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Sodium alginate emulsions for asphalt concrete modifiers encapsulating: structural rheological properties

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Abstract. The ratio of components for the production of alginate emulsions are established, which can be used in the design of compositions for encapsulating various types of reducing agents. The high resistance of alginate emulsions to segregation corresponds to a structured system, the onset of the destruction of which is determined by the ratio O/A. The structure of stable alginate emulsions is characterized by an average particle size of the dispersed phase from 5 to 7 μm , the average distance between which is from 7 to 9 μm . The change in the particle size distribution of the dispersed phase occurs during the process of water evaporation from the alginate emulsion, which is explained by a decrease in the volume of the dispersion medium, the approach of the particles, their collision, compression, and pooling. An increase in particle diameter occurs on average by 28 % over 5 days, which is accompanied by an increase in viscosity by 4 times.

1. Introduction

The developing construction industry needs materials with unique properties that increase the functionality and reduce the resource consumption of structures during construction and operation. The creation of "smart" materials is a promising solution in the field of materials science, aimed at increasing the durability of structures [1–3]. The formation of their own internal impacts, which ensures the maintenance of structure parameters at the required level, is a key condition for classifying the material as "smart". "Reactions" of the material to a change in the structure parameters under the influence of operational factors of natural and/or artificial origin are provided at the material design stage. Materials, including asphalt concrete, with the ability to restore their own functionality in construction are a promising type of smart materials for pavement. This property of the material is usually called "self-healing" [3, 4].

The durability of asphalt concrete is inversely proportional to the speed and intensity of the course of destructive processes. Thus to increase the service life of the asphalt concrete pavement solutions are needed to ensure the duration of the state of the structure of asphalt concrete without defects due to giving it unique properties, independently restore the integrity of the composite and its ability to resist influencing factors.

Developments using encapsulated reducing agents are more common among technological solutions for the formation of this new operational property [5–12]. The encapsulated reducing agent is introduced into the asphalt mix in the form of capsules of various shapes made on the basis of polymer compounds formed during curing of the capsule [13–17].

The authors of [18] proposed a technologically simple method for producing capsules that can be used as part of an asphalt mix. An aqueous solution based on sodium alginate ($\text{C}_6\text{H}_7\text{O}_6\text{Na}$), which is the sodium salt of alginic acid extracted from brown algae. is used to encapsulate the reducing agent. The effectiveness of the use of calcium-alginate capsules is determined by the complex of physical-mechanical properties of the capsules and the technological properties of the emulsion for their production. The properties of alginate emulsions determine the capsule production rate, their size and the reducing agent content [19]. Therefore, the determination of the influences of prescription factors on the structure and properties of emulsions will make it possible to control the physical-mechanical properties of capsules. This will expand the list of reducing agents that can be encapsulated using alginate technology.

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Sodium alginate is poorly wetted by water because the particles of alginate powder intensively stick together and form aggregates. Effective preparation of the emulsion using sodium alginate is achieved by high-speed mixing to destroy agglomerates [18]. During high-speed mixing sodium alginate interacts with water and forms a colloidal solution.

Capsules obtained by various methods have sizes from 10 μm to 3 mm. in which the encapsulated reducing agent is predominantly vegetable oils [20, 21, 24–27]. Industrial rejuvenator is an Alternative variant of the reducing agent, which is a mixture of low molecular weight compounds and oils [22–24, 27].

As a rule, the capsule shape is spherical or ellipsoidal, which facilitates their use in the process of mixing with other components of the asphalt mix [21, 22]. The technology has a significant drawback if the container for the reducing agent is fiber: premature destruction of the containers during mixing and during operation.

The highest content of the reducing agent in the capsules is achieved in the shells of calcium alginate, which is explained by the simplicity of the preparation technology and a wide range of variable dosages of the components.

It should be noted that bitumen has its own potential for self-healing, which depends on both physical and chemical properties. Self-healing of a binder in asphalt concrete occurs in the absence of a dynamic effect, which is aimed at restoring fatigue damage in the material. The degree of recovery is determined by the relaxation time [28].

Rejuvenators, organic oils with different molecular weights are used as a reducing agent by the authors of all the works reviewed. The mechanism of action of such a reducing agent in asphalt concrete is the dissolution of bitumen components aged during operation and local reduction of fragility. In bitumen self-healing occurs at the molecular level due to reversible hydrogen bonding with the formation of new cross-links and chains [28, 29] through ditopic and tritopic molecules [30]. In the case of oil-based oil rejuvenators these processes do not occur, therefore the effect of combining bitumen molecules is not observed. There is only a partial dilution of the components of bitumen in the rejuvenator, which contributes to reducing the potential for the development of a crack.

A search is needed for compounds that will provide a similar recovery mechanism to improve self-healing technology. However, it was established [31] that sunflower oil traditionally used as a reducing agent also has a structuring effect on the properties of the emulsion. Therefore, the reducing agent appears to be a mixture of a functional compound that provides a reduction mechanism and a structuring component (sunflower oil or similar) that provides stable emulsions for encapsulation. In this regard one of the important tasks of the technology of encapsulation of a reducing agent is to determine the maximum concentration of sunflower oil, which provides an emulsion with predetermined geometric characteristics and rheological properties.

2. Methods

An aqueous solution based on sodium alginate is used to prepare an alginate emulsion. Sodium alginate ($\text{C}_6\text{H}_7\text{O}_6\text{Na}$) is a sodium salt of alginic acid extracted from brown algae. A sunflower oil-based reducing agent has been used as an ordinary rejuvenator. The main properties of sunflower oil are presented in Table 1.

Table 1. The main properties of sunflower oil.

Parameter	Unit	Value	Method
Viscosity at 25 °C	Pa·s	0.05	–
Density at 25 °C	g/cm^3	0.918 ± 0.05	ISO 3675 ISO 3838
Acid value	mg KOH/g	0.025 ± 0.01	ISO 660
Fractional composition:			
palmitic acid	%	6.61	
stearic acid	%	3.61	
oleic acid	%	30.91	ISO 5508
linoleic acid	%	57.13	
other	%	1.74	

Samples of water emulsions with different contents of sodium alginate from 1.0 to 3.0 % and sunflower oil from 5 to 30 % were made to study the influence of prescription factors on the geometric parameters and rheological properties of alginate emulsions as well as to establish the intensity of the influence of sunflower oil on their structure formation.

Alginate emulsions were obtained by mixing sodium alginate and sunflower oil in water in various proportions and mixing for 2 minutes using an overhead stirrer with a drive rotation speed of at least 2000 rpm [32].

The dynamic viscosity and yield strength of the emulsion were determined using a MCR 101. Anton Paar rotational viscometer using a coaxial cylinder measuring system (Fig. 1). The outer cylinder was filled with an emulsion sample and the inner cylinder was lowered into the sample using an automatic drive. The sample was thermostated at 25 °C for 30 minutes before measurement. After that dynamic viscosity was determined at a constant shear rate of 50 s⁻¹. The viscosity value was calculated as the arithmetic average of 10 measurements under given conditions. At the next stage to determine the yield strength the shear stress was measured during the increase in the shear rate to 3500 s⁻¹. Yield strength was determined according to the scheme shown in Fig. 2. The arithmetic average of at least 3 measurements is the result of determining the viscosity and yield strength.

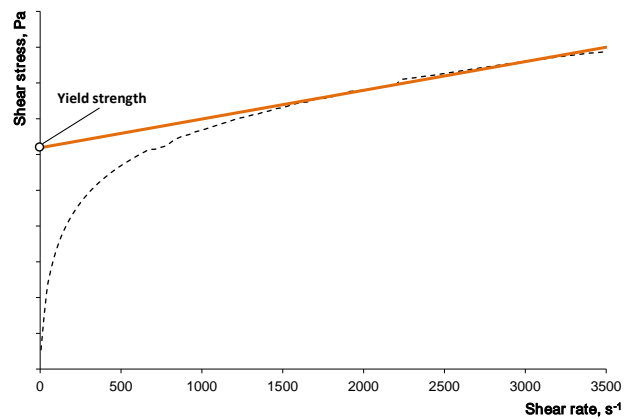
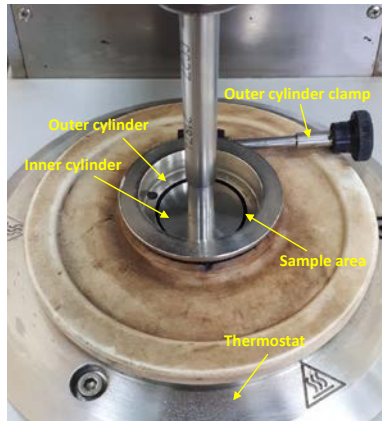


Figure 1. Coaxial cylinder measuring system. Figure 2. Yield strength determination scheme.

The structural parameters of alginate emulsions were studied using a Nikon Eclipse MA200 optical microscope using Thixomet software with calibrated electronic instruments and scales. The geometric parameters of the structure of alginate emulsions were measured at a zoom of 200 times.

Changes in the mass of alginate emulsion samples were measured every 24 hours for 18 days using electronic scales with an accuracy of 0.001 grams. Samples of alginate emulsions were stored in a beaker without a cover under natural conditions at a temperature of 24 ± 0.1 °C and air humidity of 50 %. The arithmetic average of at least 3 measurements is the result of determining the changes in the mass of alginate emulsion.

The selection of mathematical models of the dependence of changes in the properties of emulsions was carried out by the coefficient of determination coefficient, which is presented in Table 2.

Table 2. Coefficients of determination.

Parameter	Coefficient of determination R^2 for O/A ratio					Figure
	3.0	2.3	1.8	1.3	1.0	
Viscosity	0.98	0.96	0.95	0.94	0.93	3
Yield strength	1.00	1.00	1.00	0.97	0.94	5
Number of contacts	1.00	1.00	1.00	0.99	0.99	6
Changing O/A ratio			0.99			7
Start time of the destruction			0.99			9

The values recognized as an error in the measurement were not used in the calculation and the tests were repeated.

The results of statistical processing of viscosity data (standard deviation and coefficient of variation) are presented in Fig. 3.

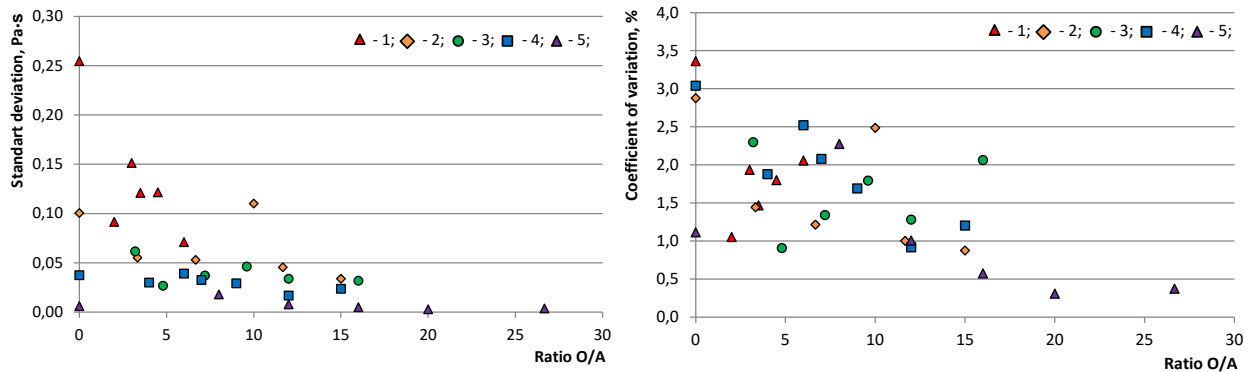


Figure 3. Results of statistical processing of viscosity data with the content of sodium alginate: 1 – 3.0 %; 2 – 2.3 %; 3 – 1.8 %; 4 – 1.3 %; 5 – 1.0 %.

It is obvious that standard deviation and coefficient of variation decrease slightly with a decrease in the content of sodium alginate and an increase in the content of reducing agent in the emulsion. This is due to the improvement of technological properties, which allows to obtain emulsions with greater uniformity. The results of statistical processing of yield stress data have a similar dependence of the change on the content of emulsion components.

The results of statistical processing of data on the particle diameter of the emulsion are presented in Table 3.

Table 3. Statistical analysis of data on the particle diameter of the emulsion.

Param.	Parameter value for O/A, μm														
	SA = 3.0 %			SA = 2.3 %			SA = 1.8 %			SA = 1.3 %			SA = 1.0 %		
	0.95	1.6	2.85	1.4	2.8	4.2	3.2	6.4	9.6	7.85	15.7	25.0	12.73	25.47	33.0
Min	2.1	2.0	2.0	1.8	0.9	1.7	2.0	2.1	1.8	2.1	1.9	2.1	2.1	2.1	2.8
Max	10.1	18.3	16.3	10.2	21.4	16.3	18.2	26.5	20.8	19.0	17.6	21.5	37.6	20.9	60.6
R	8.0	16.4	14.3	8.4	20.5	14.6	16.2	24.4	19.0	16.9	15.7	19.4	35.5	18.8	57.8
\bar{X}	4.8	5.4	7.0	5.0	5.6	6.5	5.9	6.6	7.6	7.4	6.9	8.5	14.0	8.6	12.4

Note: Param. is parameters; SA is sodium alginate content; Min is minimum sample value; Max is maximum sample value; R is sample range; \bar{X} is expected value.

The data sample size for measuring the particle diameter of the emulsion was at least 100 values.

3. Results and Discussion

The main technological property in accordance with the encapsulation technology, which ensures the formation of capsules with specified sizes and wall thickness, is the viscosity and yield strength of the emulsion. Determining the ability of alginate emulsions of different composition to resist the flow will allow establishing the presence of a structuring effect that sunflower oil has. The study of the effect of sunflower oil on viscosity was evaluated on the composition of emulsions with different contents of sodium alginate the results are shown in Fig. 4.

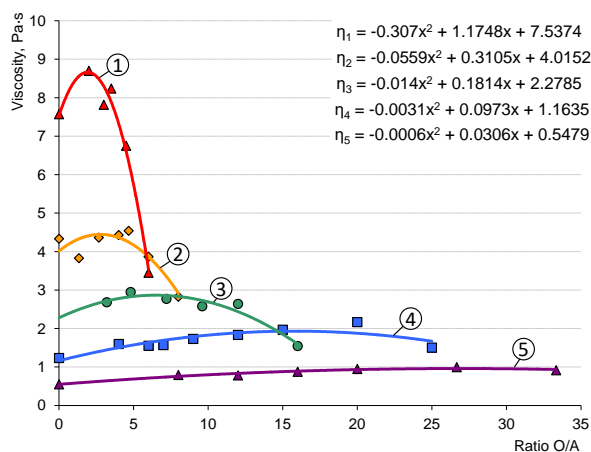


Figure 4. The dependence of the change in viscosity of emulsions on the ratio O/A (Oil content/Sodium alginate content) with the content of sodium alginate: 1 – 3.0 %; 2 – 2.3 %; 3 – 1.8 %; 4 – 1.3 %; 5 – 1.0 %.

It should be noted that the change in viscosity for emulsions with different contents of sodium alginate has extremum with the increase in the content of sunflower oil. It indicates the contribution of sunflower oil to the formation of the structure of the emulsion and its ability to resist flow. The concentration of sunflower oil the introduction of which ensures the maximum viscosity of the emulsion is equal to:

$$C_{oil} = C_{al} \frac{b}{2a}, \quad (1)$$

where C_{al} is the concentration of sodium alginate; a , b are the empirical coefficients of the equation $\eta(r) = -ar^2 + br + c$; r is the ratio of O/A.

According to the values of empirical coefficients $\eta(r) = f(r)$ presented in Fig. 3 the dependence

$$\frac{dC_{oil}}{dC_{al}} = \frac{40.5}{C_{al}^{2.5}}, \quad (2)$$

which clearly shows that the intensity of the sunflower oil structuring effect depends on the concentration of sodium alginate. From this dependence it follows that the decrease in the content of sodium alginate leads to the increase in the amount of sunflower oil necessary for maximum structural effect.

The extremum dependence of viscosity changes indicates the structural differences of emulsions with different component contents, which affect the ability to resist flow characterizing the manufacturability and efficiency of the process of obtaining capsules with a modifier.

The particle size distribution of emulsions with different contents of alginate and sunflower oil is shown in Fig. 5.

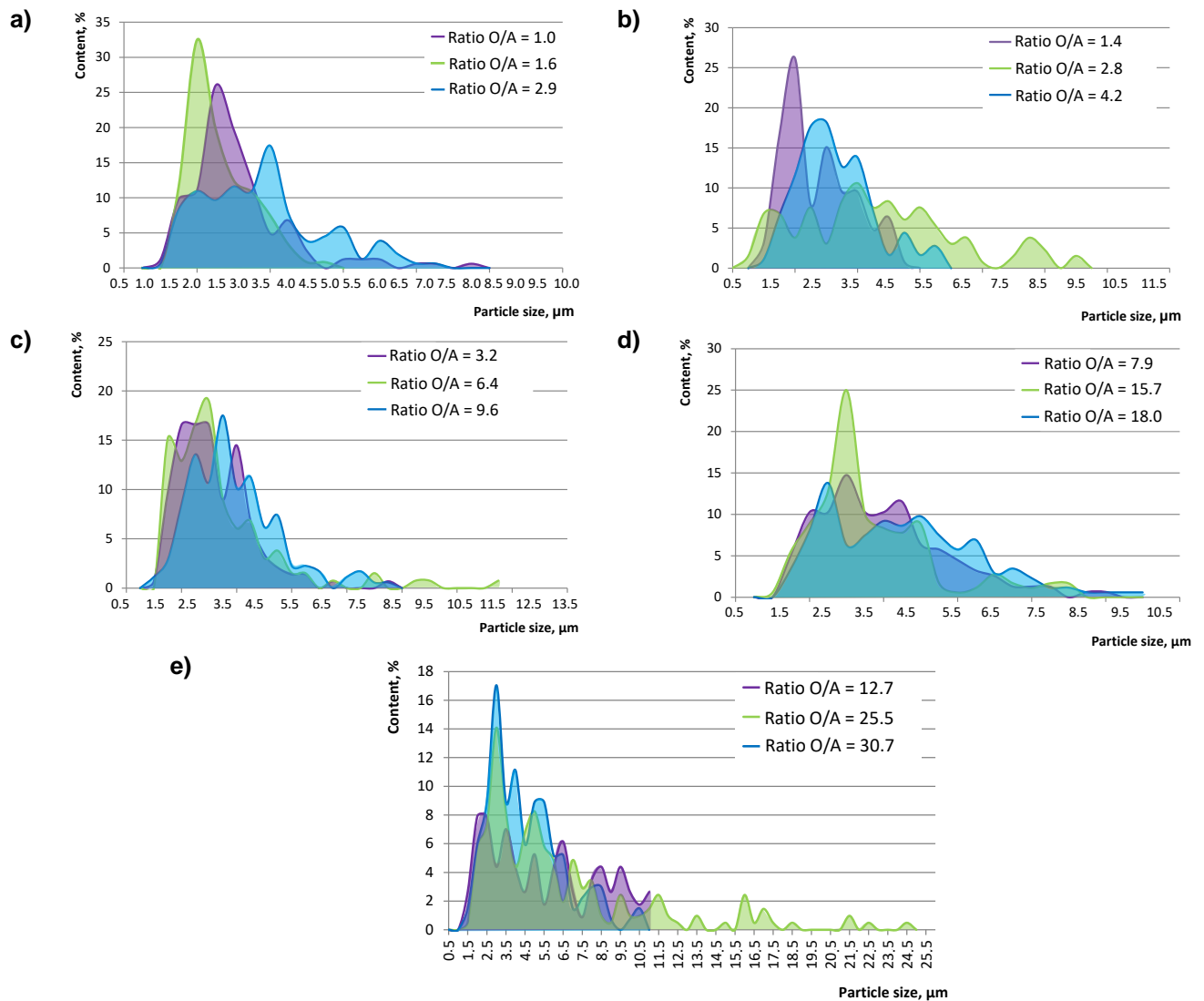


Figure 5. Particle size distribution of the dispersed phase in the emulsion with the content of sodium alginate: a) 3.0 %; b) 2.3 %; c) 1.8 %; d) 1.35 %; e) 1.0 %.

An analysis of particle size distribution of the dispersed phase in the emulsion shows that the average particle diameter increases with the increase in the O/A ratio and with the decrease in the total content of sodium alginate, which indicates a structuring effect. A detailed analysis of the particle size distribution of the ordinary emulsions and emulsions after aging is presented in Table 2. The number of contacts of the particles of the dispersed phase in the emulsion will be determined to take into account the obtained particle size distribution of its:

$$\chi = \sum_{i=1}^n \frac{C_i}{d_i^2}, \tag{3}$$

where d_i is the particle diameter of the i -th fraction, m; C_i is content of the i -th fraction; n is the number of fractions.

The results of calculating the number of contacts of dispersed phase particles in alginate emulsions are presented in Fig. 6.

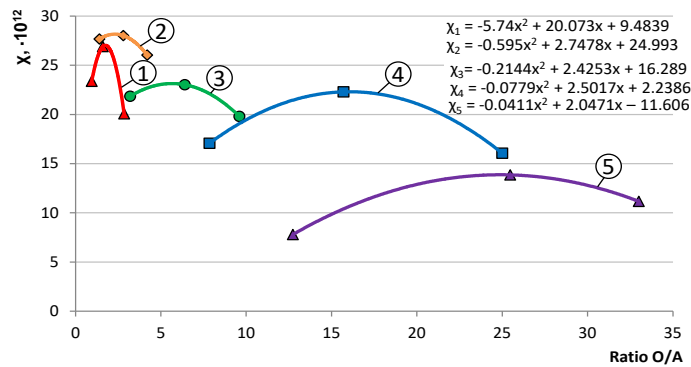


Figure 6. The dependence of the change in the number of contacts of the particles of the emulsion on the ratio O/A with the content of sodium alginate: 1 – 3.0 %; 2 – 2.3 %; 3 – 1.8 %; 4 – 1.3 %; 5 – 1.0 %.

Fig. 6 shows that the abscissas (ratios O/A) of the maxima for two groups of the indicated characteristics (viscosity and the number of contacts) are located quite close: as a rule, the maxima $\chi_i = f(O/A)$ are achieved at lower values of the ratio O/A. The general model of the interdependence of structural parameters and mechanical properties is the model of a homogeneous solid (solids with uniformly distributed bonds; for the emulsion at the first stage of analysis, this model is applicable) proposed by P.A. Rebinder [33, 34]. For the case under consideration (emulsion) the Rebinder model can be represented as:

$$\eta = k \cdot f_c \cdot \chi^n, \tag{4}$$

where f_c is the contact strength; k, n are constants (in the classical Rebinder formula $n = 2/3$).

The presented model demonstrates that the number of contacts, which in accordance with (3), nonlinearly depends on the particle diameter (particle size distribution of the dispersed phase of the emulsion) is a key factor affecting the rheological properties of the emulsion (viscosity, yield strength of the emulsion – Fig. 7).

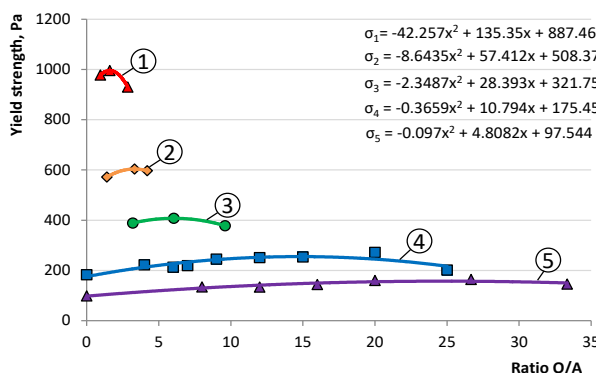


Figure 7. The dependence of the change in yield strength on the ratio O/A with the content of sodium alginate: 1 – 3.0 %; 2 – 2.3 %; 3 – 1.8 %; 4 – 1.3 %; 5 – 1.0 %.

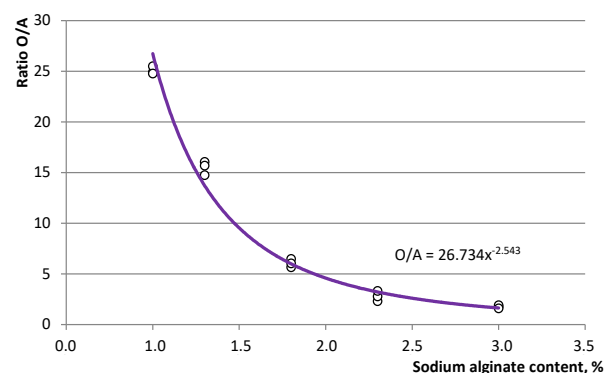


Figure 8. Dependence of changes in the ratio O/A on the content of sodium alginate for emulsions with maximum viscosity, yield strength, and the number of contacts.

An analysis of Fig. 4, Fig. 6 and Fig. 7 shows that the extremums in the graphs for the disperse systems under study with different contents of sodium alginate are observed at similar ratios O/A. It allows determining the dependence of the control parameter O/A on the content of sodium alginate for the development of encapsulation technology (Fig. 8), which is consistent with the following results [35]. From this figure the structuring function of sunflower oil is visible, which is a nonlinear dependence of its content on the amount of sodium alginate:

$$C_{oil} = \frac{26.734}{C_{al}^{1.543}} \quad (5)$$

The calculation of the curvature coefficient was carried out to analyze the obtained dependence (Fig. 9):

$$k = \frac{y'}{(1 + y'^2)^{3/2}}$$

where y' and y'' are the first and the second derivative, respectively.

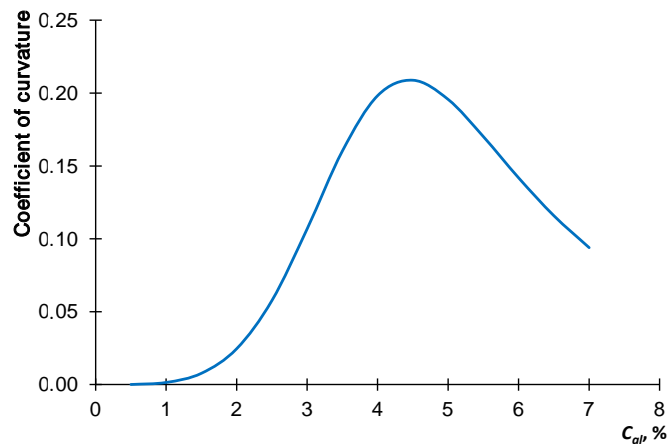


Figure 9. Curvature coefficient of the function (5).

Analysis (5) by standard methods shows that the coefficient of curvature of the function has a maximum at $C_{al} = 4.5\%$ indicating that the effect of the concentration of sodium alginate on the content of sunflower oil and its structuring effect is not significant. The resulting relationship indicates that the selected concentration of sodium alginate is correct.

The encapsulation process includes the step of dividing the alginate emulsion into individual particles and is a process with a fixed duration, which depends on the characteristics of the separatory funnels and the properties of the emulsions. Aqueous alginate emulsions and solutions obtained on their basis are unstable in time, since under natural conditions water evaporates (Fig. 10), which contributes to a change in the properties of the entire dispersed system.

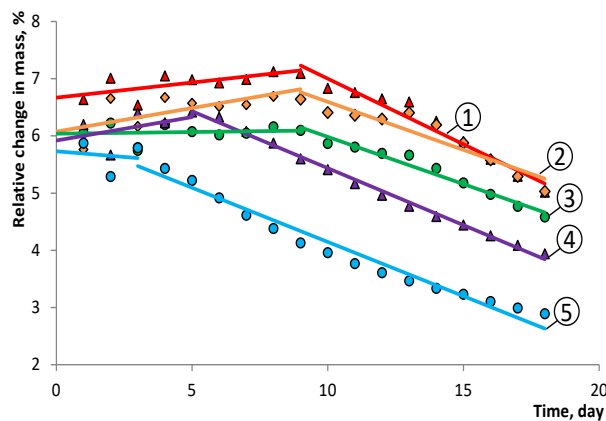


Figure 10. The dependence of the mass change of the samples of emulsions with the content of sodium alginate: 1 – 3.0 % (O/A=1.9); 2 – 2.3 % (O/A=2.8); 3 – 1.8 % (O/A=6.4); 4 – 1.5 % (O/A=9.4); 5 – 1.3 % (O/A=15.7).

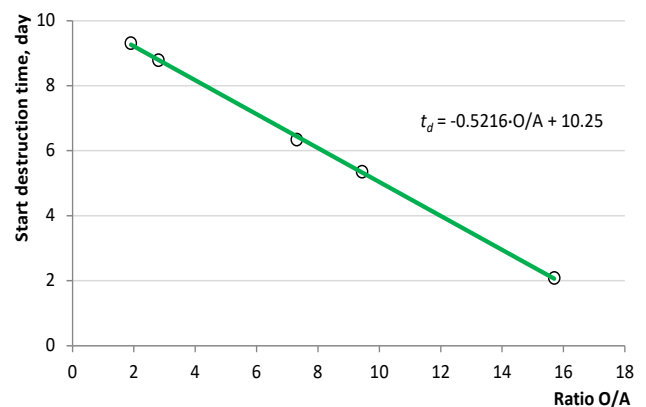


Figure 11. The dependence of the start time of the destruction of the emulsion on the ratio O/A.

The two main stages can be distinguished on the dependencies of the mass change of the emulsion samples over time, where at the initial stage a constant mass change is observed, which indicates the preservation of the dispersion system. The beginning of the next stage is the beginning of segregation and destruction of the emulsion, after which an oil film is formed on the surface of the sample and prevents further evaporation of moisture at the same rate. Water evaporation is slowed down more strongly in emulsions with lower sodium alginate content and a high O/A ratio, which indicates more intense destruction. The segregation stages in such emulsions begin earlier (Fig. 12).

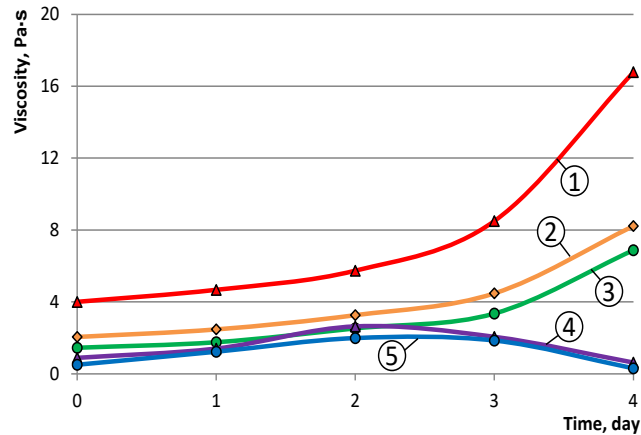


Figure 11. The dependence of the change in viscosity of emulsion samples with the content of sodium alginate: 1 – 3.0 % (O/A=1.9); 2 – 2.3 % (O/A=2.8); 3 – 1.8 % (O/A=6.4); 4 – 1.3 % (O/A=15.7); 5 – 1.0 % (O/A=19.1).

The change in the viscosity of the studied alginate emulsions confirms the time of onset of segregation in emulsions with a sodium alginate content of less than 1.3 % at a ratio $O/A > 15$, which happens two days after preparation. Maximum viscosity is observed on the second day for samples 4 and 5, after which the separation occurs. the alginate emulsion is destroyed and the viscosity is decreased. This is consistent with the results of the mass loss determination obtained previously. The increase in viscosity is observed more than 4 times for compositions 1...3 (Fig. 12) during the study period. no emulsion segregation was observed. which began not earlier than after 7 days (Fig. 11). Stability alginate emulsions increase up to 21-28 days when storing in an airtight screw-cap vessel [35, 36].

The change in the properties of alginate emulsions depends on the parameters of its structure, which are varied with a change in composition after the evaporation of water. The results of the study of changes in the structural parameters of alginate emulsions over time are presented in Table 4.

Table 4. Changes in the structural parameters of alginate emulsions.

#	Parameters	Ratio O/A	Sodium alginate content, %	The value of the parameter in a day, μm					
				0	1	2	3	4	5
1	Max diameter	1.9	3.0	18.21	18.54	18.87	19.20	19.53	19.86
2		2.8	2.3	19.26	19.50	19.74	19.98	20.22	20.46
3		6.4	1.8	29.50	28.70	27.90	27.10	26.30	25.50
4		15.7	1.3	18.97	28.14	37.31	46.48	55.65	64.82
5		27.2	1.0	22.78	25.21	27.64	30.07	32.50	34.93
6	Minimum diameter	1.9	3.0	1.90	1.96	2.02	2.09	2.15	2.21
7		2.8	2.3	1.10	1.39	1.68	1.97	2.26	2.56
8		6.4	1.8	2.47	2.10	1.73	1.37	1.00	0.63
9		15.7	1.3	1.99	2.23	2.47	2.71	2.95	3.19
10		27.2	1.0	2.40	2.20	2.00	1.81	1.61	1.41
11	Average diameter	1.9	3.0	5.44	5.66	5.89	6.12	6.34	6.57
12		2.8	2.3	5.64	5.85	6.06	6.27	6.48	6.69
13		6.4	1.8	6.68	7.05	7.41	7.77	8.13	8.49
14		15.7	1.3	7.03	7.99	8.96	9.92	10.89	11.86
15		27.2	1.0	8.95	9.04	9.12	9.21	9.30	9.38
16	The distance between the particles of the dispersed phase	1.9	3.0	8.65	6.93	5.22	3.51	1.79	0.08
17		2.8	2.3	8.96	6.82	4.68	2.54	0.41	–
18		6.4	1.8	7.62	5.96	4.29	2.62	0.96	–
19		15.7	1.3	2.85	2.33	1.81	1.29	0.76	0.24
20		27.2	1.0	2.12	1.63	1.14	0.64	0.15	–

Analysis of changes in the structural parameters of alginate emulsions shows that the particle size distribution changes during the evaporation of water the boundaries of the maximum and minimum particle sizes of the dispersed phase are increased. and the average diameter increases. The increase in particle diameter occurs on average by 28 % within 5 days. Alginate emulsions with clove oil with a particle diameter of 1 to 10 μm are also obtained in [35]. The change in the particle size distribution of the dispersed phase of the emulsion is caused by the decrease in the volume of the dispersion medium during the evaporation of water the approach of the particles together their collision, compression and pooling (Fig. 13).

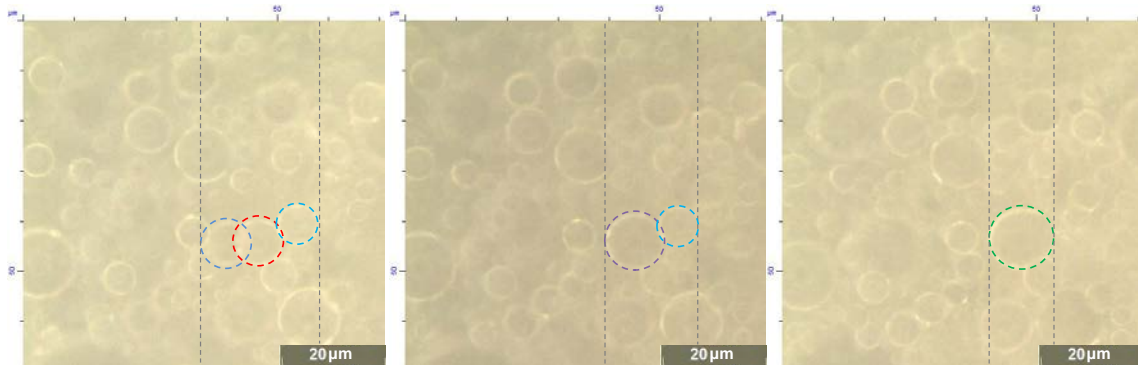


Figure 13. Micrograph of the process of pooling particles of the emulsion (from left to right).

The most part of the dispersed phase is in contact after 4 ... 5 days. which is accompanied by a sharp increase in their viscosity for stable emulsions (sodium alginate content of 1.8...3.0 %). A similar mechanism according to which there is a sequential increase in the particle size of the emulsion is observed during encapsulation, when individual drops of the emulsion are converted into capsules with an alginate shell during drying [37, 38]. Calcium is a component that prevents the premature destruction of emulsions in such technology during the formation of individual drops from emulsions [23, 25, 27, 38].

4. Conclusion

1. The ratio of components for the production of alginate emulsions are established, which can be used in the design of compositions for encapsulating various types of reducing agents. The range can be calculated by changing the O/A ratio depending on the content of sodium alginate, at which the maximum viscosity of the emulsions is achieved $C_{ai}: O/A = 26.734C_{al}^{-2.543}$.

2. The high resistance of alginate emulsions to segregation corresponds to a structured system the onset of the destruction of which is determined by the O/A ratio and can be described by the dependence $t_d = -0.5216 \cdot O/A + 10.25$.

3. The structure of stable alginate emulsions is characterized by an average particle size of the dispersed phase from 5 to 7 μm , the average distance between which is from 7 to 9 μm .

4. The change in the particle size distribution of the dispersed phase occurs during the process of water evaporation from the alginate emulsion, which is explained by the decrease in the volume of the dispersion medium, the approach of the particles, their collision, compression and pooling. The increase in particle diameter occurs on average by 28 % over 5 days. which is accompanied by the increase in viscosity by 4 times.

5. Acknowledgments

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