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Intrinsic self-healing potential of asphalt concrete

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Abstract. In the work, the influence of temperature, the time of its exposure, as well as the structural properties of bitumen and the test conditions for asphalt concrete on its intrinsic potential for self-healing was studied. Asphalt concrete samples from two types of bitumen with different group compositions were produced and tested under uniaxial compression and split. The intrinsic self-healing potential of asphalt concrete increases with an increase in temperature and exposure time. The greatest effect of temperature on the intrinsic self-healing potential corresponds to values close to the bitumen softening temperature. Temperature is a factor of double action: if there is a sufficient amount of maltene fraction in the bituminous matrix of asphalt concrete, it improves healing; in the absence of the maltene fraction, the high temperature is a condition for the aging of the binder. Aromatic compounds among maltenes are of greater importance in self-healing. The dimensions, condition of the samples, and features of the formation of defects are factors that affect intrinsic self-healing.

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1. Introduction

Asphalt concretes are operated under variable mechanical and thermal loads, due to which cracks occur in the material causing premature failure of pavements [1]. Functional modifiers in asphalt mixes are used to improve the performance of asphalt concrete. Ordinary additives improve the ability of asphalt concrete to resist dynamic impacts [2], delaying the onset of cracking. There is a new type of additives that provide self-healing of asphalt concrete, eliminate some of the defects at the stage of operation and preserve the functionality of the material. A lot of research [4–6] is aimed at developing new solutions that allow creating effective self-healing asphalt concrete.

The problem in the development of such asphalt concretes is the thermoplastic nature of the binder and the technological features of the production of the material. Since the technology of using thermoplastic bitumen as part of asphalt concrete mixture includes the stage of heating to a temperature above 100 °C when mixing the components, the use of microorganisms as a healing agent is impossible. Also, the process of compacting the asphalt mixture can prematurely destroy the capsules of the healing agent. Therefore, the technology for using an encapsulated healing agent must take these factors into account.

After cooling of the asphalt concrete mixture, the bitumen hardens, and the elastic properties prevail in the asphalt concrete. The type of defects in asphalt concrete depends on the conditions in which it is operated. At high temperatures, plastic deformations occur, and at low temperatures, cracks form due to brittleness. Cracks occur in the asphalt concrete matrix or at the binder-aggregate interface. Defects are formed at the microscale level at the initial stage, and then they combine into macrocracks [7].

Intrinsic self-healing potential is an important aspect to consider when developing self-healing methods for asphalt concrete. The intrinsic self-healing potential is the ability of a material to restore the state of the structure, due to the nature and characteristics of the substance of which it is composed. For asphalt concrete, this ability is determined by the thermoplastic nature of the bituminous binder. Therefore, when developing an effective solution for self-healing asphalt concrete, it must be taken into account that partial restoration of the state of the structure may be due to the bitumen's intrinsic self-healing potential.

Analysis of scientific studies [4–17] shows that the effectiveness of asphalt concrete self-healing depends on controllable factors, which are regulated by prescription parameters, and on uncontrollable factors, which are determined by the operating conditions of the pavement (Fig. 1).

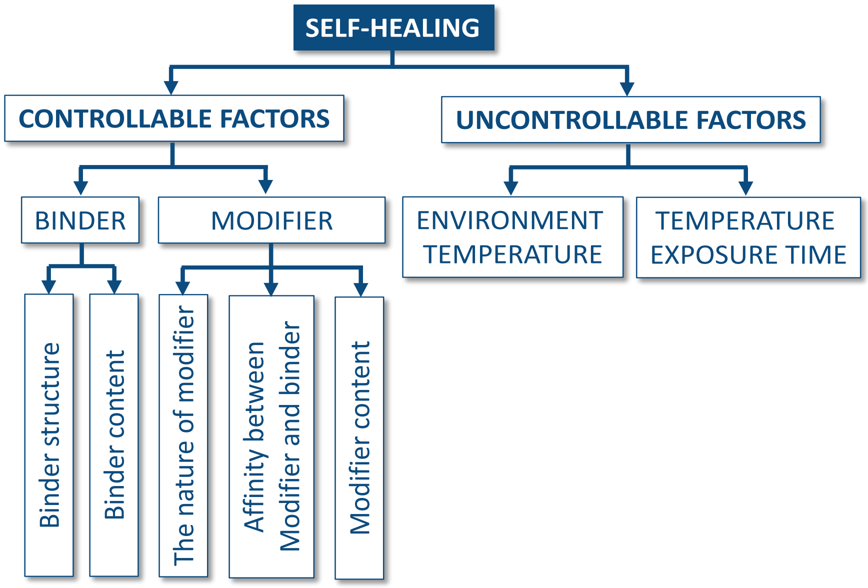

Figure 1. Decomposition of factors affecting the self-healing of asphalt concrete.

Recipe and technological parameters of asphalt concrete production, which are controlled at the stage of designing the composition of the asphalt concrete mixture, are controllable factors [5, 7]. Controlling the self-healing ability of asphalt concrete is achieved through two main components: bitumen and special modifiers [6–11].

The ability to independently eliminate defects in the structure is determined by the thermoplastic properties of bitumen and is provided by spontaneous entanglement of the molecule [8]. Also, the intergrowth of molecules in bitumen occurs at the molecular level due to reversible hydrogen bonding [9] with the formation of new crosslinks and chains [10, 11]. These processes determine intrinsic self-healing potential for bitumen in asphalt concrete, which depends on the binder content in the mixture and its group composition.

The use of various kinds of modifiers to intensify self-healing is a solution that determines its effectiveness [12–16]. In this case, the nature of the modifier used and its degree of affinity with bitumen will affect the efficiency of self-healing and the mechanism of this process. For example, an encapsulated rejuvenator is able to increase bitumen intrinsic potential self-healing by changing its group composition [12, 13]. And the encapsulated polymer modifier acts as a gluing agent, forming new structural bonds [14, 15].

Environment temperature and its exposure time are uncontrollable factors that affect the self-healing of asphalt concrete. The influence of temperature is due to the thermoplastic properties of bitumen in asphalt concrete, when the viscoelastic flow contributes to the closure of microcracks and prevents their growth. In this case, the intensity of this process depends on the intermolecular distance and the rate of thermal motion of molecules, which naturally increases with increasing temperature rise.

An important condition for the occurrence of self-healing during operation at high temperatures is the absence of significant plastic deformations. With temperature rise, the kinetic energy of the rotational and vibrational motion of the molecules of the binder increases, which leads to an increase in the distance between the molecules. The consequence of this is an increase in the volume of the material and a decrease in the viscosity of the thermoplastic material [16, 17]. In this case, a contact can occur in the defect (crack) zone if its surfaces are sufficiently close, which, as a result, contributes to their spontaneous coalescence.

It should be noted that when special additives are used in the composition of asphalt concrete, including components sensitive to healing effects, the properties and nature of these components will be an additional factor influencing the ability of self-healing [3, 18, 19]. Therefore, studies to establish the degree of influence of temperature, the time of its exposure, the composition of bitumen on the intrinsic self-healing potential of asphalt concrete, taking into account the type of testing of samples, are relevant.

In this article, the goal is to study the influence of these factors (Fig. 1) on the intrinsic self-healing potential of asphalt concrete, to achieve which the tasks of studying the influence of the temperature of the medium and the time of its action on asphalt concrete samples during the healing period after the strength test were solved. The problem of assessing the influence of the composition of bitumen and test methods on the intrinsic self-healing potential of asphalt concrete was also solved.

2. Materials and Methods

Mechanical behavior of self-healing stone mastic asphalt (SMA) mixtures is examined experimentally. The materials and tests methods used in this study are summarized in sections 2.1 and 2.2 respectively.

2.1. Materials

Bitumens BND 60/90 (Russian State Standard GOST 22245-90 Viscous petroleum road bitumen. Specifications) with a softening point of 51 °C (BND(1)) and 60 °C (BND(2)) (ASTM D36/D36M-20, EN 1427:2015) are used as a binder for asphalt concrete. BND(2) bitumen is obtained from BND(1) by aging for 8 hours at 160 °C. The basis of the binder aging technique complies with the test conditions in accordance with the Russian State Standard GOST 18180-72 Petroleum bitumen. Method for determination of change of mass after heating (EN 12607-2). The group composition of bitumen (referred to as SARA) is presented in Table 1.

Table 1. Bitumen composition SARA.

Bitumen	SARA, %			
	Saturates	Aromatics	Resins	Asphaltenes
BND (1)	7.1	38.5	32.2	22.2
BND (2)	6.5	32.9	38.5	22.1

Note: Method for Determining SARA described in the section 2.2

The SMA mixtures were prepared following the aggregate gradation presented in Table 2. The binder content is 7 % over the mineral part of the mixture, resulting in an air void content of 3 %. 0.3 % by weight of the mixture of cellulose fibers Viatop-66 is used as a stabilizing additive to prevent bitumen drainage.

Table 2. Sieve grain analysis (particle size distribution).

Parameter	Value								
Sieve size, mm	15	10	5	2.5	1.25	0.63	0.315	0.16	0.071
Passing, %	92.3	58.8	33.0	21.7	18.4	16.5	14.7	12.7	10.6

SMA cylindrical specimens with a height and diameter of 71.4 mm were manufactured, by placing the required mass of the mixture into a mold (inner height is 160 mm and inner diameter are 71.4 mm) and compacting it in two stages (Russian State Standard GOST 12801-98). The form, heated to 90...100 °C, is filled with an asphalt concrete mixture, installed on a vibrating platform, firmly fixed on it with a special device. The mixture in the mold is vibrated for 3.0 ± 0.1 min at a frequency of 2900 ± 100 min^{-1}, an amplitude of 0.40 ± 0.05 mm and a vertical load on the mixture of 30 ± 5 kPa, which is transferred to the mixture with the help of a load hung on the form. After vibrating, the mold with the sample is removed from the vibrating platform, placed on the press plate for additional compaction under pressure 20.0 ± 0.5 MPa and maintained at this pressure for 3 minutes. Then the load is removed and the sample is removed from the mold with a squeeze tool.

The main properties of SMA are summarized in Table 3.

Table 3. Main properties of SMA with different bitumen.

Parameter	Method	Unit	Standard limit	Value for bitumen	
				BND(1)	BND(2)
Average density	Russian State Standard GOST 12801-98	g/cm^3	–	2.43	2.44
Air void		%	1.5…4.5	3.0	3.0
Water saturation		%	1.0…4.0	1.9	1.2
Compressive strength at 20 °C		MPa	> 2.2	3.3	3.2
Compressive strength at 50 °C		MPa	> 0.65	1.1	0.9
Coefficient of internal friction		–	> 0.93	0.93	0.97
Shear bond at 50 °C		MPa	> 0.18	0.56	0.57
Tensile strength at 0 °C		MPa	2.5…6.0	2.6	2.5
Water resistance (long exposure)		–	> 0.85	0.92	0.90

2.2. Test methods

SARA was determined by thin layer chromatography with a flame ionization detector and with Chromarod TM type SIII quartz rods in accordance with IP 469 using an Iatroscan Mark V analyzer.

Determination of the main properties of SMA was carried out in accordance with the methods specified in Russian State Standard GOST 12801-98 Materials on the basis of organic binders for road and airfield construction. Test methods.

The effect of temperature and the structure of the binder on the self-healing of SMA were studied on cylinder samples, which were tested under two loading schemes to determine the strength: in compression and splitting.

Cylinder samples (diameter – 71.4 mm, height – 71.4 mm) were used to determine the compressive strength. Compression was carried out after thermostating the samples at a temperature of 20 °C. Thermostating of the samples was carried out in a climatic chamber for 4 hours. The compression rate corresponded to a press plate movement of 3 mm/min. During loading, the maximum load that the sample could withstand was recorded. Compression strength was calculated using the formula:

$$R_c = \frac{P}{S} \cdot 10^{-2}, \quad (1)$$

where P is maximum compression load, N; S is load distribution area, cm^2, 10^{-2} is conversion factor to MPa.

Samples-half-cylinders with a radius and thickness of 35.7 mm were sawn from standard samples-cylinders to determine the ultimate strength in splitting. Splitting was carried out after thermostating the samples at a temperature of –20 °C. Thermostating of the samples was carried out in a climatic chamber for 4 hours. The loading rate corresponded to the movement of the press plate of 3 mm/min. During loading, the maximum load that the sample could withstand was recorded. Split strength was calculated using the formula:

$$R_b = \frac{P}{2RT}, \quad (2)$$

where P is maximum breaking load, N; T is half-cylinder sample width, cm, R is half-cylinder sample radius, cm.

After the destruction of the semi-cylinder samples, the two parts were combined, pressing the fracture surfaces to each other with rubber bands.

For healing, the samples were placed in a drying chamber. The duration of recovery and the temperature of the medium were set in accordance with the two-factor experimental plan.

Two-factor composite experimental design was implemented. Temperature (X_1, °C) and exposure time (X_2, day) are selected as predictors. It was also proved during the research that polynomial regression model [20]:

$$Y = B_0 + B_1 X_1 + B_2 X_2 + B_{12} X_1 X_2 + B_{11} X_1^2 + B_{22} X_2^2, \quad (3)$$

is suitable (hypothesis test for adequacy by means of F -criterion) as a description.

Zero levels $\left(X_{0i}\right)$ and half-variations $\left(l_i\right)$ for experimental designs are summarized: X_{01} = 45 °C; X_{02} = 7 days; l_1 = 15 °C; l_2 = 3 days.

After the healing period, the specimens were re-thermostated at 20 °C or –20 °C and tested in compression or splitting, respectively.

3. Results and Discussion

Currently, there is no single approach to assess the effect of self-healing in materials. In most cases, the calculation of the relative change in strength after a rest period is used. However, this approach can not show the ability of the material to heal for various loading schemes and test conditions.

In this work, the residual strength approach was used to evaluate the effect of intrinsic self-healing potential $\left(HP\right)$.

$$HP = \frac{R_h - R_1}{R_0}, \tag{4}$$

where R_h is strength after healing, MPa; R_0 is strength before healing, MPa; R_1 is residual strength, MPa. Residual strength was measured by repeated testing on samples after determining R_0.

The results of determining the strength indicators of asphalt concrete under various loading schemes and various healing conditions are presented in Table 4.

Table 4. SMA strength for various test schemes.

#	X_1, °C	X_2, day	Strength, MPa											
			SMA based BND(1)						SMA based BND(2)					
			Compression			Splitting			Compression			Splitting		
			R_0	R_1	R_h	R_0	R_1	R_h	R_0	R_1	R_h	R_0	R_1	R_h
1	30	4			1.64			0.05			1.91			0.34
2	60	4			2.12			0.41			1.97			0.87
3	30	10			1.82			0.21			1.98			0.37
4	60	10			2.11			0.65			1.88			0.29
5	23.8	7	3.36	1.32	1.60	1.95	–	0.01	3.17	1.85	1.90	1.74	–	0.05
6	66.2	7			2.03			0.32			1.94			1.24
7	45	2.75			1.73			0.06			2.02			0.29
8	45	11.24			1.75			0.31			1.92			0.48
9	45	7			1.81			0.23			1.95			0.54

Strength indicates a greater fragility of asphalt concrete based on BND(2) bitumen, which is due to a lower content of aromatic compounds. This is explained by the fact that BND(2) contains less maltenes, including aromatic compounds that reduce hardness and increase fluidity. And resins, which are viscosity and hardness, are contained in BND (2) more [21]. Residual strength is absent for samples tested in splitting. At the same time, the residual compressive strength is 58 % of the strength of asphalt concrete based on BND(2) and 40 % of the strength of asphalt concrete based on BND(1). This is probably due to the influence of the group composition of bitumen on the deformative properties of asphalt concrete. Asphalt concrete based BND(1), where the concentration of paraffin-naphthenic and aromatic compounds is higher, is prone to large deformations, therefore, after a compression test, deformation and displacement of structural elements occur, which contributes to the formation of a less stable structure [22]. These factors must be taken into account when choosing a method for determining the ability of self-healing.

The healing characteristics were calculated using the strength, on the basis of which the regression equations were obtained, reflecting the dependence of the intrinsic self-healing potential on the temperature and time of its exposure during the rest period.

For asphalt concrete based on BND(1), the equations are:

$$HP_{C1} = 14.7 + 5.09X_1 + 0.76X_2 - 1.35X_1X_2 + 1.17X_1^2 + 0.09X_2^2, \tag{5}$$

$$HP_{B1} = 12.0 + 7.95X_1 + 4.86X_2 + 1.05X_1X_2 + 0.12X_1^2 + 0.79X_2^2 \quad (6)$$

and for asphalt concrete based on BND(2) bitumen, the equations are:

$$HP_{C2} = 3.2 + 0.05X_1 - 0.66X_2 - 1.23X_1X_2 - 0.55X_1^2 - 0.23X_2^2, \quad (7)$$

$$HP_{B2} = 30.7 + 15.27X_1 + 2.07X_2 - 8.72X_1X_2 + 2.39X_1^2 - 5.06X_2^2, \quad (8)$$

where HP_{Ci} and HP_{Bi} are the intrinsic self-healing potential of specimens during compression and splitting tests, respectively.

The dependencies under study are graphically presented in Fig. 2 for asphalt concrete based on BND(1) and in fig. 3 for asphalt concrete based on BND(2).

a) b)

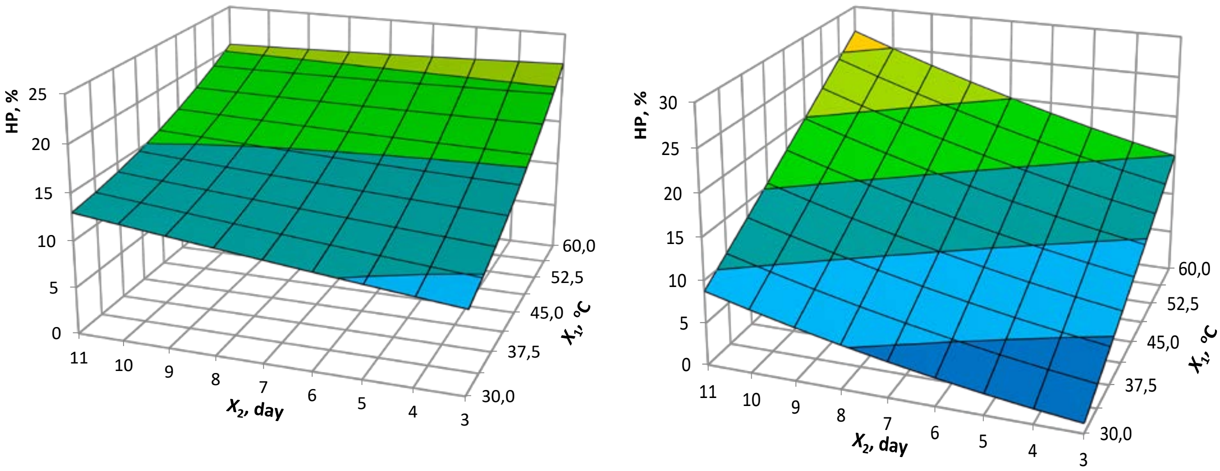

Figure 2. For asphalt concrete based on BND(1) graphical representation of equations: a – compression test (5); b – split test (6).

a) b)

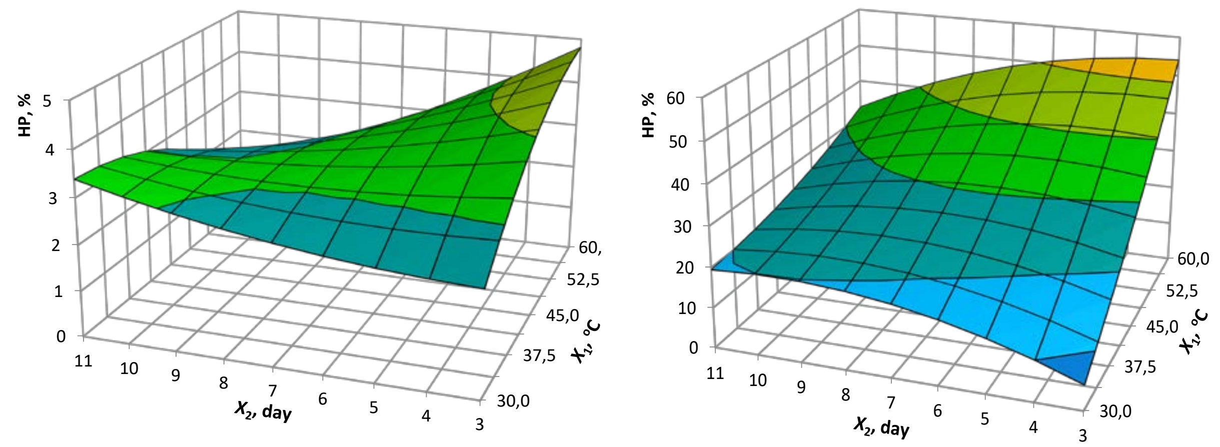

Figure 3. For asphalt concrete based on BND(2) graphical representation of equations: a – compression test (7); b – split test (8).

An analysis of the obtained mathematical models, which describe changes in the intrinsic self-healing potential with temperature and time of its action, shows that the studied factors have a different effect on samples made on the basis of bitumen with different group composition and tested under different conditions.

The temperature factor has a positive effect on the intrinsic self-healing potential of asphalt concrete: with an increase in temperature to the bitumen softening point, an increase in strength after self-healing is observed. This effect is due to a change in the rheological properties of the matrix, a decrease in the viscosity of bitumen, and an increase in the mobility of molecules. In such a state, the probability increases that the molecules spontaneously entangle with each other and grow together through the restoration of specific bonds [9, 23]. The intensity of this process depends on the intermolecular distance and the rate of thermal motion of molecules, which naturally increases with increasing temperature. In this case, a contact that promotes their spontaneous entangle can occur when the surfaces of the defect (crack) are sufficiently close. Consideration of a defect with the involvement of the Laplace pressure is a theoretical justification for the implementation of the self-healing mechanism. In this case, the defect is represented as two surfaces, between which there is a gas phase. The Laplace pressure arises on each surface, which is directed into the internal volume of the phase that forms the surface of the defect. This pressure prevents spontaneous self-healing.

It should be noted that temperature is a factor of double action. Temperature can be a factor that improves recovery, and can negatively affect the ability to self-heal. If there is a sufficient amount of maltene fraction in the bituminous matrix of asphalt concrete (Fig. 2), the temperature has a positive effect on the healing process. But with its deficiency, temperature is a condition for the aging of the binder and does not contribute to the process of spontaneous entangle of molecules (Fig. 3). The intrinsic self-healing potential for asphalt concrete based on BND(2) is significantly less than for asphalt concrete based on BND(1).

The double action effect of the influence of temperature is obvious with an increase in the time of its exposure. The time factor for asphalt concrete based on BND(1) is positive, however, at elevated temperatures, its positive effect decreases. Under conditions of lower content of light fractions in bitumen, the temperature exposure time is a negative factor influencing the intrinsic self-healing potential. In this case, the maximum values of the intrinsic self-healing potential of asphalt concrete correspond to the boundary of the studied factor space – exposure for 3 days. This is due to the aging of the binder and a decrease in the maltene part of bitumen, in which asphaltene-resin complexes are dissolved, which contributes to the deterioration of rheological properties and a decrease in the mobility of molecules.

It should be noted that the ability of bitumen to self-heal is a sensitive property, because a decrease in the content of paraffin-naphthenic and aromatic compounds by 13.5 % leads to a significant change in the degree of influence of temperature and the time of its exposure. Taking into account the differences in the group compositions of the bitumen used (Table 1), it can be concluded that a higher content of light fractions has a positive effect on intrinsic self-healing potential. When choosing bitumen and designing an asphalt concrete mixture, it is necessary to take into account the physical and chemical processes of the interaction of the binder with mineral components, as a result of which the fractions are redistributed, and the amount of free bitumen will also affect self-healing.

Intrinsic self-healing potential differs significantly when testing asphalt concrete samples using different loading schemes and testing temperatures. The test temperature affects the state of asphalt concrete at the time of loading. When tested under conditions of negative temperature, elastic properties prevail in asphalt concrete, and the sample does not deform during loading, but brittle breaks into two parts with a clear contour of the defect surfaces. When the sample is compressed at 20 °C, the work at failure is partially spent on deformation and the elastoplastic properties appear. At the same time, samples after compression are characterized by residual strength, since the maximum fixed load does not destroy all structural bonds in asphalt concrete. The main crack across the sample is formed during splitting, so there is no residual strength. Thus, the testing scheme affects the type of defects in the samples and creates different initial conditions for further self-healing.

It should be noted that when testing samples of asphalt concrete for splitting, the intrinsic self-healing potential is greater than when testing under compression for each type of bitumen. Since the determination of the splitting strength was carried out at a temperature of –20 °C, the asphalt concrete sample was brittle, which ensured good contact of the fracture planes during self-healing (Fig. 4).

a) b)

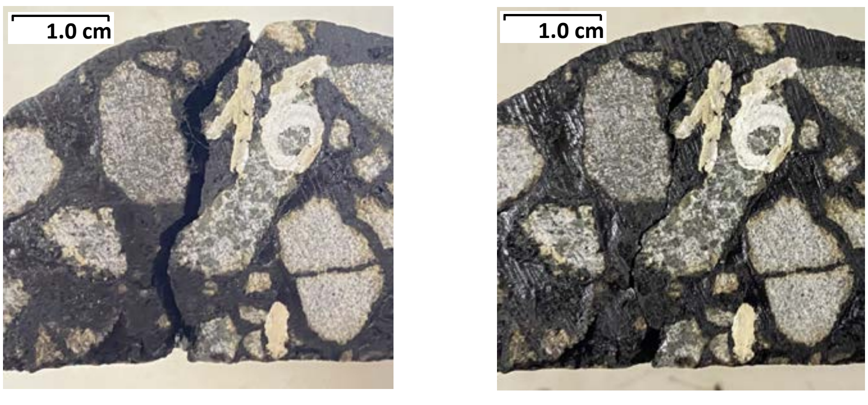

Figure 4. Asphalt concrete sample before (a) and after (b) self-healing.

Pictures of asphalt concrete samples show that defects have angular edges. The surfaces of the defect are easily joined to each other during the healing process, which will contribute to better self-healing. After healing, the defect on the front surface of the sample remains visible to the naked eye, but the strength is high.

The reason for the increased values of intrinsic self-healing potential are two main aspects. First of all, this is the brittle state of the material and the nature of the defect formed during splitting. When the sample is destroyed, the formed surfaces of the main crack have a similar relief without significant deformations, which contributes to their good joining during the rest period. Secondly, the gap between the surfaces of the defect contributes to their heating during thermostating, and during heating in the volume of the sample, a certain temperature gradient is formed. Thus, the dimensions of the tested samples, the state of the samples during testing, the features of the formation of defects are factors that affect self-healing. These factors should be taken into account when developing new ways to implement self-healing technology and a unified method for measuring self-healing indicators.

The group composition of bitumen is used to calculate the Colloidal Instability Index (CII), which is the ratio of the sum of the concentrations of asphaltenes and saturates to the sum of the concentrations of resins and aromatic compounds:

$$CII = \frac{A_s + S}{R + A_r}, \tag{9}$$

where: A_s is the weight percent of asphaltenes; S is the weight percent of saturates; R is the weight percent of resins; and A_r is the weight percent of aromatics.

This index characterizes the peptizing ability and its lower values indicate a greater stability of the bitumen structure. It was noted in [24] that lower CII values correspond to higher self-healing rates, because molecules with a less branched structure are more mobile. For the studied bitumens, the CII values differ insignificantly, for BND(1) it is 0.41, and for BND(2) it is 0.40. Thus, it is aromatic compounds that are dominant in the issue of bitumen self-healing among maltenes. This may be due to the sequence of transition of individual fractions of bitumen into the melt with increasing temperature: aromatic compounds have a lower softening point, so their molecules are more involved in free thermal motion than resin molecules.

Also, the amphoteric nature of bitumen and aromaticity affect the formation of chains in the structure and the ability to interact in several places [25]. According to [25], an increase in the acid-base surface energy and a decrease in the Lifshitz-van der Waals surface energy occur in bitumen with a low content of amphoteric substances and a high content of aromatic compounds. This confirms the importance of the influence of a higher concentration of aromatic compounds on diffusion, molecular mobility and, consequently, self-healing [26–28].

It should be noted that this article does not take into account the influence of the ratio of bulk and structured bitumen in the volume of asphalt concrete. However, it is obvious that the interaction of mineral components with bitumen will contribute to the redistribution of binder fractions [29]. The intensity of these physical and chemical processes will also affect the ability of self-healing. Therefore, this issue is relevant for further research in the field of self-healing asphalt concrete.

4. Conclusions

Temperature is a factor of double action: if there is a sufficient amount of maltene fraction in the bituminous matrix of asphalt concrete, it has a positive effect on healing; with a lack of maltene fraction, temperature serves as a condition for the aging of the binder and does not contribute to self-healing. The ability of bitumen to self-heal is a sensitive property, because a decrease in the content of paraffin-naphthenic and aromatic compounds by 13.5 % leads to a significant change in the degree of influence of temperature and the time of exposure to it. The maximum recovery of strength after thermal exposure at a temperature of 60 °C is 27 % and 55 % for asphalt concrete based on BND(1) and BND(2) bitumen, respectively.

The greatest effect of temperature on the intrinsic self-healing potential corresponds to values close to the softening point of the applied bitumen. However, under such conditions, asphalt concrete loses its bearing capacity, deforms excessively and cannot be used. Therefore, using the ability of bitumen molecules to spontaneously entangle as a mechanism for self-healing is difficult.

For the studied bitumens, the Colloidal Instability Index values differ insignificantly, for BND(1) it is 0.41, and for BND(2) it is 0.40. Aromatic compounds have a dominant role in the self-healing of bitumen among maltenes: aromatic compounds have a lower softening point, so their molecules are more involved in free thermal movement than resin molecules.

The dimensions of the tested samples, the state of the samples during testing, the features of the formation of defects are factors that affect self-healing. These factors should be taken into account when developing new ways to implement self-healing technology and a unified method for measuring self-healing indicators.

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