for VD 53 to a moisture mass fraction in the sample of 15%.

To prepare model solutions to be encapsulated, a sample of sodium alginate was dispersed in a mixture of curd whey and water taken at a ratio of 50 : 50 at a temperature of 20 to 22°C for 3–4 minutes on a magnetic stirrer. The resulting solution was placed in a refrigerated cabinet at a temperature of from 2 to 6°C for degassing the solution for 3 hours.

Capsules were produced by axial feeding into the curd whey of sodium alginate solutions through the device for producing encapsulated products [Patent RF 156197] with a fixed size of nozzle outlets. Injection of drops of the extrusion mixture in the form of a sodium alginate solution into the forming medium – curd whey – was carried out at a ratio of 1 : 10. Spheres were obtained differing in their structural and mechanical characteristics and dimensional parameters, due to the diffuse structure formation occurring over time. To maintain a constant amount of Ca^{2+} ions in the whey, its solution was constantly freshened. The finished capsule cores were washed with distilled water and dried on filter paper. The experimental setup layout is shown in Fig. 3.

Determination of the qualitative composition and identification of dry powders taken as the objects of the study was carried out by IR-Fourier spectrometry with an Agilent Cary 660 spectrometer.

Spectra were interpreted using literature data [34].

To determine the dimensional characteristics of the resulting capsules, a XSP 10-640x microscope with a MOV-1-16x screw micro-eyepiece with a measurement scale with measurement limits from 0 mm to 8 mm was used.

The capsule shape factor was determined by the formula [35]:

$$K_f = \pi \cdot \left(\frac{a}{b} + \frac{b}{a} \right), \qquad (1)$$

where *a* and *b* are semiaxes of the ellipse, mm.

The minimum possible value K_f has the circle ($K_f = 2\pi$).

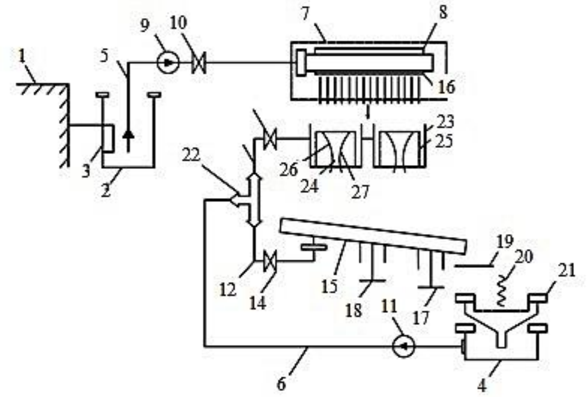

Fig. 3. Diagram of the device for producing encapsulated products: 1 – housing; 2 – vessel for the capsule filler substance; 3 – viscometer; 4 – storage tank for circulating solution; 5,6 – pipeline; 7 – encapsulation unit; 8 – capsulating head; 9 – pump; 10 – valve; 11 – pump; 12, 13 – pipeline; 14 – valve; 15 – conveying trough; 16 – nozzles; 17 – screw; 18 – adjusting screw; 19 – cone-shaped chute; 20 – pipeline; 21 – mesh tray; 22 – valve; 23 – intake vessels; 24 – gap; 25 – forming shape-generating vessels; 26, 27 – parabolic shape planes.

RESULTS AND DISCUSSION

The analysis of the patent information literature and comparative characteristic of the physico-chemical properties of sodium alginate and curd whey composition allowed to formulate the main scientific approaches to the development of encapsulation technology for food systems.

For the scientific substantiation of the use of alginates as a membrane-building material, it is necessary to establish the effect of certain process factors on the characteristics of the gels formed. The most important factors are:

– Concentration of alginate;

– Interaction of calcium ions of curd whey with carboxyl groups of sodium alginate and the ratio between gel-forming and gel-non-forming ions;

– Presence of complexing agents (phosphates, citrate, EDTA, GDL, acids).

In determining the qualitative composition and identifying the ingredients used in the encapsulation technology, the IR spectra of the study objects were obtained: curd whey, sodium alginate and binary system "sodium alginate – curd whey".

The presence of a broad absorption band in the 3600-3100 cm^{-1} region was established in the IR spectra of the curd whey sample, which indicates the presence of valence vibrations of hydroxyl groups (OH) and amino groups (NH) in the molecule (Fig. 4). The presence of absorption bands of 1650 cm^{-1} and 1550 cm^{-1} is due to valence vibrations of the C=O bond and to plane deformation vibrations of the NH bond. Thus, it is confirmed that a protein group is present in the analyte.

The IR spectrum of a sodium alginate sample coincides with the spectrum of a known ingredient recorded in the instrument library (Fig. 5). In the test sample in the 3500–3000 cm^{-1} region, absorption bands due to stretching vibrations of hydroxyl group are observed, in the region of 2800–3000 cm^{-1}, there are bands of valence vibrations of CH groups, in the region of 1000–1100 cm^{-1}, there are vibration bands of pyranose cycles, absorption bands at 1650–1550 cm^{-1} are typical for the ionized carboxyl groups of which hydrogen ions are replaced by sodium, which in turn when adding whey will be presumably substituted for the calcium ion, due to the greater reactivity of the latter.

To confirm this assumption on the interaction of study objects, a spectrum of the binary system "sodium alginate – curd whey" was obtained. The presence of absorption bands in the 1950–2150 cm^{-1} region indicates the presence of $C{=}N^{+}{=}N$ valence bonds (Fig. 6), and the valence vibrations of hydroxyl groups (OH) and amino groups (NH) in the composition under study (Table 1). This fact presumably indicates the complexation of polymers present in the system.

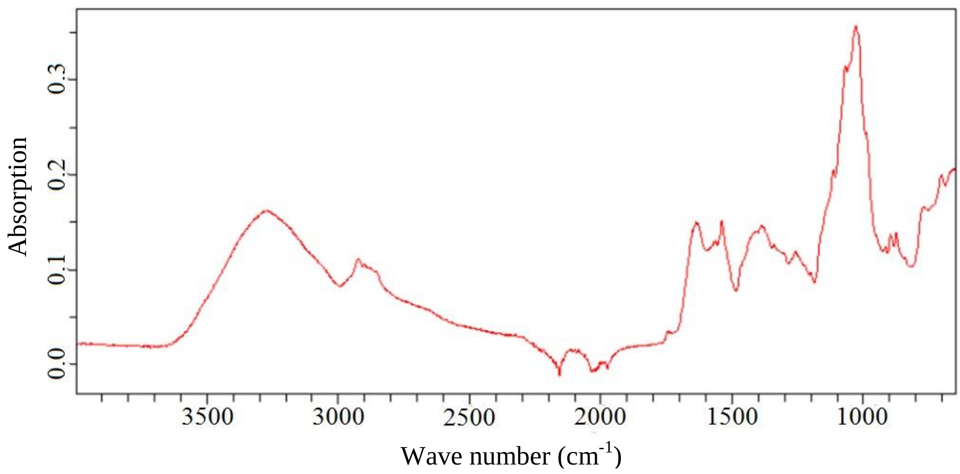

Fig. 4. IR spectra of a curd whey sample.

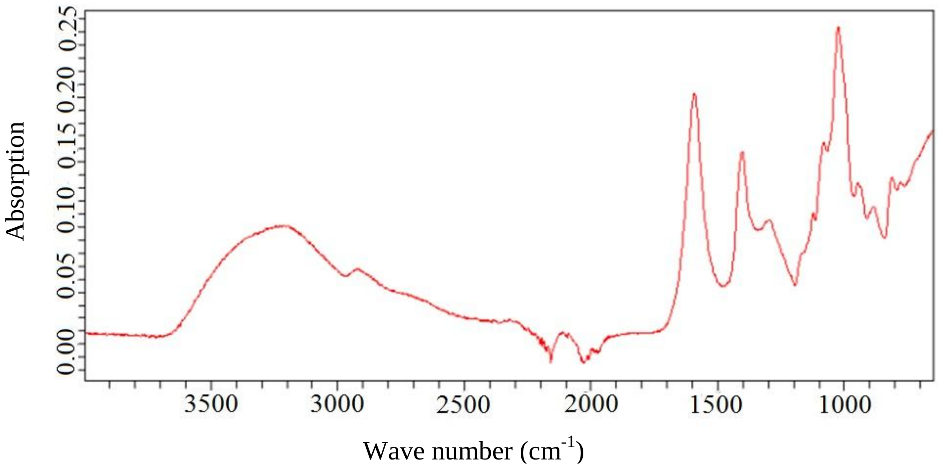

Fig. 5. IR spectra of a sodium alginate sample.

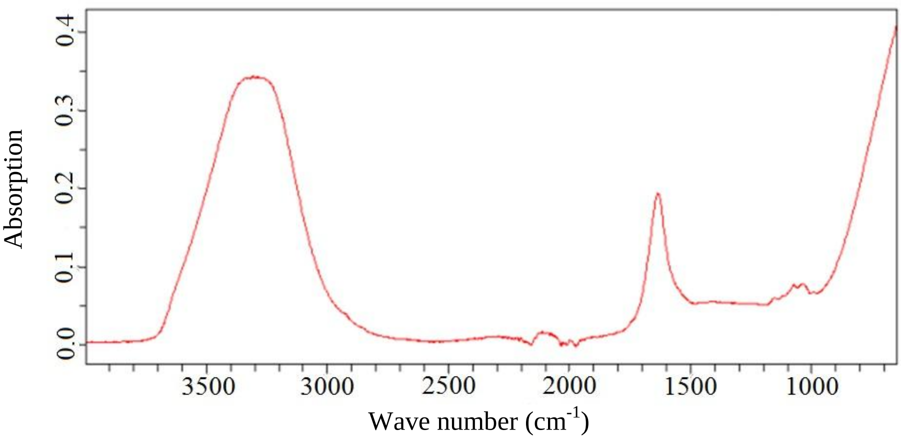

Fig. 6. IR spectra of a binary system sample based on decalcified curd whey and sodium alginate.

Table 1. Assignment of absorption bands in IR spectra of curd whey and sodium alginate before and after interaction

Wave number, cm^{-1}			Bands assignment
sodium alginate	curd whey	"sodium alginate-curd whey"	
1000–1100	–	1000–1100	Vibration bands of pyranose cycles
1260	–	–	Flat deformation vibrations of OH groups in structures
1380	–	–	Scissor vibrations of OH groups, CH_2
1550–1650	1600–1650	1550–1700	Presence of valence vibrations of the C=O bond and plane deformation vibrations of the NH bond
–	–	1950–2050	Presence of $C{=}N^{+}{=}N$ valence bonds
2800–3000	2800–3050	–	Bands of valence vibrations of CH groups
3000–3500	3050–3600	3000–3600	Presence of valence vibrations of hydroxyl groups (OH) and amino groups (NH) in the molecule

To predict the shape and size of the alginate capsules, the influence of various process parameters was studied through a two-factor experiment, planned with the help of Box-Hunter's rotational second-order plans.

As considered factors, presumably having an effect on the diameter of the capsules, the following were accepted: mass fraction of sodium alginate in the encapsulated solution in the range from 0.6 to 1.4% (factor X_1), the outlet diameter is from 1 mm to 5 mm (factor X_2) (Table 2).

The results obtained during the experiment and data processing is presented in Table 3.

As a result of the regression-correlation analysis, an array of data was obtained reflecting the general regression equation showing the relationship between the diameter of the finished capsules and encapsulation parameters under consideration: outlet diameter, mass fraction of sodium alginate

$$Y = 0.225+5.938 \cdot X_1+0{,}313 \cdot X_2 - \\ -3.281 \cdot X_1^2-0.081 \cdot X_2^2+0.625 \cdot X_1 \cdot X_2, \qquad (2)$$

The obtained results allow to draw a conclusion that, according to the degree of influence on the diameter of the finished capsules, the factors considered can be arranged in descending order: concentration of sodium alginate injected–outlet diameter (Fig. 7).

Table 2. Values of factors in natural and non-dimensional scales

Name of factors	Possible values				
	–1.414	–1	0	+1	+1.414
X_1 – amount of sodium alginate injected, %	0.6	0.7	1.0	1.3	1.4
X_2 – outlet diameter, mm	1.0	1.6	3.0	4.4	5.0

Table 3. Experiment results

Experiment No.	Varied process parameters		Y value is the capsule diameter, mm
	X_1 – amount of sodium alginate injected, %	X_2 – outlet diameter, mm	
1	0.8	1.6	4.2
2	1.2	1.6	4.5
3	0.8	4.4	4.6
4	1.2	4.4	5.4
5	1.4	3.0	4.9
6	0.6	3.0	4.0
7	1.0	5.0	5.7
8	1.0	1.0	3.7
9	1.0	3.0	5.0
10	1.0	3.0	5.0
11	1.0	3.0	5.0
12	1.0	3.0	5.0

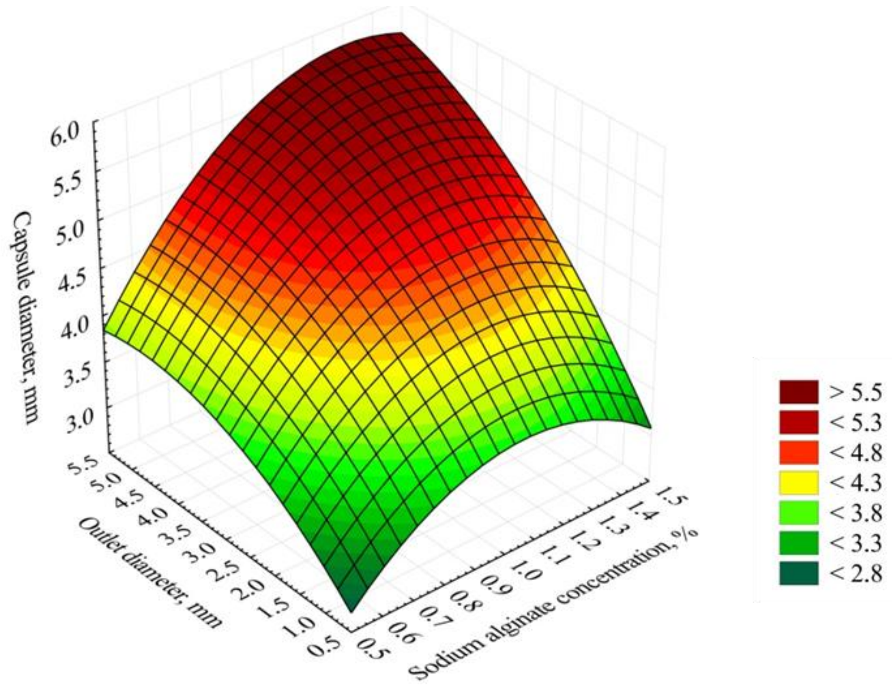

Fig. 7. Dependence of the capsule diameter on process parameters.

Preparation of the desired capsule shape (sphere) with the optimum ratio of the shell thickness to the diameter of the contents is mainly dependent on the viscosity of the encapsulated solution. It is determined that in order to obtain a capsule shape approximating to a roundly regular one, the necessary concentration of sodium alginate in the encapsulated solution should be from 0.8 to 1.2%. As the concentration of the structure-forming agent and, consequently, viscosity of the encapsulated system increases, the shape of the capsules differs from the spherical and approaches it to the elliptical. Reducing concentration of structure-forming agent below the optimal value leads to an increase in the residence time of the capsules in the forming solution, as well as to deformation during their extraction from the curd whey solution.

An increase in the diameter of the capsules is observed with an increase in the concentration of the structure-forming agent in the encapsulated solution and with an increase in the outlet diameter. The diameter of the resulting capsules under given conditions varies from 3.7 to 5.7 mm. At a low concentration of sodium alginate, the axial feed of the solution to the curd whey is not easily implemented, which leads to impossibility of controlling consumption of the filler material of the capsules. This pattern is also observed with an increase in the outlet diameter.

Generalization of the experiment results served as the basis for developing the technology of encapsulated food products by the method of "spherification" based on the gelling of sodium alginate with the calcium-containing component – curd whey.

The encapsulation technology involves extrusion of a capsule mixture containing 1% sodium alginate by axial feeding into a forming solution (curd whey) through a 3-mm diameter outlet (Table 4). In this way, spherical capsules of round-regular shape with a diameter of 5 mm and high organoleptic parameters are obtained.

The proposed technology is tested in production conditions, a sweet dish is prepared: a lemon starch drink, containing a capsular side dish based on raspberry juice and tarragon extract.

Implementation of the developed technology in the food industry will expand the range of innovative culinary products that will increase the competitiveness of industry enterprises.

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Table 4. Process parameters of production of capsular side dish by the "spherification" method

Process stage and process mode	Process mode value
1 Preparation of the liquid to be spherified	
1.1 Neutralization of curd whey (to pH ≥ 4.2) amount of acidity regulator, %	1.5
1.2 Dissolution of sodium alginate	
concentration, %	1
volume of decalcified curd whey, %	67.5
volume of encapsulated ingredient, %	30
structure-forming agent expanding, min	40
1.3 Mixing of prepared components	
mixing type	manual/auto
time, sec	30
2. Solution degassing	
cooling, °C	4 ± 2
time, min	30
3. Preparation of the forming solution	
curd whey thermostating	
time, min	30
temperature, °C	20–22
4. Encapsulation	
ratio of the encapsulated mixture to the forming solution, g/g	1 : 10
dynamic viscosity of the encapsulated solution, cP	280–1000
outlet diameter, mm	3
capsule diameter, mm	5
capsule residence time in the forming solution, sec	120
5. Spheres washing	
capsule-water ratio, g/g	1 : 4
time, sec	30
6. Capsule drying	until moisture removal from the surface

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