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Method of forecasting the effectiveness of cationic bitumen emulsions

Метод прогнозирования эффективности катионных битумных эмульсий

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Ключевые слова: битумная эмульсия; эмульгатор; поверхностно-активные вещества; поверхностное натяжение; межфазные границы

Abstract. Approbation of the procedure for predicting the production of effective bitumen emulsions at the stage of water phase preparation and also predicting the compatibility of the mineral aggregate and the surface-active substance used to emulsify the bitumen was carried out. As criteria characterizing the optimal composition of water phases, the following indicators were proposed: the surface tension of the water solutions under investigation and the wetting contact angle. The study of the dynamics of changes in the properties of the water phase and surface tension at the interface of the "water phase–mineral substrate" system during the emulsifier concentration change made it possible to establish that in the range of the emulsifier content for the bitumen emulsion recommended by the manufacturer, there are ineffective concentrations, the effect and variability of which is manifested in case of contact with mineral materials of different nature. The possibility of early prediction of the compatibility of the emulsifier used to prepare the bitumen emulsion and the mineral material, which will interact with the production emulsified binder, was evaluated.

Аннотация. Проведена апробация метода прогнозирования получения эффективных битумных эмульсий на стадии подготовки водной фазы, а также прогнозирования совместимости минерального заполнителя и поверхностно-активного вещества, используемого для эмульгирования битума. В качестве критериев, характеризующих оптимальные составы водных фаз, предложены показатели: поверхностного натяжения исследуемых водных растворов и краевого угла смачивания. Изучение динамики изменения свойств водной фазы и поверхностного натяжения на границе раздела системы «водная фаза-–минеральная подложка» при изменении в ней концентрации эмульгатора, позволило установить, что в рекомендуемом производителем интервале содержания эмульгатора для битумной эмульсии, существуют неэффективные концентрации, действие и изменчивость которых проявляется при контакте с минеральными материалами различной природы. Произведена оценка возможности раннего прогнозирования совместимости эмульгатора, используемого для приготовления битумной эмульсии и минерального материала, который будет взаимодействовать с производственным эмульгированным вяжущим.

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1. Introduction

The present moment can be attributed to the era of high-tech and economical technical solutions in virtually all segments of the economy. These factors determine the increased interest of various fields in emulsions – dispersed heterogeneous colloidal systems. In road construction, as well as in the production of protective coatings and adhesives in the construction industry, the bitumen emulsion is also a popular and relevant type of binder. This is due to the fact that emulsions combine many advantages.

However, it is a heterogeneous – inhomogeneous system, consisting of heterogeneous phases, differing in composition and properties, and separated by the interface. Therefore, it is difficult to develop and produce an emulsion, regardless of the field of its application, which consists in the correct choice of surface-active substances, as well as their concentrations to exclude or reduce the destruction processes and increase the kinetic stability of the entire system. As practice shows, the calculation of the amount of surface-active substances that is necessary for the stabilization of heterogeneous systems is often carried out experimentally without justification from the point of view of the colloidal system.

In the road construction segment, bitumen road cationic emulsions (BRCE) are the most common ones; they most often refer to the type of oil-in-water emulsions when bitumen is dispersed in the water phase [1–4]. To ensure this process, it is necessary to equalize the surface tension of two phases – bitumen and water. For this purpose, a certain amount of emulsifier is introduced into the water phase. It must be taken into account that the properties of colloidal dispersions depend significantly on the nature of the interface between the dispersion phase and the dispersed medium. Despite the large surface-to-volume ratio, the amount of surface-active substances required for a significant change in the volumetric properties of colloidal disperse systems is very small [5]. In this case, each emulsifier has a minimum limit of its concentration, at which emulsification is possible. A large amount of surface-active substances is an undesirable factor contributing to a slowing down of the emulsion breakdown rate – the time for which the adhesion-cohesive bonds will reach the level necessary for the formation of the composition structure, and also leading to an increase in the cost of production and the total cost of production [6].

These factors contribute to the fact that at the present time, when developing the formulation of bitumen emulsions and determining the optimum content of surface-active substances in them, test batches of emulsified binder with various surface-active substance concentrations are prepared. With the subsequent study of the main processing and operational characteristics of the prepared disperse systems. At this, such selection is long, energy-intensive and resource-intensive, and also does not take into account the specifics of a particular type of the aggregate that will contact the emulsion. The importance of the compatibility of the mineral substrate with the bitumen emulsion was noted in the study [7], where it is proposed to optimize the choice of the emulsifier in accordance with the type of mineral material used in the composition in order to achieve the maximum efficiency of preparation and use of the bitumen emulsion. The efficiency is evaluated on the system "stone material-bitumen emulsion".

Based on the foregoing, the goal of scientific research was formulated, it consists in developing the procedure for predicting the efficiency of cationic bitumen emulsions at the early stage of their selection. To do this, it is necessary to solve the problem of identifying algorithms for determining the effective content of the emulsifier in the water phase, to ensure the production of stable cationic bitumen emulsions, and also to predict the compatibility of the mineral aggregate and surface-active substances used to emulsify the bitumen.

2. Methods

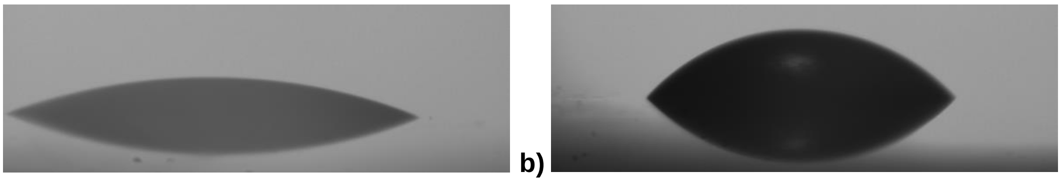
In this study, the procedure is tested for determining the effective content of an emulsifier in the water phase for the preparation of stable cationic bitumen emulsions, and also predicting the compatibility of the mineral aggregate and surface-active substances used for the emulsification of bitumen. The following indicators were taken as criteria characterizing the optimum concentrations of emulsifiers in the water phase: surface tension of the solutions under study and their wetting contact angle for substrates of red granite and white marble.

The proposed method is based on the study of the dynamics of changes in the properties of the water phase and the interface of the "water phase – mineral substrate" when its surface-active substance concentration changes.

The choice of substrate type is explained by the opposite of their chemical properties. Granite refers to acid rocks, and marble to basic ones. In this connection, it can be assumed that the manifestation of the properties of the water phase upon contact with these mineral materials will be different. The substrates of the rocks used were previously prepared: sawn to plates, washed, dried, ground, then washed again and dried.

The surface tension of the water phases was studied on the Lauda TVT 2 instrument, which allows to determine the dynamic parameters of the surface and interfacial tension of liquids by the drop volume method [8]. The method is based on an accurate measurement of the volume of droplets when they fall from the dosing capillary. The value of surface or interfacial tension is calculated on the basis of the volume of droplets, depending on whether droplets form in the air, or in another, immiscible phase (oil). Measuring of droplets accurate to microliter provides high accuracy and reproducibility of measurement results.

To study the wetting contact angle, there were used droplets (Fig. 1), which were obtained with a digital camera. The wetting angle was calculated using the droplet height and width values obtained experimentally [9].



a) b)

Figure 1 Form of the water phase droplet with the REDICOTE EM-44 emulsifier on the substrate of a) marble, b) granite

The contact angle of wetting is a characteristic of the hydrophilicity or hydrophobicity of the surface, and also serves as a characteristic of the adsorption that occurs when the water phase and the mineral substrate come into contact. Such contact, in some cases, can be accompanied by chemical interaction of liquids and minerals, ion exchange, and also dissolution and electrokinetic phenomena [5].

3. Results and Discussion

The experiment was performed on cationic emulsifiers produced by AkzoNobel: REDICOTE E-11 and REDICOTE EM-44. The choice of additives is justified by the difference in the mechanism of their action on the emulsion. In accordance with the manufacturer's product specification, REDICOTE EM-44 is a liquid emulsifier for bitumen cationic road emulsions with a rapid breakdown rate (EBKD B), which also acts as an adhesive additive. The recommended concentration for quick-breaking emulsions is 0.12–0.25 %, REDICOTE E-11 is a liquid emulsifier for bitumen road cationic emulsions with a slow decay rate (EBDK M), its recommended concentration in the water phase is 0.6–1.5 %. The basis for the production of REDICOTE additives is polyamines [4, 10–12].

The laboratory studies performed earlier [13] made it possible to determine the serrated profile dependencies of the surface tension and the wetting contact angle of the water phase on the emulsifier concentration in its composition. It was assumed that in the manufacturer's recommended range of emulsifier content for the bitumen emulsion, there are inefficient concentrations, the effect and variability of which is manifested by contact with mineral materials of different nature.

To confirm or refute the suggested hypothesis, there were chosen a number of concentrations of emulsifiers in the water phase with a minimum surface tension for industrial tests. This factor should lead to the formation of effective adsorption layers stabilizing the bitumen emulsion.

The composition and properties of water phases prepared in industrial conditions are shown in Table 1.

Table 1. Compositions and properties of the water phase

Emulsifier type							
REDICOTE E-11				REDICOTE EM-44			
No of composition	content, %		PH of the water phase	No of composition	content, %		PH of the water phase
	emulsifier	acid			emulsifier	acid	
1	0.15	0.07	2.21	9	0.10	0.13	2.08
2	0.20	0.07	2.19	10	0.15	0.16	2.08
3	0.25	0.08	2.14	11	0.25	0.20	2.05
4	0.60	0.10	2.04	12	0.30	0.21	2.07
5	0.80	0.08	2.16	13	0.40	0.48	2.05
6	1.01	0.08	2.15	14	0.50	0.60	2.00
7	1.30	0.08	2.10	15	0.60	0.69	1.90
8	1.51	0.09	2.19	16	0.80	0.91	1.93

Note: Compositions No. 1–12 are prepared using hydrochloric acid (HCl), formulations No. 13–16 using orthophosphoric acid (H_3PO_4)

To obtain stable bitumen emulsions, the pH of the water phase should be in the range of 1.5–2.5. The achievement of the specified parameters was carried out by varying the acid: orthophosphoric and hydrochloric one, depending on the emulsion obtained (slow-breaking or quick-breaking emulsion).

Surface tension is the determining factor of many manufacturing processes, including the emulsification of bitumen. Thus, at very low values of the surface tension at the interface of two phases, spontaneous emulsification of the system is possible [14].

According to the generally accepted opinion [5, 11, 14, 15], if the system is heterogeneous, the interface necessarily exists between the phases. And although the "interface surface" is noted most often, in reality it is a definite transition layer of finite thickness. With the passage of time, after the phases come in contact, there is a mutual diffusion of the molecules. As a result, the equilibrium distribution of the components in the volumes of the contacting phases and at the interface is gradually achieved [15].

Two types of the emulsifier were considered for the preparation of quick- and medium- (EM-44), as well as slow-breaking (E-11) emulsions. Disintegration is a controlled emulsion breaking, resulting from a disruption of the structure of the emulsifier adsorption layers or with a decrease in their stabilizing ability. Thus, due to the difference in the mechanism of the effect of the emulsifier on the emulsion breakdown rate, the intensity of mutual diffusion of the water phase components and the disruption of the structure of the adsorption layers will change when the surface-active substance is changed, leading to changes in the surface tension of the system.

In accordance with the data of Table. 1, the surface tension dynamics was studied as a function of the emulsifier concentration and the lifetime of the water phase solution Figure 2.

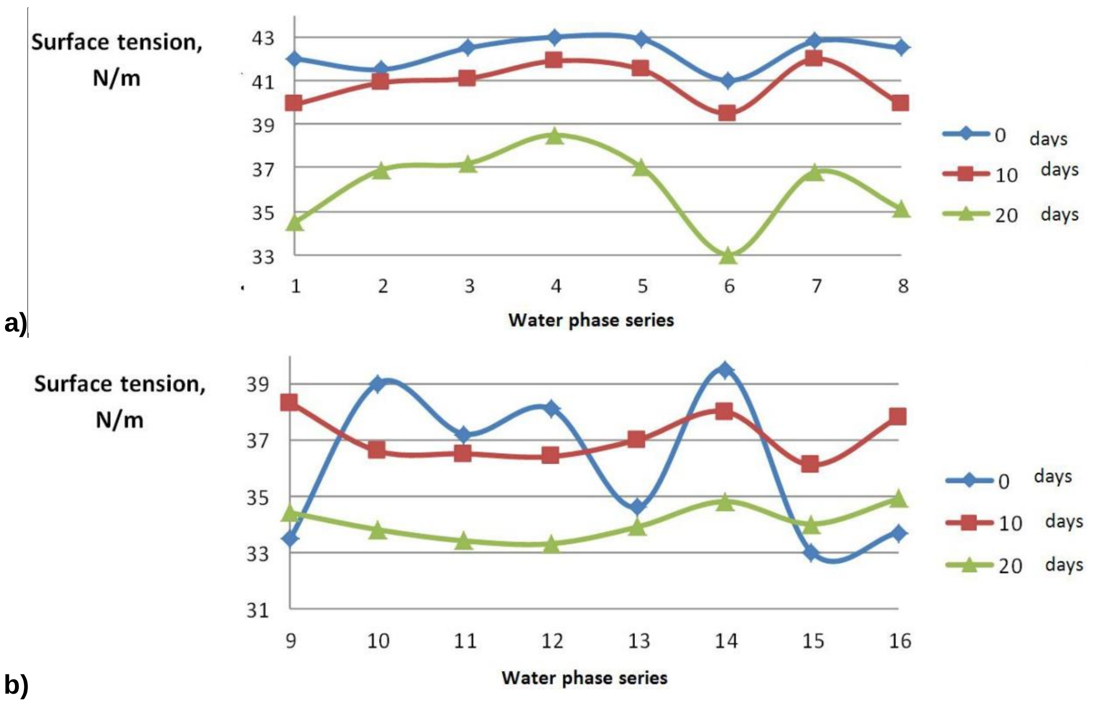
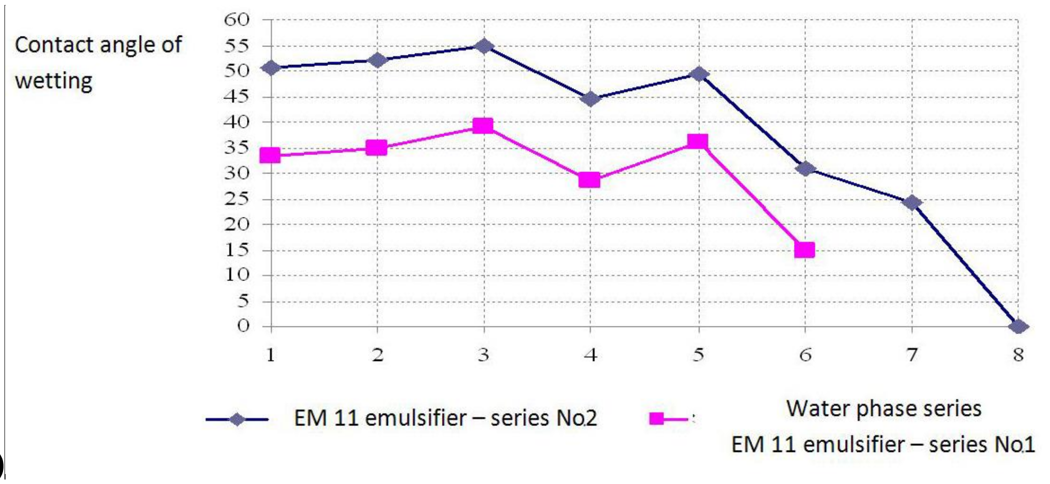


Figure 2. Surface tension of the water phase prepared on the emulsifier
a) REDICOTE E-11, b) REDICOTE EM-44

The exposure time was 0 days, which corresponds to the test of the freshly prepared water phase, and also 10 and 20 days. To eliminate the effect of moisture and evaporation, tubes with ground glass stoppers were stored in a dark place at a constant temperature.

As you can see at the Figure 2 the dynamics of the water phase surface tension in time on the basis of REDICOTE E-11 and REDICOTE EM-44 emulsifiers is opposite. So, with the passage of time, the effectiveness of the REDICOTE E-11 emulsifier in the water phase increases which can be judged by the reduction of its surface tension and the detection of a clear extremum in Figure 2a. According to the data obtained, the most effective composition is water phase series No. 6 with an emulsifier concentration of 1.01 %. At the same time, regardless of the exposure time of the solution, the nature of the surface tension dependencies on the emulsifier concentration (compositions No. 1–8), Figure 2a, varies insignificantly, which indicates that the selection of stable slow-breaking emulsions is possible already at an early stage of the selection of the water phase. When considering the system "water-emulsifier REDICOTE EM-44" Figure 2b, the surface tension of the water phase having an initially sinusoidal nature of the variation of the dependencies on the emulsifier concentration over time takes a smoothed out form. Obviously, this effect can be explained from the point of view of the functionality of emulsifiers. In connection with this, when selecting the compositions of bitumen emulsions, it is necessary to take into account that the stabilizing capacity of emulsifiers in the water phase for quick-breaking emulsions is short, while the "viability" and efficiency of the "water-emulsifier" system for slow-breaking emulsions grows in time, which confirms the dependencies established earlier [13].

When investigating the spreading ability of the water phases under study, depending on the concentration and type of the emulsifier on the surface of mineral materials, interesting dependences were recorded (Figure 1), which were used to assess the possibility of early prediction of the compatibility of the emulsifier used to prepare the bitumen emulsion and mineral material which will subsequently interact with the production emulsified binder. Therefore, to study the wetting contact angle, as before, substrates were used: white marble and red granite. To verify the reproducibility and reliability of the experiment, substrates were selected from different batches of materials, it is displayed as two lines in Figures 3–4. It is obvious that the substitution of the substrate affected the changes in the wetting contact angle. However, the general pattern within one type of stone material was preserved.



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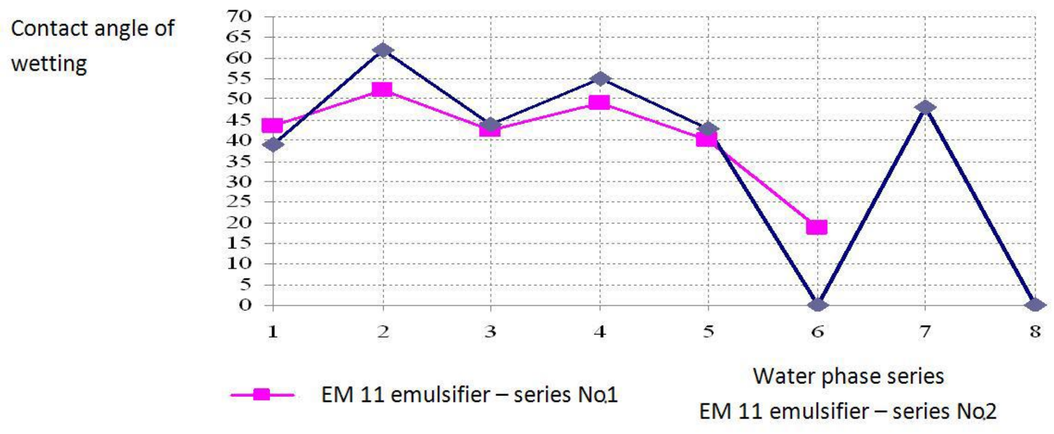
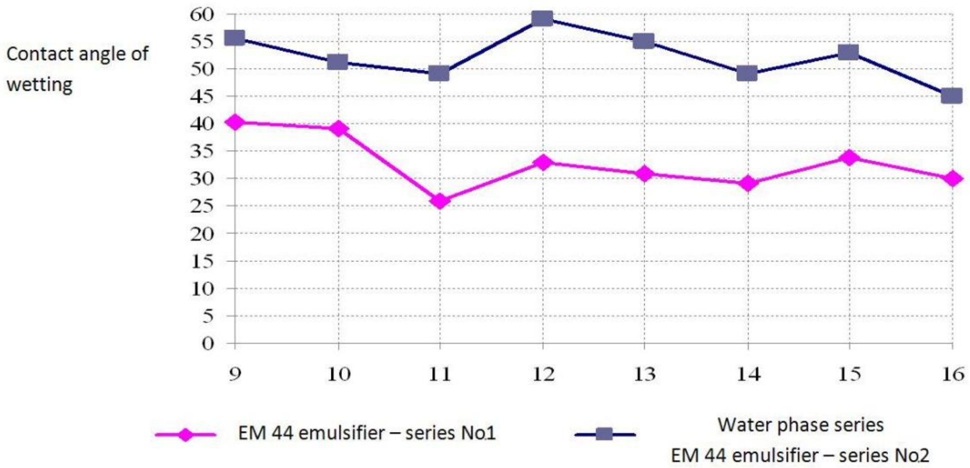


b)

Figure 3. Dynamics of the contact angle of wetting with a water phase prepared on REDICOTE E-11, a mineral substrate of: a) white marble, b) granite

In the process of setting up the experiment, data were obtained confirming the provisions of the developed hypothesis, as described in previous works [13, 16].

In the general case, upon contact of different phases on the interface, a monolayer is formed. Upon contact of the water phase with the mineral substrate, the monomolecular layer is formed due to adsorption of the surface-active substances from the solutions under study. With an increase in the emulsifier concentration in the water phase, the monomolecular layer can transform into a polymolecular surface layer [9], as a result of which the properties of the mineral surface vary significantly. And it can be accompanied by the transformation of mineral substrates from hydrophilic to hydrophobic ones in case of contact with various emulsifiers, which subsequently leads to hetero-alescence of bitumen particles from bitumen emulsions on the mineral surface [7]. This phenomenon is based on the selective adsorption of the emulsifier in points of contact with the mineral surface from the water phase. In the system "mineral substrate – cationic emulsifier" adsorption occurs due to physical processes, such as electrostatic attraction and bonding on the surface of the hydrocarbon chain of the emulsifier. Electrostatic forces are created due to the attraction of the positively charged polar groups of the emulsifier ions and the negatively charged point of the mineral material ions when the critical concentration is exceeded, which depends on the nature of the stone substrate and the pH of the medium [7]. These connections can be of various types. Electrostatic characteristics of the mineral surface and properties of the water phase are the most significant parameters that initiate adsorption processes of the emulsifier in the system.



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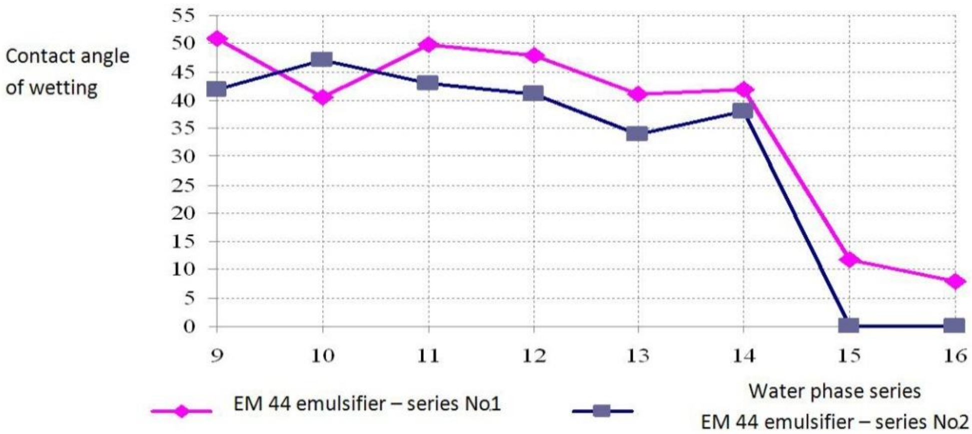


b)

Figure 4. Dynamics of the contact angle of wetting with a water phase prepared on REDICOTE EM-44, a mineral substrate of: a) white marble, b) granite

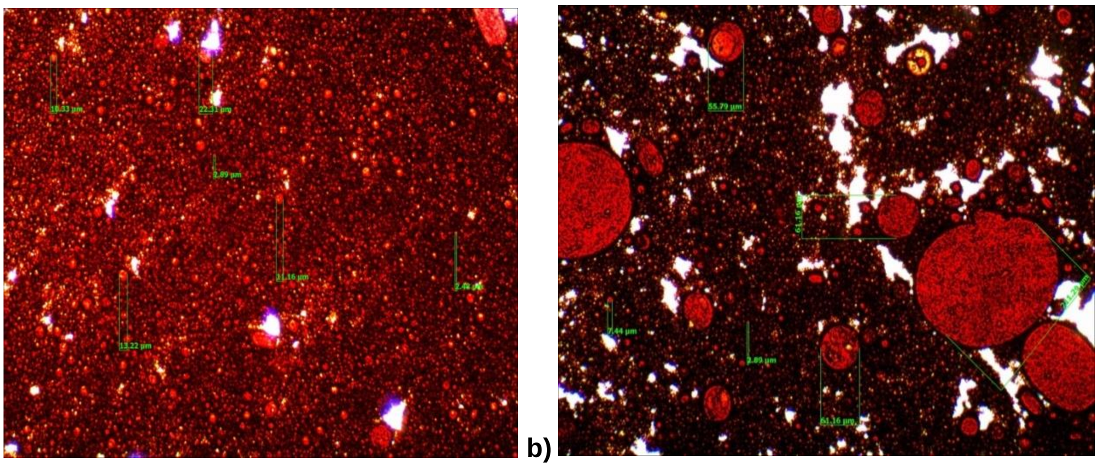
Also, it is necessary to note a high correlation of the results on the surface tension of the water phase prepared on the REDICOTE E-11 emulsifier, with the data obtained in studying the wetting contact angle on granite. And the similarity of trend lines in Figures 2a and 3a rises as the exposure time of the water phase increases. What is natural, in view of the fact that the emulsifier under consideration relates to the cationic one according to the mechanism of action and is intended for efficient work when combined with mineral materials of acidic nature, including granite.

The most significant differences in the dependences of the wetting contact angle on the concentration of the emulsifier, Figure 4, obtained on various mineral substrates, are observed at high concentrations of the emulsifier (0.6–0.8 %). In this case, upon contact with the granite substrate, a full spread of the droplet of the water phase occurs. Which is more than understandable, since, in accordance with the manufacturer's recommendations [4, 9], the concentration range for the preparation of the medium-breaking emulsion is 0.25–0.6 %.

Analysis and comparison of the obtained data showed that in accordance with the proposed method for prediction of the properties of bitumen emulsion compositions, the graphs show the most effective concentrations corresponding to the series of water phases on the emulsifier REDICOTE E-11 – No. 6 and No. 8, on REDICOTE EM-44 – No. 9, 11 and 13, and also No. 15 and No. 16. Such a choice is justified by the low interfacial tension at the interface of the phases of the "water solution- surface-active substances" system, which predetermines the high dispersity of the emulsion prepared on its basis, and the small values of the wetting contact angle of the mineral substrate surface show high compatibility of the system "water phase – stone material". Thus, it can be assumed that bitumen emulsions prepared using the indicated series of water phases will be kinetically stable, with the presence of adsorption layers of the emulsifier that inhibit the flocculation and coalescence of emulsion droplets and will be characterized by the relative one-dimensionality of the droplets of disperse systems [17–20].

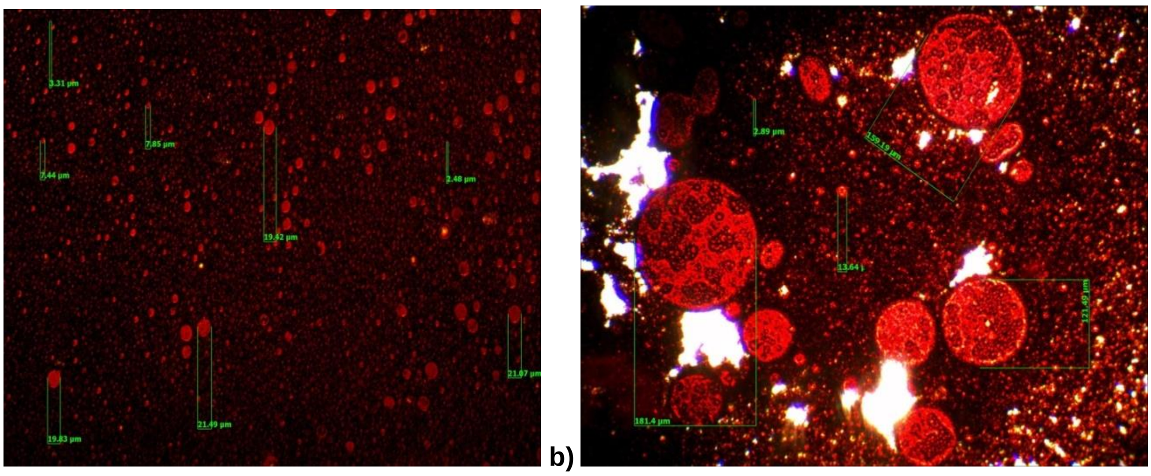
One of the main methods for studying the structural features and properties of the bitumen emulsion is optical microscopy [18], which allows one to obtain an image of particles of emulsified bitumen without overlapping one another.

In this connection, at the next stage of the experiment, in order to study the structural features under production conditions, experimental batches of bitumen emulsions were prepared and samples of emulsified binder were selected, the surface-active substance content of which, according to the performed studies (Figures 2–4), was effective (compositions No. 6 and 13) and ineffective (compositions No. 7 and 14). The choice of compositions is justified by a lower content of emulsifier, which determines the economic effectiveness of the studied compositions. The evaluation was carried out using the MICMED-1 microscope, using the transmitted-light brightfield method, the essence of which is as follows: in the absence of the specimen, a light beam from the condenser passing through the lens gives a uniformly illuminated field near the focal plane of the eyepiece, in the presence of an absorptive element in the specimen, partial absorption and partial dispersion of light shone upon it occurs, which makes it possible to obtain an image of the emulsified bitumen in the volume. The transfer of the image to the computer monitor and the preservation of the pictures were carried out by a special camera for the DCM 310 microscope (with a resolution of 3 MPix). The samples were examined under a microscope at the magnification factor of 400. The obtained photos were processed and saved using the image editor "AxioVision". The results of the study are shown in Figures 5, 6.



a) b)

Figure 5. Microphotography of the structure of the bitumen emulsion on the REDICOTE E-11 emulsifier with the concentration: a) 1.01 % series No. 6, b) 1.30% series No. 7



a) b)

Figure 6. Microphotography of the structure of bitumen emulsion on the REDICOTE EM-44 emulsifier with concentration: a) 0.40 % series No. 13, b) 0.50 % series No. 14

As can be seen, the emulsions, Figures 5a and 6a, prepared on effective water phases from series No. 6 and No. 13, are characterized by homogeneity of the particle size distribution in geometrically correct shape, which will have a positive effect on their stability. The emulsions, Figures 5b and 6b, prepared on solutions with a high content of emulsifier, which, according to preliminary tests of water phases (Figures 2–4), were attributed to ineffective, are characterized by a different scale of structure, a coalescence effect is observed, which is evidence of low efficiency of the adsorption layers of the emulsifier. As a result, this system becomes unstable.

To ensure the production of an effective bitumen emulsion, it must be characterized by structural stability, that is, by property retention in time, but at the same time it must break upon contact with the surface of mineral materials at a certain rate due to technological requirements, providing strong adhesion-cohesive bonds to the surface to be applied.

Structural stability and consistency of bitumen emulsions were studied by the nature of the change in their properties for seven days (Table 2). The emulsion breakdown rate upon contact with the mineral material was evaluated by determining the breakdown index. This method, performed in accordance with Russian State Standard GOST R 55420-2013 (EN 13808: 2011), allows one to study the degree of the emulsion breakdown and consists in determining the maximum amount of the disperse quartz filler of a strictly defined granulometric and mineralogical composition that is mixed with the standard amount of the emulsion. The larger the value of this index is, the slower the breakdown proceeds. Table 2 presents data on the determination of the stability and breakdown index of bitumen emulsions.

Table 2. Stability and breakdown index of bitumen emulsions

Emulsifier type							
REDICOTE E-11				REDICOTE EM-44			
No of composition	When stored after 7 days		Breakdown index*	No of composition	When stored after 7 days		Breakdown index *
	Storage stability according to screening residue N 014,%	Segregation resistance, method A, %			Storage stability according to screening residue N 014,%	Segregation resistance, method A, %	
GOST R 55420-2013	EBDK M			GOST R 55420-2013	EBDK B		
	0.30	5	>80		0.30	5	20-50
6	0.20	3	82.3	13	0.20	2	48.30
7	0.31	4	84.2	14	0.32	5	64.70

* The breakdown index was determined using powder quartz

Analyzing the obtained results, it can be seen that compositions No. 7 (slow-breaking) and composition No. 14 (quick-breaking) cationic bitumen emulsions are characterized by unsatisfactory structural stability, which agrees with the preliminary results on the study of water phases (Figures 2–4).

Thus, as a result of the conducted study, an algorithm was developed for the method of early prediction of the efficiency of cationic bitumen emulsions, which consists in studying the change in surface tension as a function of the concentration of the emulsifier at the initial time of the "life" of the water phase solution, in connection with the established constancy of the character of changes in the studied factors in the time of exposure of the solution. And in the subsequent study of the wetting contact angle of the investigated water phases of the surface of mineral materials of different nature depending on the concentration and type of the emulsifier. This allows us to select the optimal ratio of the components of the water phase already at the early stage of preparation of bitumen emulsions and to predict the compatibility and activity of the interaction of the emulsified binder with the mineral substrate, to rank the mineral material by the efficiency of the interaction with the bitumen emulsion, which provides for obtaining improved and stable composite indicators. It makes the procedure of early prediction particularly relevant. Because at the present moment [5, 6], when developing the formulation of bitumen emulsions and determining the optimum content of surface-active substances in them, test laboratory batches of emulsified binders with different concentrations of surface-active substances are prepared, which is a long, energy and resource intensive process, and it also does not take into account the specifics of a particular type of aggregate, which will be in contact with the emulsion, and therefore, to evaluate the performance of the composite, it is necessary to perform a number of additional tests to assess the compatibility of bitumen emulsions with the mineral aggregate.

4. Conclusions

1. Thus, it has been established and confirmed that the proposed method for studying the dynamics of changes in the properties of the water phase and the interface of the "water phase-mineral substrate" with a change in the surface-active substance concentration allows for the determination of optimal concentrations of various emulsifiers at the early stage for the preparation of effective cationic bitumen emulsions. In the work, at the stage of preparation of the water phase, optimum concentrations of emulsifier were selected, composition No. 6 (REDICOTE E-11 – 1.01 %) and No. 13 (REDICOTE EM-44 – 0.40 %), the use of which allowed obtaining effective bituminous emulsions characterized by structural stability.

2. It has been found that, under certain optimal concentrations of emulsifiers, a higher degree of structuredness of the prepared bitumen emulsion is observed, it is expressed in a more homogeneous and uniform distribution of particles throughout the volume and the possibility of regulating the production breakdown rate, which indicates the sedimentative and aggregative stability of the emulsion. The ability to predict the effectiveness of cationic bitumen emulsions at the stage of the water phase preparation can significantly reduce the time and material funds in the process of their production, while improving the quality of the final product, which is an actual and promising direction for ensuring the development of the road construction industry.

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