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Influence of Adhesive Additives on the Properties of Bitumen and Asphalt Mixtures

ABSTRACT

This article presents the results of experimental studies of the influence of adhesive additives on the basic properties of modified bitumen and asphalt concrete mixtures based on it. The introduction of adhesive additives improves the basic properties of bitumen, which are necessary for obtaining high-quality asphalt concrete mixtures. Modification of bitumen increases adhesion to glass and crushed stone. Using methods of mathematical planning of experiments, experimental and statistical models of water saturation and compressive strength of asphalt concrete mixtures at temperatures of 20°C and 50°C were obtained. The introduction of modifying additives can significantly reduce the water saturation of asphalt concrete mixtures and increase strength at temperatures of 20 °C and 50 °C.

Keywords: bitumen, asphalt mixtures, strength, adhesive additives, penetration, softening point, ductility.

1. INTRODUCTION*¹*

One of the main tasks of modifying construction bitumen is to improve its adhesion ability to aggregates. The adhesion between mineral components and bitumen is an important criterion that affects the quality of the asphalt mixture, asphalt pavement characteristics and its resistance to damage. Nowadays, there is no generally accepted theory that satisfactorily explains the bonding process.

One of the earliest attempts that was proposed to explain the bonding process is the Mc Bain hypothesis [1], which considers this process as a mechanical "jamming" of the adhesive substance into the pores (or recesses) of the adhesive material. However, the positions put forward by Mc Bain were refuted in subsequent works [2, 3]. Later, considerations of the so-called "specific" adhesion appeared. Adhesion is generally understood as the stickness that occurs between two dissimilar materials brought

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into contact. In the case of stickiness connections, adhesion is the bond between the adhesive and the surface to which the adhesive adheres When considering adhesion phenomena, it is necessary to take into account cohesion, which is the stickiness within the bonded materials. Currently, the most important theories are the adsorption, electrical, diffusion, and chemical ones.

The adsorption theory of adhesion considers the formation of a bond between the adhesive and the bonded material (substrate) as a result of intermolecular forces. For the first time, the importance of adsorption phenomena for the adhesion process was pointed out [4] in 1926. However, the basics of the adsorption theory of adhesion were developed much later by Debroyne [5] and Mc Laren [6, 7].

According to Mc Laren, the formation of an adhesive layer occurs in two stages. At the first stage, the adhesive molecules move (migrate) to the surface of the substrate and the polar groups of the adhesive molecules approach the polar regions of the substrate, and at the second stage the sorption occurs. At a sufficiently close distance between the adhesive and substrate molecules, molecular forc-

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es (dispersion, induction, electrostatic) begin to act, leading to the formation of various bonds (dipole-dipole, dipole-induced dipole, hydrogen bond, etc.).

Being non-polar, bitumen has high water resistance, but mechanical impacts from vehicles accelerate the water penetration into the pores of asphalt concrete, as a result it leads to a decrease the asphalt pavement stability [8]. To reduce the negative effects of moisture, it is necessary to ensure the increased stickiness of the bituminous binder to the mineral components of the asphalt mixture.

Active adhesion is the ability of the binder to completely envelop the aggregate particles during the mixing of the mixture components. This adhesion occurs due to the mutual attraction of positively charged surfactants of bitumen molecules to negatively charged aggregate molecules. This type of interaction allows for the water displacement at the interface and ensures maximum coverage of the mineral material surface with bitumen [9, 10]. In contrast to active adhesion, passive adhesion occurs as a result of external forces, for example, due to increased pressure in the pores of asphalt concrete. Passive adhesion indicates the ability of bitumen to be bonded to the surface of the mineral material throughout the entire service life of asphalt concrete without the risk of destruction of these bonds under the influence of mechanical stress and water [11]. The loss of passive adhesion can cause premature pavement deterioration [12].

The literature describes the main theories that explain the concept of bitumen-aggregate interaction: chemical reaction, surface energy, molecular orientation, and mechanical adhesion [13, 14]. The adhesion between bitumen and aggregate depends on the surface tension of the bitumen, the chemical composition of the bitumen and aggregates, the viscosity of the bitumen, the porosity and purity of the aggregates, the moisture content of the aggregates, and the temperature during the preparation of the asphalt mixture (AM) [15, 16].

Robertson [18] argues that adhesion connecting bitumen and aggregates occurs between the poles of the bitumen and the polar surface of the aggregate. He also asserts that the polarity of the bitumen alone is not sufficient to achieve high adhesion because the bitumen is affected by external mechanical factors. It is assumed that the adhesion between bitumen and aggregate is provided by surface energy because the surface of the aggregate decreases together with the process of the bitumen adsorbtion [19]. The modification of bitumen by surfactant additives is based on the Dupre-Young equation, which relates the work of adhesion Waq to the surface energy of the solid:

$$
W_{aq} = V_{\tau} - V_{\tau} \quad (m + \cos \theta), \tag{1}
$$

where $\bm{{\mathsf{v}}}_{_{\mathsf{t}}}$ is the surface energy of a solid; $\bm{{\mathsf{v}}}_{_{\mathsf{t}}}^{\ast}$ is the free surface energy of a solid in an atmosphere of vapor and gases; $m = v_{w}^{*} / v_{w} > 1$ (v_{w}^{*} is the surface tension of the liquid oriented under the influence of the force field of the solid surface; v_{w} is the surface tension of the wetting liquid); $θ$ – is the edge angle of wetting.

It follows from equation (1) that to achieve high adhesive strength, it is important to ensure the necessary wettability of the aggregate with the binder and to reduce the interfacial surface energy, which is achieved by treating the aggregate with surfactants. The decrease in interfacial surface energy during creation of an adsorption-active environment is determined from equation (2).

$$
\Delta v_{T.w} = K T , \int_{0}^{c} n_s(c) dlnc
$$
 (2)

where $\Delta v_{\tau w}$ is the difference in interfacial surface energy without surfactants and in the presence of surfactants with a concentration of c ; $n_{\rm s}$ is the adsorption value determined by the number of surfactant molecules adsorbed per 1 cm² of the interface; ; K is the Boltzmann constant; T is the absolute temperature, °K.

The influence of the adsorption-active medium on the value of rises with an increase in aggregate dispersion and concentration, which is associated with an expansion in the interfacial surface and, accordingly, excess surface energy.

Mechanical adhesion depends on the physical and mechanical properties of the aggregates. An important condition for good adhesion is the wetting of the aggregate, which is necessary for contact between the materials and thus for the establishment of the basic physical and chemical forces that are ultimately responsible for adhesion. Aggregates are generally classified as hydrophobic (acidic aggregates) and hydrophilic (basic aggregates), which react differently to the adhesion process [20]. Authors [21] argue that acidic aggregates, in particular granite, which are most widely used for asphalt mixtures in Ukraine and other European countries, show a high loss of adhesion. In addition, the surface texture of aggregates affects the adhesion capacity, and rough surfaces with a larger contact area are desirable for its increase [14].

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Poor adhesion between the bitumen and aggregate leads to binder delamination in the presence of water, which eventually leads to potholes. Moisture damage to the asphalt pavement is considered the main cause of destruction to the asphalt pavement layers. The abrasion resistance of bitumen is also determined by its adhesion to the surface of the aggregate, it must be not only high but also stable over time, which is one of the conditions for the durability of the pavement.

Increasing the adhesion and ensuring the durability of asphalt pavements is possible by modifying bitumen with adhesive additives [22]. The introduction of appropriate additives into the asphalt concrete composition is important to ensure strong stickiness (adhesion) to aggregates [23]. These additives increase the inter-phase adhesion in wet conditions [22].

The purpose of this work is to obtain quantitative characteristics of the influence of the content of bitumen and adhesive additives and their interaction on the basic properties of modified bitumen and asphalt concrete mixtures based on it, which determine the durability of road surfaces.

2. MATERIALS AND METHODS OF RESEARCH

In our research, we used petroleum road bitumen PRB 70/100 that meets the requirements of EN 12591, the characteristics and standard values for which are given in Table 1.

Table 1. Test results of PRB 70/100

Granite crushed stone with fractions of 5-10 mm, 10- 20 mm, and 20-40 mm, bulk density 1420 kg/m 3 , as well as sand from granite crushing screenings with fractions of 0-5 mm were used, bulk density 1480 kg/m 3 .

The used as filler non-activated carbonate mineral powder maximum grain size 0.315 mm and bulk density 988 kg/m³ to DSTU B.V.2.7-121-2014 (Ukr.Standard).

The following adhesive additives were used for modification of bitumen:

- Wetfix BE, a cationic surfactant with increased thermal stability, manufactured by Nouryon (Sweden).

- Stardope 130 P, an additive based on vegetable oil phosphates for hot asphalt mixtures, manufactured by Star Asphalt (Italy).

For the obtained modified bitumen binders, standard quality indicators were determined – penetration at 25 °C (Fig. 1), softening point (Fig. 2) and extensibility (ductility) (Fig. 3). For asphalt mixtures were determined water saturation and compressive strength at 20 °C and 50 °C.

 Figure 1. Determination of the penetration depth

 Figure 2. Determination of softening point using the "Ring and Ball" device

Figure 3. Determination of the bitumen extensibility (ductility)

The method of the adhesion determination is based on the ability of the binder applied to the glass surface to resist peeling under the influence of water. The peeling resistance is determined by the area that remains covered with bitumen binder

Adhesion to glass (C), as ideal adhesive, is determined by the formula:

$$
C = \frac{Sp}{st} \times 100\tag{3}
$$

where, $S_p -$ area of the plate covered with bitumen, before testing, mm2

 S_r – area of the plate covered with bitumen, after testing, mm 2 .

Determination of adhesion to crushed stone (Fig. 4) is based on the ability of the binder applied to the surface of the crushed stone to resist peeling under the influence of water. The resistance to delamination is determined by the area that remains covered with bitumen binder.

The test results should not differ from each other by more than 0.5 points, otherwise the test should be repeated.

Determination of asphalt concrete samples' water saturation (Fig. 5) and compressive strength at 20 °C and 50 °C were performed according to DSTU B V.2.7-319:2016 (Ukr. Standard). The main idea test of the water saturation is in calculating the mass of water that the sample can absorb at a certain saturation mode. Water saturation (W) is determined by the formula:

where $\mathsf{m}_{_3}$ is the mass of the sample saturated with

$$
W = \frac{m_3 - m}{m_2 - m_1} \times 100. \tag{4}
$$

water and weighted in air, g; m is the mass of the

sample weighted in air, g; $m₂$ is the mass of the sample kept for 30 minutes in water, weighted in air, g; $\mathsf{m}_\text{\tiny{1}}$ is the mass of the sample kept for 30 minutes in water, weighed in water, g.

Figure 4. Determination of the adhesion to the crushed stone surface

 Figure 5. - Samples for determination of water saturation and compressive strength, at 20 °C and 50 °C (a),determination of water saturation (b)

The essence of the asphalt concrete strength test is to determine the compressive strength of laboratory specimens that have been pre-cured in a thermostat at 20 °C and 50 °C, respectively.

The compressive strength (R_{c} , MPa) is calculated by the formula:

$$
R_c = \frac{P}{F} \tag{5}
$$

where: P is the destructive load, N ;

F is the cross-sectional area of the sample, m^2 .

The methods of mathematical planning of exper-

iments have been used in research, which promote algorithmization of the experiments according to a certain scheme [24].

The most convenient way is to represent the unknown function as a polynomial:

$$
\hat{y} = b_0 + \sum_{i}^{k} b_i x_i + \sum_{i < j}^{k} b_{ij} x_i x_j + \sum_{i}^{k} b_{ii} x_i^2 + \dots,\tag{6}
$$

where b_i, b_{ii}, b_{ij} – regression coefficients; x_i, x_j - factors; k – number of factors.

The experiment has been planned in accordance with a typical matrix, which contains a set of combinations of factors varied relative to a certain origin or zero (basic) level. The permissible area of variation of the factors (factor space) was chosen based on a preliminary study of the object in accordance with the goal.

The results of the experiments were processed using mathematical statistics, obtaining quadratic regression equations for the required number of factors.

To assess the significance of the coefficients and the adequacy of the regression equations, statistical analysis was carried out using Fisher test and Student test.

3. EXPERIMENTAL RESULTS AND THEIR ANALYSIS

Experimental studies were conducted in two stages.

> Ω 10 20 30 40 50 60 70 80 90 without additive Additive content 0,3% content 0,4% content 0,5% Additive Additive *Bitumen adhesion, %* **PRB 70/100** ■ Wetfix BE ■Stardope 130 P

b)

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At the first stage, the performance of bitumen modified with adhesive additives Wetfix BE, Stardope 130 P was studied. The bitumen was modified using a laboratory mixer by mingling the bitumen with the additive for 15 minutes at a temperature of 145 ± 5°C, which fully ensured the uniform distribution of the adhesive additive. The obtained research results are shown in Table 2.

On the basis of the experimental data (Table 3), graphical dependences of the bitumen adhesion indicators with glass (Fig. 6 a) and crushed stone (Fig. 6 b) were constructed.

Table 2:Bitumen test results

The test results showed that with the addition of adhesive additives to the bitumen, the penetration of the modified bitumen compared to the original PRB 70/100 increases by 0.1 mm at a surfactant Stardope 130 P content of 0.5% in the bitumen, and by 0.2mm at an additive amount of 0.5%. At the same time, the effect of Wetfix BE surfactant is slightly higher.

According to the results obtained, the softening point and elongation of bitumen, which is different from the penetration, practically do not change in the studied range of adhesive additives consumption.

Comparing the results of the original PRB 70/100 with the modified bitumen, the adhesion to glass increases by 32...34% with the addition of 0.3% surfactant, by 34...36% with 0.4%, and by 36...38% with 0.5% in bitumen.

The results of the adhesion of modified bitumen test with crushed stone showed that at 0.3% surfactants in bitumen, the grafting increases by 20%, at 0.4% surfactants in bitumen by 23%, and by 31% at 0.5% surfactants in bitumen.

Therefore, it can be concludedthat adhesion additives affect the quality of bitumen, improving them. This primarily applies to adhesion parameters, adhesion to glass and crushed stone. At the same time, the impact of Wetfix BE surfactant is slightly higher compared to Stardope 130 P.

At the second stage, we were exploringthe effect of modified bitumen on the main properties of asphalt mixtures. For this purpose, algorithmic experiments were performed in accordance with the three-level two-factor plan B $_{\textrm{\tiny{2}}}$ [24]. The conditions for planning experiments are given in Table 3.

Table 3. Conditions for planning experiments

Influencing factors		Levels of variation			Inter-
Natural view	Coded view			+1	val
Bitumen content in the mixture, $%$, (B)	Χ,	4,6	5,0	5,4	0,3
Additive content, % by weight of bitumen, (A)	Х.		0,4	0,5	0.1

The asphalt mixtures were tested in accordance with DSTU B B.2.7-319:2016 (Ukr. Standard).

At the next stage, during the research, cylinder specimens were manufactured at each point on the basis of modified bitumen in accordance with the composition given in Table 4.

The composition of the asphalt concrete mixture was selected in accordance DSTU B B.2.7-119: 2011 (Ukr. Standard). Asphalt mixtures were prepared using a laboratory mixer.

The planning matrix and the experimental results are shown in Table 5.

Table 4. Asphalt mix composition

Nº.	Material	Composition of the mixture (bitumen and additives over 100%), %	
	Crushed stone fr. 20-40 mm	27	
2	Crushed stone fr.10-20 mm	20	
3	Crushed stone fr.5-10 mm	10	
4	Screening fr. $0-5$ mm	37	
5	Mineral powder	6,0	
6	PRB 70/100	According to the planning matrix	
7	Adhesion additive	According to the planning matrix	

The study and comparison of surfactants effect on the characteristics of the asphalt mixture was carried out in terms of water saturation, compressive strength at 20 °C and 50 °C.

Based on the data obtained, the experimental-statistical models were built:

When using the Wetfix BE additive
\nWater saturation
\nW= 2,01 – 0,62·x₁ – 0,28·x₂ + 0,1·x₁x₂ – 0,01·x₁² – 0,01·x₂² (6)
\nCompressive strength at temperature, 20°C
\n
$$
R^{20} = 4,54 + 0,2·x_1 + 0,17·x_2 0,05·x_1x_2 – 0,08·x_1^2
$$
 (7)
\nCompressive strength at temperature, 50°C
\n $R^{50} = 1,9 + 0,24·x_1 + 0,14·x_2 – 0,07·x_1x_2 – 0,07·x_1^2 + 0,06·x_2^2$
\nWhen using the Stardope 130 P additive
\nWater saturation
\nW= 2,15 – 0,67·x₁ – 0,33·x₂ + 0,08·x₁x₂ + 0,13·x₁² – 0,07·x₂²
\nCompressive strength at temperature, 20°C
\n $R^{20} = 4,16 + 0,23·x_1 + 0,22·x_2 + 0,05·x_1x_2 + 0,01·x_1^2$
\nCompressive strength at temperature, 50°C
\n(10)
\nCompressive strength at temperature, 50°C

$$
R^{50} = 1,78 + 0,23 \cdot x_1 + 0,18 \cdot x_2 - 0,03 \cdot x_1 x_2 - 0,06 \cdot x_1^2 + 0,04 \cdot x_2^2
$$
 (11)

Based on the experimental and statistical mod- $\,$ tion (Fig. 7) and compressive strength at 20 $^\circ$ C (Fig. els (6-11), graphical dependences of water satura-8) and 50 °C (Fig. 9) were drawn.

 Figure 7. Graphical dependences of water saturation of samples using using Wetfix BE (a), Stardope 130 P (b),

Based on the data obtained (Table 5) and graphical dependencies, it can be concluded that modification of bitumen with adhesive additives leads to a decrease in water saturation of the samples and an increase in strength.

Evaluating the Stardope 130 P additive, it can be concluded that with an increase in the content of bitumen and surfactants, water saturation

decreases from 3.3% to 1.3%, with the influence of bitumen being more significant compared to surfactants. It should be noted that, according to the mathematical model (9), there is a certain interaction of the influence factors, in particular, with a smaller amount of bitumen in the mixture, the influence of the additive is more significant.

Figure 8. Graphical dependences of the compressive strength at 20°C when using Wetfix BE (a), Stardope 130 P (b)

 Figure 9: Graphical dependences of the compressive strength at 50°C when using Wetfix BE (a), Stardope 130 P (b)

Varying factors have a similar effect on the compressive strength at temperature 20°С. Under these conditions, the strength increases from 3.8...4.2 MPa to 4.25...4.75 MPa. However, at a temperature of 50°С, the effect is somewhat different the influence of the additive decreasing.

When bitumen amount is up to 5.0%, the increase is more significant compared to the amount of 5.0...5.4%, which can be explained by the excessive amount of modified binder in the asphalt mixture.

Tests of the Wetfix BE additive have shown that an increase in the content of the additive in bitumen also has a positive effect on the properties of asphalt mixtures. The water saturation of the samples decreases from 3.0% to 1.1% at the maximum content of bitumen and additive within the range of variation.

An increase in the compressive strength at temperature 20°С from 4,15…4,4 MPa to 4,45…4,9 MPa can be obtained at the maximum content of bitumen and additives within the range of variation. However, at a bitumen consumption of more than 5.0 % with a minimum content of surfactants, the strength practically does not increase. The influence of the studied factors on the strength at the temperature of 50°С is somewhat different, with the optimal bitumen content in terms of strength being 4.7...5.2%. With a further increase in bitumen, the effect of the additive fades.

To compare the effectiveness of adhesion additives, the properties of asphalt mixtures without surfactant additives were determined. The composition of the mixtures is given in Table 4. The bitumen content was 5%, and the surfactant additives were 0.3%, 0.4%, and 0.5%. The obtained experimental results are shown in Table 6 and graphical dependencies in Figs. 10... 12.

Figure 10: Graphical dependences of water saturation of samples asphalt mixtures

Figure 11. Graphical dependence of compressive strength of asphalt concrete at 20°C

Analyzing the data obtained (Table 6) and the graphical dependences of water saturation and compressive strength at 20°C and 50°C of asphalt mixtures (Figs. 10... 12), it can be concluded that samples without the use of adhesive additives have significantly lower corresponding indicators. The water saturation of asphalt concrete without surfactant additives is more than 3.5%, which does not meet the requirements of the regulatory document (≤3.5%) (DSTU B B.2.7-319:2016 (Ukr. Standard). Thus, it is possible to ensure all regulatory indicators with the use of adhesive additives.

Figure 12. Graphical dependence of compressive strength of asphalt concrete at 50°C

4. CONCLUSIONS

1. The introduction of adhesive additives improves the basic properties of bitumen, which are decisive for obtaining high-quality asphalt concrete mixtures. Modification of bitumen increases adhesion to glass by 75...85%, and to crushed stone by 40...50%.

2. Using methods of mathematical planning of experiments, experimental and statistical models of water saturation and compressive strength of asphalt concrete mixtures at temperatures of 20°C and 50°C were obtained. Analysis of the models made it possible to find the optimal content of adhesive additives depending on the bitumen content in the mixture.The introduction of modifying additives can significantly reduce the water saturation of asphalt concrete mixtures by up to 50%, and increase the strength by 25...40% and 32...65% at temperatures of 20°C and 50°C, respectively.

3. Compositions of asphalt concrete mixtures containing adhesive additives have been proposed that provide the necessary standardized indicators.

4. Further research is planned to be carried out in the direction of the influence of the composition of the asphalt concrete mixture on the effectiveness of the use of modifying additives

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IZVOD

UTICAJ ADITIVA LEPILA NA SVOJSTVA BITUMENA I ASFALTNIH SMEŠA

U ovom radu su prikazani rezultati eksperimentalnih istraživanja uticaja adhezivnih aditiva na osnovna svojstva modifikovanih bitumenskih i asfaltbetonskih mešavina. Uvođenjem adhezivnih aditiva poboljšavaju se osnovna svojstva bitumena, neophodna za dobijanje visokokvalitetnih asfaltbetonskih mešavina. Modifikacija bitumena povećava adheziju na staklo i drobljeni kamen. Korišćenjem metoda matematičkog planiranja eksperimenata dobijeni su eksperimentalni i statistički modeli zasićenosti vodom i čvrstoće na pritisak asfaltbetonskih mešavina na temperaturama od 20°C i 50°C. Uvođenje modifikujućih aditiva može značajno smanjiti zasićenje vodom asfaltbetonskih mešavina i povećati čvrstoću na temperaturama od 20 °C i 50 °C.

Ključne reči: bitumen, asfaltne mešavine, čvrstoća, adhezivni aditivi, penetracija, tačka omekšavanja, duktilnost.

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