

Self-healing intensity, rate and durability of asphalt concrete

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Abstract. When developing self-healing materials, methods to test self-healing are needed. Three parameters for assessing the self-healing of asphalt concrete are proposed in this article. The self-healing coefficient is proposed to assess the intensity of self-healing. It is proposed to use the dependence of changes in relative strength over time to estimate the rate of self-healing. The time to reaching the critical value of the strength, is proposed to assess the durability of asphalt concrete after healing. The generalized quality criterion is proposed to assess the effectiveness of self-healing. The efficiency of self-healing using encapsulated ARP is 1.87 times greater than when using encapsulated SfO. The self-healing rate for SMA with encapsulated SfO is 71% greater than ordinary asphalt concrete, and for SMA with encapsulated ARP it is 75% greater. The failure rate for SMA with encapsulated SfO is 5% greater than ordinary asphalt concrete, and for SMA with encapsulated ARP it is 70% greater. The use of encapsulated SfO makes it possible to get SMA with a self-healing ability that is 36% greater than ordinary SMA. The use of encapsulated ARP makes it possible to get SMA with a self-healing ability that is 79% greater than ordinary SMA. The self-healing ability of asphalt concrete with encapsulated SfO is 36% greater than ordinary asphalt concrete. The self-healing ability of asphalt concrete with encapsulated ARP is 79% greater than ordinary asphalt concrete. Requirements for new methods for testing the self-healing ability of asphalt concrete are formulated.

1 Introduction

Increasing the durability of asphalt concrete is the main task for material development in the field of pavement. Encapsulated modifiers are a new component with the help of which the self-healing effect of the material is realized [1...3]. It is important to synthesize capsules that are resistant to the temperature at which the asphalt concrete mixture is prepared. The shell material of the capsules must ensure their integrity during the process of compacting the asphalt concrete mixture, preventing premature release of the modifier [4, 5].

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The integrity of the asphalt concrete structure is disrupted and cracks form under the influence of operational factors. This process leads to the destruction of the capsules, which facilitates the release of the modifier. The modifier is released from the capsules, partially fills the defect, wets the edges of the crack and diffuses into the matrix. Partial restoration of the integrity of asphalt concrete and its ability to resist mechanical stress is the result of the process of secondary structure formation. Thus, the quality of self-healing asphalt concrete is determined both by the parameters of the initial structure of the material and by parameters reflecting the effectiveness of the healing process.

The main quality indicators of asphalt concrete, including self-healing asphalt concrete, are indicators reflecting structural properties and operational properties. Average density, air voids and water saturation are indicators characterizing the structural properties of asphalt concrete. Strength, shear stability, water resistance and climate resistance are indicators characterizing the performance properties of asphalt concrete [6, 7]. Typically, the requirements for these indicators are established in the standards for asphalt concrete [8, 9]. For self-healing asphalt concrete, the quality criterion is the self-healing properties. Self-healing intensity (degree of self-healing), self-healing rate, self-healing timelines (start and end self-healing) and durability of the structure after self-healing are main indicators characterizing the self-healing properties [10].

There are no general approaches to assessing self-healing properties, so the authors of various works use their own methods [11, 12]. More often, methods are used that allow assessing only the intensity of self-healing [13]. This work proposes methods that make it possible to quantify healing process, which makes it possible to compare the effectiveness of encapsulated modifiers with different mechanisms of action.

2 Materials and methods

Organic compounds such as industrial or vegetable oils, rejuvenates, polymers or mixtures thereof are traditionally used as a healing agent. The mechanism of action of such healing agents is different and depends on its nature and solubility in bitumen. The healing agents were selected according to [14]. Two healing agents (HA) were investigated in this work. As healing agents for self-healing, conventional sunflower oil (SfO) and a thiol-containing urethane AR-polymer (ARP) were used. Dynamic viscosity at 25°C of SfO is 0.05 Pa·s, acid value – 0.015...0.035 mg KOH/g.

AR-polymer is a thiol-containing urethane polymer with terminal mercaptan groups (SH-), produced by «PolyMix Kazan» LLC by TU 2226-001-90014974-11. Dynamic viscosity at 25°C of ARP is 9.7 Pa·s, SH-group content – 1.5...2.5.

The hardener for the polymerization of AR-polymer is an activator consisting of technical sulfur, manganese oxide (IV) and tetramethyl thiuram disulfide, mixed in a ratio of 6.1: 3.7: 1.0. An activator was used to increase adhesive strength after restoration.

The SMA-15 mixtures were prepared following the optimal aggregate gradation, the binder content is 7 %, resulting in air void content of 3 %. 0.3 % by weight of cellulose fibers Viatop-66 is used as a stabilizing additive to prevent segregation.

BND 60/90 bitumen (State standard 22245-90) is used as a binder for SMA. Bitumen penetration at temperature 0 and 25 °C are 36 and 67 0.1 mm. Bitumen softening point is 52 °C.

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 and compacting it in two stages. First, vibration compaction has been applied for 3 minutes, followed by hydraulic press compaction.

Self-healing is a structure formation process that can be assessed by intensity and speed. Also, important is the durability of the structure, which is formed after self-healing.

2.1 Self-healing intensity of asphalt concrete

Self-healing intensity was assessed by two indicators: the healing index and the strength loss coefficient.

The healing index was determined by the compressive strength at 20 °C. The asphalt concrete testing procedure included the following stages: samples were thermostated in a climate chamber; compressed with a press until destruction; 7 days of rest. This test was repeated 4 times.

The second indicator for assessing self-healing intensity was the self-healing coefficient, which takes into account the relative difference in the loss of strength of asphalt concrete with and without the use of an encapsulated modifier. The strength loss index is the reciprocal of the recovery index, which will be calculated as $1-R_h/R_0$. The self-healing coefficient was calculated using the formula:

$$k_h = \frac{IR'}{IR} \cdot 100\%, \quad (1)$$

where $IR'=1-R'_h/R'_0$ is the strength loss index of asphalt concrete without capsules; $IR=1-R_h/R_0$ is the strength loss index of asphalt concrete with encapsulated modifier; R'_0 and R_0 are the strength of asphalt concrete before restoration without capsules and with an encapsulated modifier, respectively, MPa; R'_h and R_h are strength indicators of asphalt concrete after restoration without capsules and with an encapsulated modifier, respectively, MPa.

2.2 Durability of asphalt concrete after self-healing

The cylinder specimens were repeatedly tested in compression to evaluate the time to reach the critical value to establish the durability of the asphalt concrete after self-healing. The obtained values of compressive strength in section 2.1 were used to establish the kinetics of changes in this indicator and regression analysis.

The obtained values of compressive strength were used to establish the kinetics of changes in this indicator and regression analysis. The obtained empirical coefficients of the regression mathematical model equations $f(t) = b_1x + b_0$ were used to estimate the time to reach the critical value R_{cr} , which was accepted in accordance with the State standard (GOST 31015-2002). The durability of the asphalt concrete structure after self-healing was assessed using the failure rate, which was calculated:

$$k_F = \frac{(b'_0 - R_{kp}) \cdot b_1}{b'_1 \cdot (b_0 - R_{kp})}, \quad (2)$$

where R_{cr} is the critical value of the structure-sensitive parameter (compressive strength), assumed to be 2.2 MPa; b_1 and b'_1 are coefficients of the regression equation $f(x) = b_1x + b_0$, describing the decrease in strength for asphalt concrete without capsules and with an encapsulated modifier, respectively.

2.3 Self-healing rate of asphalt concrete

Cylinder samples were manufactured using standard technology to evaluate the self-healing rate of asphalt concrete. Asphalt concrete samples were tested under compression at 20 °C, after which they were rested at 20 °C. The rest time was not the same for the samples: part of samples the rest was 1 day, the second part was 3 days, and the third part was 7 days. After rest, the samples were re-tested for compressive strength. The obtained compressive strength values were used to establish the kinetics of changes in the relative strength R_i/R_0 and regression analysis. The obtained empirical coefficients a in the regression mathematical model $f(t) = a \cdot R_i/R_0 + b$ and $f(t) = a \cdot \ln(R_i/R_0) + b$, reflecting the rate of change $f(t)$, were used to estimate the relative rate of self-healing.

3 Results and discussion

The study of self-healing properties was carried out on crushed stone-mastic asphalt concrete SMA-15, which was modified using encapsulated modifiers. The main properties of ordinary SMA-15 are presented in Figure 1.

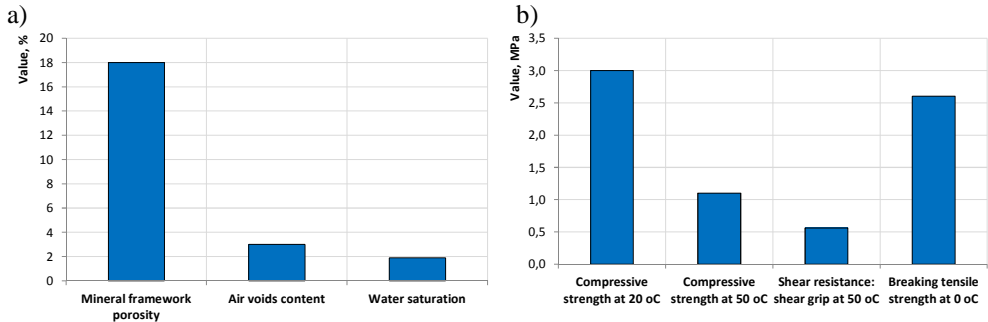


Fig. 1. The main properties of SMA-15: structural (a) and strength (b) parameters

The self-healing technology, which is implemented using an encapsulated modifier, is aimed at initiating a secondary process of structure formation during operation. Self-healing is caused by the formation of new structural bonds in the material, resulting in the restoration of the balance of structural bonds and restoration of the ability of the material structure to function in the road structure.

Using an enough amount of encapsulated modifier will restore a greater number of structural bonds than due to the binder's own restorative potential. To study the restoration effect for asphalt concrete, encapsulated sunflower oil and encapsulated AR polymer were used. Together with the encapsulated AR polymer, an activator of 3.5% by weight of bitumen was used, which was added to the asphalt concrete mixture.

3.1 Determination of self-healing intensity of asphalt concrete

The results of determining the tensile strength during 4 successive cycles of compression tests and recovery periods of asphalt concrete with different contents of encapsulated modifier are presented in Figure 2.

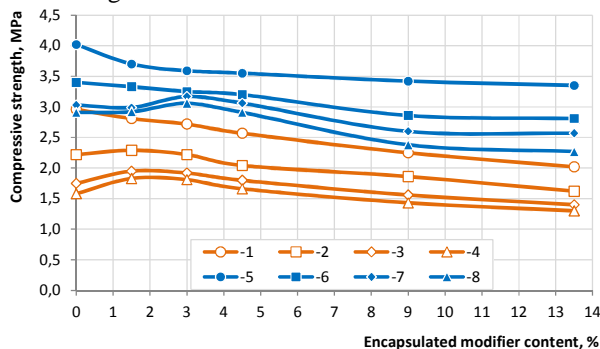


Fig. 2. Compressive strength of asphalt concrete with encapsulated SfO: 1 – first test; 2 – second test; 3 – third test; 4 – fourth test; and encapsulated ARP: 5 – first test; 6 – second test; 7 – third test; 8 – fourth test

Partial restoration of the number of structural bonds, which ensure the state of the structure to resist greater loads than before the restoration process, is the reason for

restoration when using an encapsulated modifier. Strength restoration occurs more with the use of encapsulated SfO than with asphalt concrete without capsules. This occurs as a result of the release of SfO from the capsules, their diffusion into the matrix, dissolution, reducing fragility and increasing the mobility of binder molecules. As a result of the spontaneous entanglement of long chains of molecules, new bonds are formed and the ability to resist stress increases.

The use of encapsulated ARP provides greater strength recovery than asphalt concrete without capsules. After the destruction of the capsules, the ARP polymerizes and glues the edges of the crack. As the number of capsules increases, healing increases as the amount of adhesive agent increases. But a large number of capsules negatively affects healing, because the capsules themselves do not have high strength. The results of calculating the self-healing parameters are presented in Figure 3.

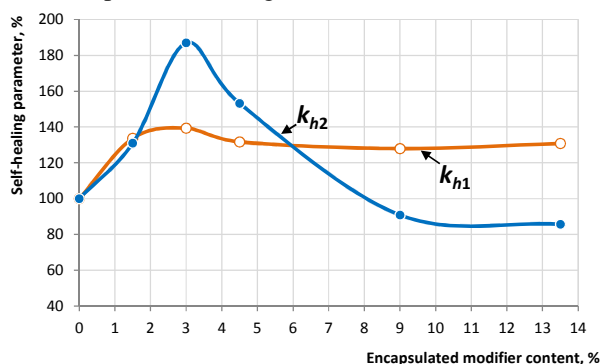


Fig. 3. Dependence of self-healing parameters after 4 compression tests on the content of capsules for asphalt concrete: k_{h1} – with encapsulated SfO; k_{h2} – with encapsulated ARP

Analysis of Figure 3 shows that the maximum healing effect k_h is observed at a concentration of encapsulated SfO and encapsulated ARP equal to 3% by weight of bitumen. This value is the optimal content in the asphalt concrete structure, at which the negative effect from the presence of capsules in the volume of the material is minimal, and the restorative effect is maximum. The total strength loss, taking into account the effect of the modifier, turned out to be 28% less than the optimal content of encapsulated SfO. The total strength loss, taking into account the effect of the modifier, is 46% less for asphalt concrete with the optimal content of encapsulated ARP. The efficiency of self-healing using encapsulated ARP is 1.87 times greater than when using encapsulated SfO.

Thus, the optimal content of the encapsulated modifier, providing the maximum restorative effect of asphalt concrete, is 3%. Further studies of self-healing asphalt concrete were carried out using compositions with an optimal content of encapsulated modifier.

3.2 Determination of durability of asphalt concrete after self-healing

Obviously, to assess the self-healing ability of asphalt concrete, it is necessary to take into account additional property indicators that characterize the effectiveness of this process. For this purpose, the work carried out a study of the self-healing rate.

It is natural to assume [15, 16] that the presence of self-healing ability for asphalt concrete will help to increase the period when the material meets the requirements. To determine the durability of asphalt concrete after self-healing, cylinder samples were subjected to repeated compression tests to estimate the time to reach the critical strength value. Asphalt concrete samples were tested for compression, after which the samples were stored for 7 days at a temperature of 20 °C, then subjected to compression again.

Compression testing of the samples was repeated 3 times every 7 days. The change in strength is presented in Figure 4.

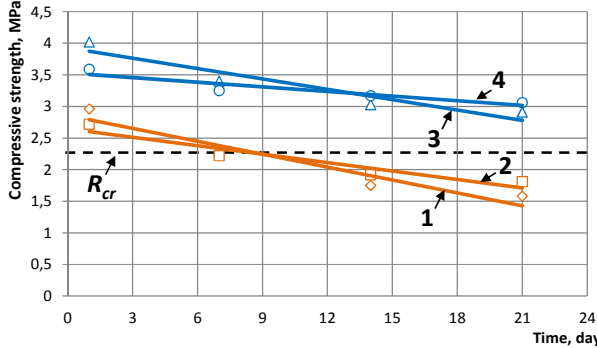


Fig. 4. Change in strength during repeated compression tests of SMA-15: 1 – ordinary; 2 – with encapsulated SfO; 3 – with activator; 4 – with activator and encapsulated ARP

Empirical coefficients of the regression equations, which reflect the time to reaching the critical value of the structure-sensitive parameter (strength), were used to calculate the failure rate (formula 2). The critical value of compressive strength is taken to be 2.2 MPa for SMA-15. The coefficient values are presented in Table 1.

Table 1. Calculation of failure rate

№	Material	Modifier	Empirical coefficients		k_F
			b_0	b_1	
1	SMA-15	–	2.86	-0.0861	–
2		Encapsulated SfO	2.65	-0.0447	1.05
3	SMA-15	–	3.93	-0.0546	–
4		Encapsulated ARP	3.53	-0.0246	1.70

The results of the Failure Rate calculation show that for asphalt concrete samples with capsules with AR polymer, due to self-healing processes, the failure period is longer. The failure rate for SMA-15 composition 4 is 5.6 times greater than ordinary composition 1 and 1.7 times greater than composition 3. These results indicate that the developed asphalt concrete with encapsulated AR polymer is more resistant under repeated mechanical loads due to self-healing processes.

3.3 Determination of self-healing rate of asphalt concrete

To assess the rate of the self-healing process in asphalt concrete, the kinetics of changes in compressive strength were studied. The results of determining the kinetics of changes in compressive strength are presented in Figure 5.

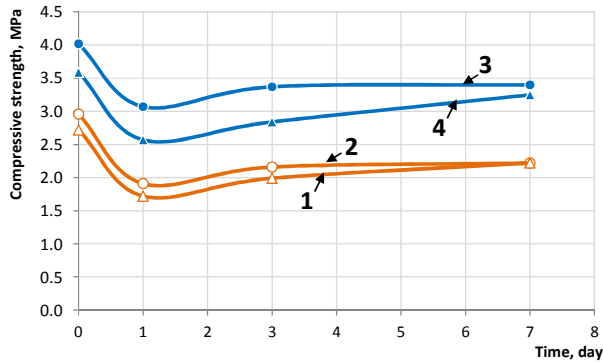


Fig. 5. Kinetics of changes in compressive strength of SMA-15: 1 – ordinary; 2 – with encapsulated SfO; 3 – with activator; 4 – with activator and encapsulated ARP

Self-healing processes occur within 7 days both in the control samples SMA-15 and with the encapsulated modifier. This is explained by the Intrinsic self-healing potential of asphalt concrete due to the thermoplastic properties of the binder, which was proven in [17], as well as due to the encapsulated modifier. To assess the self-healing rate, the kinetics of changes in the relative strength R_t/R_0 was assessed (here R_t is the strength after self-healing, R_0 is the initial strength).

Analysis of the obtained dependencies shows that within 7 days, there is an increase in the relative strength index, which indicates an increase in the self-healing effect over time. This process is typical for both asphalt concrete samples without an encapsulated modifier and with capsules in the composition. The use of an encapsulated modifier allows for faster self-healing, which proves its positive effect on restoring the ability to resist mechanical stress. To quantify the rate of self-healing and the influence of the encapsulated modifier on this process, regression analysis was performed (Table 2).

Table 2. Parameters of regression equations for changes in relative strength

№	Material	Encapsulated modifier	R_t/R_0 after day			Equation Parameter Values					
						Linear			Logarithmic		
			1	3	7	a	b	R^2	a	b	R^2
1	SMA-15	SfO	0.65	0.73	0.75	0.016	0.651	0.74	0.055	0.653	0.93
2		–	0.63	0.73	0.82	0.029	0.619	0.95	0.094	0.631	0.99
3	SMA-15 with activator	ARP	0.76	0.84	0.85	0.012	0.772	0.65	0.043	0.772	0.87
4		–	0.72	0.79	0.91	0.031	0.690	0.99	0.096	0.707	0.96

In the work, the analysis of dependencies was carried out using linear and logarithmic regression equations, for which coefficient a characterizes the rate of change of R_t/R_0 . The obtained values of coefficients a show that the encapsulated modifier affects the rate of self-healing. Analyzing linear regression, the self-healing rate for SMA with encapsulated SfO is 81% greater than for ordinary asphalt concrete, and for SMA with encapsulated ARP it is 93% greater. Analyzing logarithmic regression, the self-healing rate for SMA with encapsulated SfO is 71% greater than for ordinary asphalt concrete, and for SMA with encapsulated ARP it is 75% greater. The results obtained show that the rate of self-healing of asphalt concrete with encapsulated ARP is higher than that of asphalt concrete with encapsulated SfO.

The obtained values of self-healing intensity, durability after self-healing, self-healing rate can be used to select an encapsulated modifier with greater efficiency in the

development of self-healing asphalt concrete. The effectiveness of the self-healing ability of asphalt concrete with an encapsulated modifier was assessed using a generalized criterion using a function of the form:

$$F_{SH} = \sqrt[3]{k_h k_F k_R}, \quad (3)$$

where k_h is self-healing intensity; k_F is durability after self-healing; k_R is self-healing rate.

The calculation results are presented in Table 3.

Table 3. Values of criteria reflecting the self-healing of asphalt concrete

Parameter	Symbol	Value of parameter for SMA-15		
		Ordinary	With encapsulated SfO	With activator and encapsulated ARP
Self-healing intensity	k_h	1.00	1.40	1.93
Durability after self-healing	k_F	1.00	1.05	1.70
Self-healing rate	k_R	1.00	1.71	1.75
Self-healing efficiency	F_{SH}	1.00	1.36	1.79

The results of the F_{SH} calculation show that the use of encapsulated SfO makes it possible to obtain SMA with a self-healing ability that is 36 % greater than ordinary SMA. The use of encapsulated ARP makes it possible to obtain SMA with a self-healing ability that is 79 % greater than ordinary SMA. This proves the greater self-healing effect of asphalt concrete when using encapsulated AR-polymer than encapsulated sunflower oil.

4 Conclusion

Three parameters for assessing the self-healing of asphalt concrete are proposed in this article. The self-healing coefficient is proposed to be used to assess the intensity of self-healing. It is proposed to use the dependence of changes in relative strength over time to estimate the rate of self-healing. The failure rate, which reflects the time to reaching the critical value of the strength, is proposed to be used to assess the durability of asphalt concrete after healing. The generalized quality criterion is proposed to assess the effectiveness of self-healing. The proposed parameters allow a comprehensive assessment of the self-healing process [18...21]. This will allow you to establish the numerical value of the intensity and speed of self-healing and will allow you to determine the preferred technology.

The efficiency of self-healing using encapsulated ARP is 1.87 times greater than when using encapsulated SfO. The self-healing rate for SMA with encapsulated SfO is 71% greater than ordinary asphalt concrete, and for SMA with encapsulated ARP it is 75% greater. The failure rate for SMA with encapsulated SfO is 5% greater than ordinary asphalt concrete, and for SMA with encapsulated ARP it is 70% greater. The use of encapsulated SfO makes it possible to obtain SMA with a self-healing ability that is 36 % greater than ordinary SMA. The use of encapsulated ARP makes it possible to obtain SMA with a self-healing ability that is 79 % greater than ordinary SMA.

The proposed set of parameters, including self-healing intensity, durability after self-healing, self-healing rate, makes it possible to evaluate the effectiveness of self-healing when using various modifiers encapsulated.

The results obtained allow us to formulate requirements for new methods for testing the self-healing ability of materials with encapsulated modifiers:

- the method should consist of a procedure for the destruction of samples using external mechanical action, which destroys the capsules inside the material;

- the method should consist of a rest procedure, during which self-healing is realized;
- the kinetics of changes in parameters should be assessed by periodic tests;
- to assess the self-healing of a material, various structure-sensitive parameters (compressive strength, splitting strength, etc.) can be used;
- to assess the self-healing of a material, various test conditions can be used, under which different degrees of destruction are achieved (preliminary thermostating of samples at a temperature not higher than the softening point of the binder);
- to assess the self-healing of a material, various resting conditions for the material (time, temperature, humidity, etc.) can be used.

The healing index was determined by the compressive strength at 20 °C. The asphalt

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