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INVESTIGATION OF THE WETTING EFFECT OF SURFACTANTS OF VARIOUS NATURE IN THE BITUMEN COMPOSITION

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Abstract. *Introduction.* Asphalt concrete coverings are short-lived and are destroyed under the influence of the external factors. The insufficient degree of adhesion at the interface between the bituminous binder and the hard surface of the mineral material causes a low hydrophobicity of asphalt concrete coatings, which is the main cause of damage and premature wear of the roadway. *The purpose*is to establish the influence of various modifiers on the processes of wetting the surface of crushed stone with a bituminous composition, as well as to study the regularities of the influence of modifiers on the hydrophobicity of bituminous films.*The methodology* of this work included experimental determination of the wetting contact angle θ of a solid surface by the lying drop method at T=298 K.*Results.*Increasing the AC-2K content in bitumen from 0 up to has 1% reduced the contact wetting angle by 11.31° (relative to the base version without the modifier) to 114.35°. The AC-2 modifier contributes less to improving the wettability of the surface of the basic crushed stone with bitumen; with C_m=1%, the contact wetting angle is reduced to 118.68°. The amino derivative AC-2K (C_m=1%) has the greatest water-repellent effect among the studied modifiers, increasing the wetting edge angle up to 99.07°.*Conclusion*. The ultrasonic cavitation-derived AC-2K can be recommended for use in asphalt concrete coatings as an adhesive additive, which provides a good wetting effect of bitumen in relation to the surface of the mineral filler while also increasing the hydrophobicity of bitumen films.

Keywords: asphalt concrete coatings, bitumen, mineral fillers, modifiers, adhesion, wetting, hydrophobicity of bitumen films.

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1. Introduction

The most common type of road coverings are asphalt-concrete mixtures based on bitumen. One of the most important requirements for the road bitumen properties is a high adhesive ability, which allows the mineral filler particles to be retained. Furthermore, bitumen, which fills cracks on the surface of mineral particles, has water-repellent properties and prevents moisture from destroying the mineral structure of the material [1-2]. Inadequate adhesion at the interface between the bituminous binder and the hard surface of the mineral material causes low hydrophobicity of asphalt concrete coatings, which is the primary cause of roadway damage and premature wear [3]. The inability of bitumen to wet mineral material results in a low adhesion. This is especially true for acidic crushed stone [4].

Bitumen composition contains a variety of functional groups, allowing it to be modified with various additives. The addition of various modifying agents to bitumen can intensify the processes of wetting the solid surface with bitumen and increase the hydrophobicity of the surface, films formed by it [5-10].

Polymer additives are currently of great interest in terms of bitumen modification. This is primarily due to the fact that synthetic polymer material production is increasing at a rapid pace each year. Another important consideration is that the process of modifying bitumen imparts useful properties in the operation of road surfaces, which are typical of polymers: elasticity, ductility, strength, and resistance to aggressive environments [11-12]. Polymer-bitumen binders (PBB) have been widely used in advanced countries over the last 15-20 years; in Europe, this figure is around 20%, while in the US, it is around 15%. The use of PBB in the production of asphalt mixes extends the service life of coatings and lowers the cost of road repairing [13]. Meanwhile, Kazakhstan is rich in raw materials and has a large amount of petrochemical wastes, which allow for the synthesis of surface-active modifiers with the possibility of using them as cheap, effective, and competitive additives in bituminous compositions. The addition of modifiers to asphalt concrete coatings improves the performance characteristics such as frost and heat resistance, hydrophobicity, and strength. Adhesive polymer additives help to improve a binder distribution on the surface of the mineral filler [14-18].

Thus, the addition of modifying additives allows for fine-tuning of the hard surface wetting processes and influence of the hydrophobic properties of bituminous coatings.

The goal of this study has been to determine the effect of various modifiers on the wetting processes of bituminous compositions.

This has necessitated a research into the wetting processes of various mineral fillers, depending on the quantitative content of the polymer in the bitumen; - research into the regularities of the influence of modifiers on the hydrophobicity of bitumen films.

2. Experimental part

Viscous road bitumen of the BND 90/130 brand (GOST 22245-90) was used in the study. We used:

- mineral filler-crushed stone of two different types: acidic alaskite (red granite crushed stone) and basic dolomitized limestone (gray crushed stone);

- the following modifiers:

1. polyphenylsiloxane (PPS) (GOST11066-74);

2.иamine-derived surfactant AC-2, obtained by amination of cubic petrochemical residues KON-92 [19].

3. amino derivative of the surfactant AC-2K, synthesized, using the same method as AC-2, but with the addition of ultrasonic cavitation.

The surfactant AC-2 was prepared in accordance with TU 655-RK 05606434-001-2000 and the recommendations in the patent link [19]. The reaction of carbamide amination of cubic residues of KON-92 (a mixture of esters, organic acids, and alcohols) in the presence of glacial acetic acid was the basis for the synthesis. One part of glacial acetic acid was added to ten parts of KON-92. The reaction mixture was brought to a boil and held for 5 minutes. After that, 5 parts of urea were added. The mixture was separated in a dividing funnel after cooling (the target product was the upper fraction). AC-2K, a surfactant, was synthesized, using the same technological scheme, but with the addition of ultrasonic cavitation. The frequency was kept constant at 60 kHz.

The wetting contact angle, defined as the angle between the solid surface and the tangent at the point of contact of the three phases, was a criterion for experimentally determining a wetting capacity of the liquid (Figure 1 a, b).

Figure 1 – Images of a drop on a solid surface: a – a drop of water on a bitumen film; b-a drop of bitumen on the surface of crushed stone.

The lying drop method was used to measure the contact angles of wetting the surface of bitumen films with water. The effect of the additive concentration in bitumen on the wetting processes was investigated using an ACAM series device.

3. Results and Discussion

The quantitative contents of modifying additives have an effect on the wetting index of crushed stone of various types, as shown in Figures 2-3.

According to the results of the analysis, the surface of the crushed stone is lyophobic to bitumen. The edge angle on the surface of red granite rubble is 126.87° in the absence of modifiers in bitumen, and 125.66° in the case of gray rubble. The greatest wetting activity of the modifiers is demonstrated in relation to the basic nature's surface. Modifier concentrations of 1% account for the maximum wetting effect (the minima in Figure 2, curves 1, 2, 3). A comparison of the depth of the extremes at a given concentration (C_m=1%) reveals that the greatest decrease occurs in the presence of AC-2K. Thus, increasing the AC-2K content in bitumen from 0 up to 1% has reduced the contact wetting angle by 11.31° (relative to the base version without the modifier) to 114.35°. The AC-2 modifier contributes less to improving the wettability of the surface of the basic crushed stone with bitumen; with C_m=1%, the contact wetting angle is reduced to 118.68°. When modifying bitumen, PPS has recorded a significantly smaller decrease in the wetting contact angle at a higher concentration (C_m=2%) - by 2.4° (from 125.66° up to 123.26°).

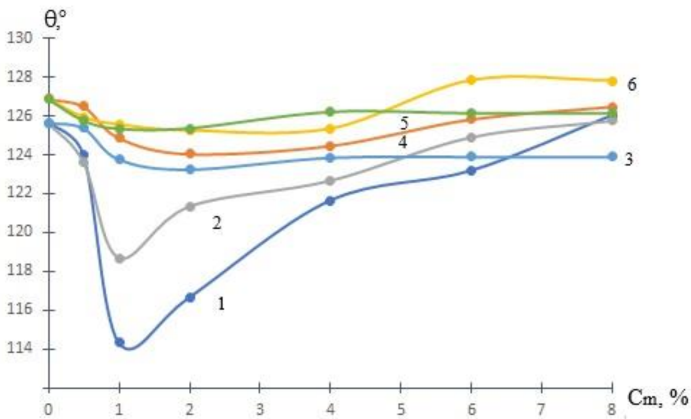

Figure 2 - The effect of the modifier concentration on the contact angle of wetting of the surface of crushed stone of basic (1, 2, 3) and acidic (4, 5, 6) nature: 1, 4 – AC-2K; 2, 5 – AC-2; 3, 6 – PPS.

Outside of these concentration zones (C_m>1%), there is a significant increase in θ for the composition of bitumen with two types of amino derivatives, as well as a significant decrease in wetting. As the concentration of AC-2K in the composition has increased (over 6%), the wetting contact angle begins to approach that of unmodified bitumen (θ=126.02° at C_m=8%). A similar phenomenon has been observed with an increase in θ in the AC-2K content of more than 6%. The addition of more than 2% of PPS to the bituminous composition had no effect on the wetting ability of the gray crushed stone surface.

The wetting activity of modifying additives Has been lower in red granite rubble than in gray rubble (Figure 2, curves 4, 5, 6). Another feature of this surface is the leveling of the difference in the wetting activity between all modifiers, which has been established for the basic nature surface. When the quantitative content of AC-2K in bitumen has changed within the same limits (C_m=2%), the values of θ on the acidic surface has decreased only by 2.81° relative to the base version, which is four times less than the effect of the same modifier on the basic surface.The AC-2 modifier contributes to the reduction of the wetting contact angle to a lesser extent; the greatest reduction is achieved at C_m=2% (θ=1.63°).

The maximum wetting activity of PPS on the surface of red granite rubble has been observed when the modifier content in bitumen ranges from 2 to 4%. The wetting angle has decreased by a very small amount (θ=1.52°-1.54°) in this zone of polyorganosiloxane concentration and has amounted to 125.35-125.37°.The linear sections of the wetting isotherms in the presence of the modifiers (Figure 2) have been approximated by an equation 1 of the form:

$$\cos\theta = z\, C_m + \cos\theta_0, \qquad (1)$$

$\cos\theta_0$ – the cosine of the angle formed by the bituminous binder without a modifier;

z – constant.

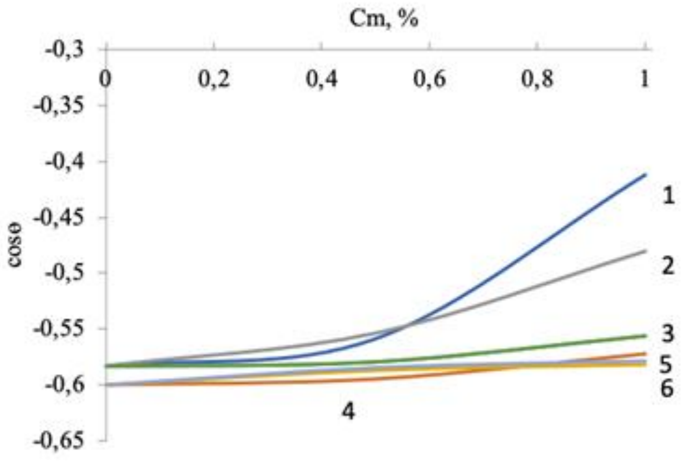

Figure 3 – The effect of the modifier concentration on the wetting index of basic (1, 2, 3) and acid (4, 5, 6) crushed stone: 1, 4 – AC-2K; 2, 5 – AC-2; 3, 6 – PPS.

When we differentiate equation (1), we get:

$$d\cos\theta / dC_m = z \qquad (2)$$

Table 1 displays the corresponding z values.

Table 1 – The value of the wetting ability criterion of z additives in relation to the surfaces of the acidic and basic nature of crushed stone

Nature of crushed stone	**AC-2**	**AC-2K**	**PPS**
Gray crushed stone (plagiogranite)	0.103	0.17	0.027
Red crushed stone (alaskite)	0.018	0.028	0.021

According to the data analysis, the addition of AC-2K most effectively contributes to the reduction of surface tension at the interface between the phases "bituminous composition - gray crushed stone". This is demonstrated by the composition's maximum wetting capacity, $d\cos\theta/dC_{AC\text{-}2K}$=0.17 (Table 1). The compensation of excess surface energy at the interface with gray crushed stone is lower in the AC-2 compositions than in the AC-2K compositions. The wetting ability of such compositions is $d\cos\theta/dC_{AC\text{-}2}$=0.103, which is 1.7 times less than the similar characteristics of bitumen modified by AC-2K. PPS has a negligible hydrophobic effect ($d\cos\theta/dC_{PPS}$=0.027). The effect of changing the surface tension at the interfacial boundary "bitumen-modifier-red crushed stone" is less pronounced for the same compositions. All additives have a similar bitumen wetting ability on the surface of red crushed stone (0.018-0.028), which is an order of magnitude lower than their ability to wet gray crushed stone.

In the absence of the modifiers, the contact angle of wetting the bitumen surface with water is 95.05°, according to experimental data (Figure 4). According to the well-known Young's equation [20], the surface is hydrophilic when θ=90°. If the surface tension at the bitumen-gas boundary ($\sigma_{b\text{-}g}$) is less than the surface tension at the bitumen-water boundary ($\sigma_{b\text{-}w}$) and θ>90°, water does not wet the surface of bitumen, indicating that bitumen has water-repellent properties. As a result, the bitumen surface (θ=95.05°) is located on the wettability/non-wettability boundary.

The addition of the surfactants to bitumen has resulted in an increase in the wetting contact angle in a narrow concentration region (Figure 4). AC-2K exhibits the greatest water-repellent effect with a low modifier concentration (C_m=1%), with a value of θ, increased by 4.02º relative to the base version and totaling 99.07º. (Fig. 4, curve 1). A slight deterioration in the water-repellent properties of bituminous compositions and stabilization of wetting edge angle values have been observed outside of the specified concentration area. AC-2 has a lower contribution to the increase in hydrophobicity of bituminous films ($\Delta\theta$=1.56° at C_m=1%). PPS has almost no effect on the hydrophobization of bituminous film surfaces (Figure 4, curve 3).

When the modifiers are introduced, the obtained wetting isotherms act as the detectors of changes in the intermolecular interactions within the bitumen structure. The formation of the mixed spatial structures in a narrow concentration range ($C_m \leq$1%) contributes to the weakening of the intermolecular interactions within the compositions, resulting in an increase inθ. As their concentrations rise further, the system becomes less discrete, resulting in a slight decrease and stabilization of the wetting contact angle.

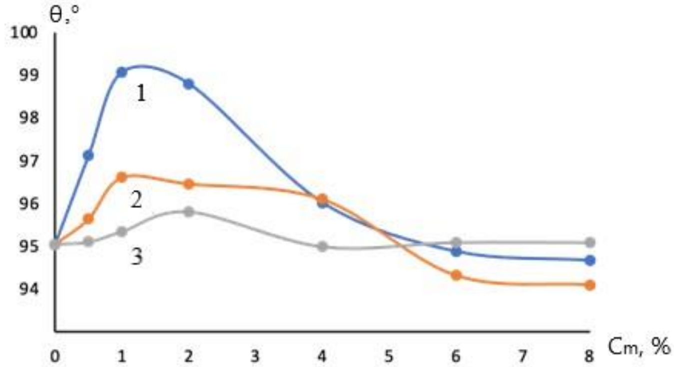

Figure 4 – The effect of the modifier concentration on the edge angle of wetting of bituminous films: 1 – AC-2K; 2 – AC-2; 3 – PPS.

Thus, the amino derivative AC-2K (C_m=1%) has the greatest water-repellent effect among the studied modifiers, increasing the wetting contact angle to 99.07°.

4. Conclusion

The amino derivative AC-2K has been discovered to possess the greatest effect on the wetting processes of the mineral fillers and the hydrophobicity of bituminous films of the studied modifiers. The addition of 1% of AC-2K to bitumen has reduced the wetting angle by 11.31° (relative to the base version on the surface of the grey crushed stone) to 114.35°. Of the two varieties of amino derivatives, the one synthesized with ultrasonic cavitation shows the best wetting effect.

The water-repellent properties of the bituminous compositions are enhanced ($\Delta\theta$=4.02°)in the narrow range of concentrations of the modifier AC-2K (with C_m=1%).

The ultrasonic cavitation-derived AC-2K can be recommended for using in asphalt concrete coatings as an adhesive additive, which provides a good wetting effect of bitumen in relation to the surface of the mineral filler, while also increasing the hydrophobicity of bitumen films.

БИТУМ ҚҰРАМЫНДАҒЫ ӘР ТҮРЛІ СИПАТТАҒЫ БЕТТІК БЕЛСЕНДІ ЗАТТАРДЫҢ СУЛАНУ ӘСЕРІН ЗЕРТТЕУ

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Түйіндеме. *Кіріспе.* Асфальтбетон жабындары қысқа мерзімді және сыртқы факторлардың әсерінен жойылады. Битумды байланыстырғыш пен минералды материалдың қатты беті арасындағы интерфейсте адгезияның жеткіліксіз дәрежесі асфальтбетон жабындарының төмен гидрофобтылығын тудырады, бұл жол төсемінің зақымдануының және мерзімінен бұрын тозуының негізгі себебі болып табылады. Әр түрлі модификациялаушы агенттерді енгізу арқылы битум құрамын оңтайландыру қатты бетті битуммен сулау процестерін күшейтіп, ол түзетін беттік пленкалардың гидрофобтылығын арттыра алады. Бұл жұмыстың *мақсаты*– әртүрлі модификаторлардың қиыршық тастың бетін битуммен сулау процестеріне әсерін анықтау, сонымен қатар модификаторлардың битум пленкаларының гидрофобтылығына әсер ету заңдылықтарын зерттеу. *Методологиясы*. Бұл зерттеудің әдістемесі–T=298 K кезінде жатқан тамшы әдісімен қатты беттің θ сулануының шеткі бұрышын эксперименттік анықтауды қамтыды. *Нәтижелер.* Битумдағы АС-2К құрамының 0-ден 1%- ға дейін артуы сулануының шеткі бұрышын11,31°-қа (модификаторсыз негізгі нұсқамен салыстырғанда) 114,35°-қа дейін азайтты. АС-2 модификаторы аз дәрежеде негізгі қиыршық тастың бетін битуммен ылғалдандыруды жақсартуға ықпал етеді; $C_м$=1% сулануының шеткі бұрышы 118,68°дейін төмендейді. ПФС битумын өзгерту кезінде жоғары концентрацияда ($C_м$=2%) сулануының шеткі бұрышының айтарлықтай аз төмендеуі байқалды-2,4° (125,66°-123,26°). АС-2К амин туындысы ($C_м$=1%) зерттелген модификаторлар арасында ең үлкен су өткізбейтін әсерге ие, сулануының шеткі бұрышын 99,07°дейін арттырады. *Қорытынды.* Битумға 1% АС-2К енгізу ылғалдандыру бұрышын 11,31°-қа (негізгі қиыршық тас бетіндегі негізгі нұсқаға қатысты) 114,35°-қа дейін азайтты. Амин туындыларының екі түрінің ішінен ультрадыбыстық кавитация арқылы синтезделген түрі ең жақсы ылғалдандыру әсерін көрсетеді. Битум пленкаларының су өткізбейтін қасиеттері АС-2К модификатор концентрациясының тар диапазонында ($C_м$=1%) жоғарылайды (Δθ=4,02°). АС-2К ультрадыбыстық кавитация арқылы алынған асфальтбетон жабындарында жабысқақ қоспа ретінде пайдалану ұсынылуы мүмкін, бұл битум пленкаларының гидрофобтылығын арттыра отырып, минералды толтырғыштың бетіне қатысты битумды сулаудың жақсы әсерін қамтамасыз етеді.

Түйінді сөздер: асфальтбетонды жабындар, битум, минералды толтырғыштар, модификаторлар, битум қабықшаларының адгезиясы, сулануы, гидрофобтылығы

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ИССЛЕДОВАНИЕ СМАЧИВАЮЩЕГО ЭФФЕКТА ПОВЕРХНОСТНО-АКТИВНЫХ ВЕЩЕСТВ РАЗЛИЧНОЙ ПРИРОДЫ В СОСТАВЕ БИТУМА

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Резюме. *Введение***.** Асфальтобетонные покрытия недолговечны и разрушаются под воздействием внешних факторов. Недостаточная степень адгезии на границе раздела между битумным вяжущим и твердой поверхностью минерального материала обуславливает низкую гидрофобность асфальтобетонных покрытий, что является основной причиной повреждения и преждевременного износа дорожного полотна. *Цель* данной работы - установить влияние различных модификаторов на процессы смачивания поверхности щебня битумом, а также изучить закономерности влияния модификаторов на гидрофобность битумных пленок. *Методология* данного исследования включала экспериментальное определение краевого угла смачиванияθ твердой поверхности методом лежащей капли при T=298 K. *Результаты.* Увеличение содержания АС-2К в битуме с 0 до 1% уменьшило краевой угол смачивания контакта на 11,31° (по сравнению с базовой версией без модификатора) до 114,35°. Модификатор АС-2 в меньшей степени способствует улучшению смачиваемости поверхности основного щебня битумом; при $С_м$=1% угол смачивания уменьшается до 118,68°. Аминопроизводное АС-2К ($С_м$=1%) обладает наибольшим водоотталкивающим эффектом среди изученных модификаторов, увеличивая угол смачивания до 99,07°. *Заключение*. Из двух разновидностей аминопроизводных та, которая синтезирована с помощью ультразвуковой кавитации, демонстрирует наилучший смачивающий эффект. Водоотталкивающие свойства битумных пленок повышаются (Δθ=4,02°) при узком диапазоне концентраций модификатора АС-2К (при $С_м$=1 %). Полученный с помощью ультразвуковой кавитации АС-2К может быть рекомендован для использования в асфальтобетонных покрытиях в качестве адгезивной добавки, которая обеспечивает хороший эффект смачивания битума по отношению к поверхности минерального наполнителя, одновременно повышая гидрофобность битумных пленок.

Ключевые слова: асфальтобетонные покрытия, битум, минеральные наполнители, модификаторы, адгезия, смачивание, гидрофобность битумных пленок

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