Представлено аналітичне дослідження моделі процесу капсулоутворення системи «харчові ліпіди – альгінат кальцію», починаючи з оцінки технічних можливостей гідравлічної екструзійної головки для капсулювання до етапу формування термодинамічно стабільної ліпідної капсули. Одержані рівняння та моделі являються аналітичним описом капсулоутворення та обґрунтовують тривалість утворення капсули, концентрації реагуючих компонентів та параметри технологічного циклу

Ключові слова: процес капсулоутворення, альгінат натрію, альгінат кальцію, екструзійна головка, поверхневий масообмін

Представлено аналитическое исследование модели процесса капсулообразования системы «пищевые липиды – альгинат кальция», начиная с оценки технических возможностей экструзионной головки для капсулирования до этапа формирования термодинамически стабильной липидной капсулы. Полученные уравнения и модели являются аналитическим описанием капсулообразования и обосновывают продолжительность образования капсулы, концентрации реагирующих компонентов, параметры технологического цикла

Ключевые слова: капсулообразование, альгинат натрия, альгинат кальция, экструзионная головка, поверхностный массообмен

ANALYTICAL STUDY OF THE MODEL OF CAPSULE FORMATION OF THE SYSTEM "FOOD LIPIDS — CALCIUM ALGINATE"

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1. Introduction

Under conditions of competition, production of food products is aimed at ensuring their quality and safety, giving the consumer a wide range of products with high nutritional value and at affordable price. Under these circumstances, producers face the task of introducing new competitive technologies, of enhancing nutritional value, extending the range of products, bringing down costs and prolonging shelf life of foods. Modern product range, technological process of production, formulation composition of products is the result of innovative processes of scientific and technical activity aimed at improving technological and technical-economic level of production, providing better quality and meeting consumer demand.

An analysis of consumer market in Ukraine reveals a trend of the increasing demand for new types of products, including those obtained by encapsulation, which possess certain advantages compared with the traditional food products. Obtaining encapsulated products opens up prospects in the area of processing raw materials of plant or animal origin, makes it possible to purposefully influence the composition and structure of products.

Encapsulation and granulation of food products in technological processes are relatively new principles of technological impact on food systems, which is why it is rather difficult to give a clear-cut categorization of the scope of their application. On the one hand, scientific and technical information is insufficient and hard to access, on the other hand, research is being dynamically developed. This is associated with both the discovery of new patterns in encapsulation and technological support for these processes and the growing volume of information on the practice of the application of technologies implemented in production.

Encapsulation is a technological direction, which every year takes a more prominent place in many sectors of food industry, ensuring the achievement of qualitatively new effects. All this makes it a relevant task to scientifically substantiate and develop principles of technical provision and to systematize experimental data on the use of encapsulated lipids in the technology of food products. This would create a new scientific and technological direction of the processing of food raw materials based on the new principles of processing. The result would mean an improvement in the technological and physical-chemical stability of the fat component, as well as creation of products with new consumer properties. The relevance of such a research is ever more important given the possibility of using new data in the theory and practice of special types of nutrition, as well as in the fields related to human health – medicine, pharmaceutical industry, microbiology, etc.

2. Literature review and problem statement

An analysis of the scientific literature testifies to the larger application, during encapsulation of different food systems and medicines, of sodium alginate (AlgNa) [1]. It is used as a gel-like matrix for the inclusion of these components or as a modifying additive in combination with other polymers [2]. From the technological point of view, this is predetermined by the capability of salts of alginic acid, under certain conditions, to form a structure accompanied by the obtaining of spherical shapes [3] or other geometrical shapes [4]. From a biological point of view, it is capable of biodegradation, it is characterized by the absence of toxicity, stability under physiological conditions of the oral cavity and the ability to form edible shells [5]. Alginates that consist of the residues of β-D-mannuronic (M-blocks) and α-guluronic (G-blocks) acids are capable of forming a gel, including films, in the presence of two-valence cations [6], specifically calcium.

A generally accepted method for obtaining alginic granules is the extrusion of AlgNa solution with various fillers in the form of droplets the size of 0.1...3.0 mm to the forming environment – a solution of chloride calcium ($CaCl_2$). This method is known as the diffusion external gel-formation [7].

At the same time, obtaining alginic capsules is achieved through the extrusion of encapsulant, which contains Ca^{2+}, into the medium of AlgNa solution. Known technical solutions are not applicable for the encapsulation of hydrophobic substances, including dietary lipids [8]. Under conditions of employing a well-known principle of encapsulation, the encapsulant is formed by a lipid raw material in which the dissolution of calcium is impossible for objective reasons [9].

Obtaining alginate-calcium shells for the encapsulation of food lipids (in general terms of food products) in a continuous flow is relevant both scientifically and practically. Obtaining the encapsulated products that contain dietary lipids inside refers to complicated problems. They follow from objective physical laws, taking which into consideration underlies the formation of the physical body and the shape of the capsule. At the same time, chemical laws are considered at the stage of formation of the hydrogel shell, which is implemented on the principles of occurrence of a "chemical gel" due to the emergence of a polyelectrolyte complex of ionotropic polysaccharide. These tasks should be solved taking into consideration the requirements to product-technological, functional-technological, microbiological, organoleptic indicators, etc.

3. The aim and objectives of the study

The aim of present experiment is to study analytically a model of encapsulation, and to determine conditions for reaching the thermodynamically equilibrium state by the sphere "food lipids – calcium alginate". The sphere is formed in a two-pipe extruder head designed as "pipe in pipe" in the medium of a transporting functional fluid of Ca^{2+} solution. At the same time, the use of the head designed to change a position of the inner pipe is substantiated. This enables centric encapsulation of the content of the sphere – a capsule with a uniform thickness of walls by a vertical vector.

To achieve the set aim, the following tasks have been solved:
– by applying an analysis of the principle of action of a two-pipe extrusion head, to substantiate a design feasibility of the "up-an-down" shift of the inner extrusion pipe of the encapsulant in the medium of a drop of the shell-former;
– by employing analytical modeling of behavior of the sphere in a flow of the accepting medium of Ca^{2+} solution, to determine a duration of the structure-formation and the transition of a quasi-stable capsule into the thermodynamically equilibrium state with preset technological indicators.

4. Materials, objects, methods of research

The methodology of the research and description of constructive provision of the technological process of encapsulation of the system "food lipids – Alg_2Ca" are given in more detail in [10].

4. 1. Materials and objects of research

The objects of the present research were: a device with a two-pipe extrusion head designed as "pipe in pipe" (Fig. 1, a), a forming medium with Ca^{2+}, gels of calcium alginate (Alg_2Ca), an encapsulated semi-finished product with food lipids inside, encapsulated food products with food lipids inside.

Solutions of the AlgNa shell-former were obtained by dispersing a batch of AlgNa in the prepared drinking water at temperature $t=18...20\,°C$ with further exposure for $\tau=(3...4)\times 60^2$ at $t=2...6\,°C$. We used soluble salt $CaCl_2$ as a source of Ca^{2+}, its estimated amount was dissolved in the prepared drinking water for $\tau=(8...10)\times 60$ s at $t=18...20\,°C$. The resulting solution was filtered. Images of the examined samples are shown in Fig. 1, b.

a

b

Fig. 1. Photographic images of the examined objects: *a* — industrial extruder of the UZhK-100 brand for the encapsulation of food lipids; *b* — examined encapsulated food lipids

### 4. 2. Procedure of the encapsulation of food lipids

The formation of a quasi-stable sphere-capsule by extrusion was carried out using a vertical two-pipe extruder head

of the device UZhK-100 (Ukraine) [10, 11]. Such a design enables uniform laminar flow of the shell-former (external pipe) and food lipids (inner tube). The result is the formation, at the output of the head, in the air medium, of a two-phase drop-sphere, filled with controlled food lipids. Operation of the device is enabled by a spontaneous controlled separation of a quasi-stable capsule from the extruder head and its displacement under the action of gravity force into a transporting medium of Ca²⁺ solution. The technique of the encapsulation of hydrophobic substances was designed and technical solutions for obtaining capsules with the hydrophobic contents were patented [12, 13].

5. Analytical study of the model of formation of a thermodynamic stable capsule with the contents of food lipids

It was technologically predicted that a quasi-stable capsule, formed in the air medium, is separated from the nozzles of the UZhK-100 device head, and, traveling by air, it enters the transporting-accepting medium of Ca²⁺ solution to be subsequently fully immersed.

The transition of a capsule from the quasi-stable state to the thermodynamic stable state takes place in the accepting transporting medium of the device through the realization of chemical potentials. Due to the chemical interaction between AlgNa, which is constituent part of the solution of shells of quasi-stable capsules, and the ions of bivalent metal calcium, which is constituent part of the accepting medium, there forms a film-like phase of the capsule − Alg$_2$Ca by the scheme (1):

$$Na_{(x+y)}Gul_xMan_y + Ca^{2+} \rightarrow CaCul_4 \times$$
$$\times Na_{(y+x+4)}Gul_{x-4}Man_y + 4Na^+. \tag{1}$$
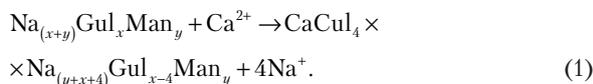
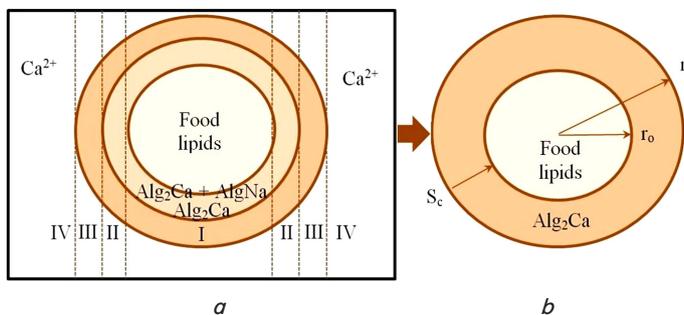Development model of the formation of a thermodynamically stable capsule is shown in Fig. 2.

Fig. 2. Development model of the formation of a thermodynamically stable capsule with the contents of food lipids: a — first (instantaneous) stage (I — food lipids; II — aqueous solution of AlgNa; III — gel Alg$_2$Ca; IV — accepting medium with Ca²⁺), b — formation of capsule with radius r_c and surface S_c

When estimating states of the system in Fig. 2, we can state the emergence of zones and their division. The obligatory stage of the process of formation of the shell which is associated with the regularities of mass transfer of Ca²⁺ from the accepting medium to the thickness of the wall. The period of existence of state (a) will depend on many factors, but time (τ) of the transition to state (b) is the magnitude, which is subject to the parameterization.

An analysis of the transformations according to the hypothesis and development of the system to level (b) occurs in the thickness of the shells of layers in zones II, III at the point of instantaneous contact with Ca²⁺ solution. This is possible only at full immersion of the capsule.

At the initial period, several zones emerge in the wall of the capsule relative to the accepting medium (zone IV) and the encapsulant (zone I).

When examining the boundaries of zones III and IV, it is clear that gel Alg$_2$Ca, which instantaneously forms in the contact between a quasi-stable capsule and the accepting medium, cannot diffuse to zone IV. This can be explained by the fact that this substance is a hydrophobic polymer with high molecular weight (>1,000 kDa), which has a low affinity with water while zone IV is a water solution of Ca²⁺. The low solubility of salt of Alg$_2$Ca does not allow its dissociation into ions under condition of excessive concentration of Ca²⁺ in water, which is why the diffusion of Alg$_2$Ca towards aqueous solutions, specifically zone IV, is impossible.

The concentration of Ca²⁺ in zone IV will always exceed the concentration of Ca²⁺ in zone III, which is the reason for the emergence and existence of Alg$_2$Ca in a stable state.

Also impossible is the diffusion of Alg$_2$Ca inside the sphere towards zones II and I because there will be an aqueous medium inside the sphere in the form of a mixture of salts of Alg$_2$Ca and AlgNa. Under condition $D_{AlgNa} > D_{Alg_2Ca}$, only the diffusion of AlgNa from zones I and II is possible. However, the low affinity of Alg$_2$Ca with water makes this diffusion impossible.

The condition for a full immersion of a quasi-stable capsule into the accepting medium of aqueous solution of CaCl$_2$, to reach state (b), is technologically mandatory, however, as shown by experimental research, is impossible without special technical solutions. Under these conditions, there is a contradiction to basic physical laws since $\rho_c < \rho_{solution\ Ca2+}$ and it cannot be satisfied without additional structural-technological solutions. Such an inequality follows from the physical characteristics of the capsule's component oil, for example, sunflower oil, for which $\rho=920$ kg/m³, which is less than the density of the accepting medium ($\rho=1,000$ kg/m³). Under these conditions, the capsule will be characterized by the pronounced flotation (Fig. 3) and the presence of a chemically passive zone S_s. The only possible condition for the mass exchange of ions (level 1) with Ca²⁺ through the surface of the capsule (S_c) is set the capsule into axial rotation in the liquid at speed (ω), which would enable controlled mass exchange through the surface.

Since the active surface of mass exchange is surface S_i, and surface S_s will objectively contact the air, then the actual specific surface of the contact when a capsule is in the accepting medium will be determined by the following expression:

$$S_{exchange\ capsule} = \frac{(S_c - S_s) \times \omega}{60}, \tag{2}$$

where S_c is the surface of the capsule; S_s is the part of the capsule surface that contacts the accepting medium; ω is the speed of capsule rotation in the accepting medium.

The speed of capsule rotation must exceed the rate of flow of AlgNa solution down the Plateau channels within the range of $r_c - r_o$ (Fig. 2), where r_c is the radius of the sphere of capsule, r_o is the radius of the sphere of an oil capsule inside the capsule.

With respect to the equation that the transformation rate of AlgNa to Alg_2Ca can be described by the following expression:

$$-\frac{dC_{Alg^-}}{d\tau} = \frac{dC_{Alg_2Ca}}{d\tau}, \tag{3}$$

where $-\frac{dC_{Alg^-}}{d\tau}$ is the speed of the transformation of sodium alginate in time, $\frac{dC_{Alg_2Ca}}{d\tau}$ is the rate of the formation of calcium alginate in time, then conditions for the material balance at each point in time

$$C_{Alg_2Ca} + C_{Alg^-} = 1, \tag{4}$$

where C_{Alg^-}, C_{Alg_2Ca} are the concentrations of sodium alginate and calcium alginate, respectively, then we can write the equation of kinetics:

$$-\frac{dC_{Alg^-}}{d\tau} = \frac{d[Alg_2Ca]}{d\tau} = k[Alg^-] \times [Ca^+], \tag{5}$$

where k is the rate constant of direct reaction in the graphic simulation of capsule as a sphere with cavity with known dimensional characteristics.

Since the formation of shell in the forming transporting liquid is the result of mass exchanges of participants in equation (1), it is theoretically important to estimate time (τ) of the process completion. Formation of the walls of the capsule is actually described by the equation of cation substitution. This means that the quantity of formation of shell $\frac{dC_{Alg_2Ca}}{d\tau}$ will be the magnitude proportionally connected to the transformation rate of AlgNa $\frac{dC_{Alg^-}}{d\tau}$. Given dimensional characteristics of the capsule, fixed through radius (r_c), and internal contents of lipids (r_o), it is possible to calculate the time of transition of a quasi-stable capsule to the thermodynamically equilibrium state with preset technological indicators.

Then the concentration of AlgNa Alg^-, and its change $\frac{dC_{Alg^-}}{d}$ in the technological flow can be recorded with respect to the ratio of the volume "wall of the capsule + lipid".

$$\frac{dc_{Alg^-}}{d\tau} = [Alg^-] = (V_c - V_o) \times c_{Alg^-} \times \rho_{solution\,Alg}, \tag{6}$$

where C_{Alg^-} is the concentration of sodium alginate by batch.

In the accepting medium of Ca^{2+} solution a change in the content of AlgNa in the capsule shell $-\frac{dC_{Alg^-}}{d}$ over time τ through surface S_c will be proportional to the variable concentration of calcium ions [C_{Ca}^{2+}].

$$[C_{Ca^{2+}}] = C_{Ca^{2+}} \times D \frac{dC_{Alg^-}}{d} \times \tau \times S_k, \tag{7}$$

where $D\frac{dC_{Alg^-}}{d\tau}$ is the concentration of sodium alginate, which passes into a gel of calcium alginate over time (τ).

Such an inequality will hold only under condition when the entire quasi-stable capsule is immersed into accepting medium of solution $C_{Ca^{2+}}$ at concentration $C_{Ca^{2+}}$, which will ensure the diffusion of Ca^{2+} through surface S_c at diffusion coefficient value D but under condition of inequality (8):

$$\rho_к < \rho_{Ca^{2+}}. \tag{8}$$

Archimedes' pushing power (F) during immersion of a capsule into accepting medium will cause capsule's flotation. A distribution model of the ion exchange surface area on the surface that is in contact with the air S_s and immersion S_i is shown in Fig. 3.
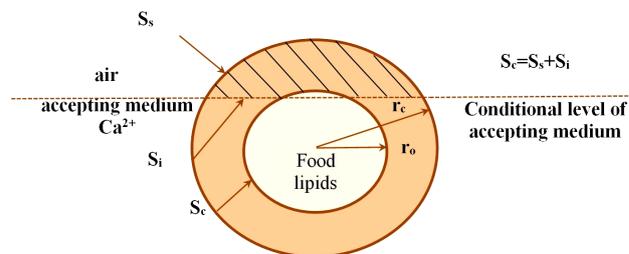

Fig. 3. Conditional distribution model of capsule surface (S_c) when entering the accepting medium of Ca^{2+}: ▨ — flotation; ◠ — part of the immersed capsule

Hence, effective area $S_c = S_s + S_i$ of ion exchange S_i will equal:

$$S_i = S_c + S_s. \tag{9}$$

Under these conditions, the formation of a capsule wall as a result of the accumulation of Alg_2Ca can be described by equation:

$$\frac{dc_{Alg_2Ca}}{d} = \left[k(V_c - V_o)c_{Alg^-} \times \rho\right] \times$$
$$\times D - \frac{dc_{Alg^-}}{d} \times \frac{(S_c - S_s) \times \omega}{60}. \tag{10}$$

Hence, the time of formation of the encapsulated product with preset properties that are derived from ratio $\frac{Alg_2Ca}{Alg^-}$, will equal:

$$\tau = \frac{60 \times \Delta X_2}{c_{Ca^{2+}} \times D \times \Delta X_1 \times S_i \times \omega \times k(V_c - V_0) \times c_{Alg^-}}. \tag{11}$$

Note that $\Delta X_1 - D\frac{dC_{Alg^-}}{d\tau}$; $\Delta X_2 - \frac{dC_{Alg_2Ca}}{d\tau}$. Then, taking into consideration that the extrusion head has extrusion channels of shell-former (r_c) and of food lipids (r_o), fixed to the ratio of the radii, and the volume of a sphere is calculated from formula $V = 3/4\pi r^2$, the expression takes the following form:

$$\tau = \frac{180 \times \Delta X_2}{4c_{Ca^{2+}} \times D \times \Delta X_1 \times S_i \times \omega \times (r_c - r_0) \times c_{Alg^-}}. \tag{12}$$

This equation describes patterns of capsule formation in time (τ) and reveals significant factors that are parts of

the equation. They should be fixed as the parameters of technological process – the concentrations of AlgNa and Ca^{2+}, dimensional characteristics of a capsule, frequency of capsule rotation in the accepting medium, etc. It is obvious that experimental research into relations between significant factors makes it possible to substantiate both parameters of the technological process and design features of encapsulation devices. Only when these conditions are met, it is possible, on the basis of analysis of the developed model, to devise a technological scheme of the actual technological process. The execution of the model would ensure obtaining food encapsulated lipids at stable and predictable quality level.

6. Discussion of results of analytical study of the encapsulation model of the system "food lipids – Alg_2Ca"

We developed and scientifically substantiated a technological process for obtaining encapsulated food lipids (using oils as an example) in the ionotropic shells based on Alg_2Ca. The analytical study is described of the encapsulation model of the system "food lipids – Alg_2Ca", which is carried out by the extrusion capsule formation through air in line with the principle "pipe in pipe". This process implies the creation of laminar flow of the shell-former and internal lipid contents. Next, the formation of a capsule takes place with the provision of shell-former's walls symmetry, followed by the separation of a quasi-stable capsule from the head's nozzle and its transportation through air into the accepting medium, which is the source of Ca^{2+}. The capsule has a phase of the contents of food lipids and the outer layer of a shell-former, which coats the contents. During contact between a drop of capsule and the accepting medium the shell-former enters chemical reaction with Ca^{2+}. The discovered floatation of a quasi-stable capsule leads to the formation of zone of "non-immersion" of the capsule, which makes the formation of a thermodynamic stable capsule impossible.

The gel-formation of shell in the technological flow is "broken"; the formation of the capsule "food lipids – Alg_2Ca" occurs, which is why a transition of the quasi-stable capsule to the thermodynamic stable state is, in its essence, a purely chemical process. The formation and spontaneous separation of a quasi-stable capsule follows from the laws of gravity [14], which predetermined technological and structural solutions for the extrusion of fluids vertically from top to bottom. In order to maintain stability of the technological process, potentials that are used to enable the motion of fluids must ensure a laminar flow of both liquids – the shell-former and the food lipid at the same preset rate, which considerably simplifies manageability of the technological process.

Application of the obtained results, specifically the establishment of dependences of the time of formation (12) of the thermodynamic stable capsule "food lipids – Alg_2Ca" on the magnitudes, which can serve as parameters of the technological process (the concentration of AlgNa (C_{AlgNa}) and ions of calcium ($C_{Ca^{2+}}$), dimensional characteristics of a capsule (r_c), mass ratios of a shell-former and lipid contents (r_o), design features of the device that enable rotation of a capsule around its axis (ω)), all this makes it possible to obtain a stable controlled technological process. It was established

that the axial rotation of a capsule in the laminar flow of accepting medium at speed $\frac{\omega}{60}$, which is ensured by a special design of the device, allows the formation of a capsule with preset product-technological properties over analytically determined time τ.

Given this understanding, the represented analytical study into encapsulation model of the system "food lipids – Alg_2Ca" is of practical significance and must be taken into consideration in the actual technological processes of manufacturing encapsulated food lipids.

There appears a prerequisite for the encapsulation of fats, solid at room temperature, in the molten state (at elevated temperatures). Obtaining such technological systems opens up prospects of optimization of many technological food processes, specifically when preparing products from dough, where hard-to-melt fats are the components of formulations. This applies primarily to the technology of yeast and puff pastry dough.

The possibilities of using in the composition of capsule shells additional gel-forming agents, such as agar, pectin, carrageenan, would requires further experimental research. A clear understanding of behavior of the technological system at the level of the examined model would provide reference points for exploring the influence of new components on the development of the proposed model. This makes the presented analysis of the encapsulation model a scientific foundation and a comparative basis for the development of technology.

Practical verification of analytical study and design solutions, in parallel with the substantiation of a composition formulation within the range of concentrations of AlgNa and Ca^{2+}, adequately confirm the data predicted.

Taking the analytical data into consideration, we designed an industrial sample of the device for obtaining capsules with the contents of food lipids; we devised technological regulations of the process, normalized quality indicators of the finished product; we developed and approved regulatory and technological documentation.

7. Conclusions

1. We designed and scientifically substantiated a technological process for obtaining encapsulated products with the contents of food lipids. The study of the analytical model of the encapsulation of the system "food lipids – Alg_2Ca" allowed us to establish the feasibility of extruding working liquids, the participants of the technological process, vertically, from top to bottom, employing the principle "pipe in pipe". We proved the necessity for the axial rotation of the formed quasi-stable capsule in the accepting medium of Ca^{2+} solution. It is confirmed that the establishment of dependences of the capsule formation time on the concentration of AlgNa (C_{AlgNa}) and calcium ions ($C_{Ca^{2+}}$), dimensional characteristics of a capsule (r_c), mass ratios of a shell-former and the lipid contents (r_o), design features of the device that ensure capsule rotation around its axis (ω), makes it possible to accomplish a controlled technological process.

2. We studied, substantiated, and devised a model of the technological process for obtaining thermodynamically stable capsules based on the shell-former AlgNa with the contents of food lipids. The required time (τ) is calculated,

needed by a quasi-stable capsule to be in the accepting technological medium of Ca^{2+}. The analysis reported is a scientific foundation and a scientific basis for the development of technology aimed at introducing to the technological process of lipids, different in origin and properties, and us-ing in the composition of capsule shells various gel-forming agents, such as agar, pectin, carrageenan, etc. This ensures the obtaining of encapsulated products with the contents of food lipids with high preset product and technological characteristics.

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