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INFLUENCE OF POTASSIUM HUMATE ON THE TECHNOLOGICAL AGING PROCESSES OF OXIDIZED PETROLEUM BITUMEN

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Abstract. The possibility of using potassium humate as an inhibitor of the technological aging of oxidized petroleum bitumen has been investigated. Samples of potassium humate obtained from various raw materials, in particular peat and leonardite, were selected and compared. Aging coefficients have been calculated and operational properties of modified bitumen as a binding material for hot mix asphalt have been investigated. It was established that 3.0 wt. % of PH-3 added to oxidized petroleum bitumen, slows down the technological aging processes and allows obtaining asphalt concrete with better characteristics compared to the coating made on the basis of unmodified bitumen.

Keywords: bitumen, bitumen aging, modification, oxidized bitumen, potassium humate, pavement.

1. Introduction

One of the main factors affecting the duration of the pavement operation is the bitumen ability to retain its binding properties for a long time. In this regard, the issue of petroleum bitumen resistance to technological aging is of particular importance.

It is known that technological aging is a set of processes, which occur during production, pumping of hot bitumen through gutters and storage of the finished product in tanks with a large area of contact with air, as well as during the subsequent production of ready-made hot mix asphalt. At the same time, at this stage, the main reasons for the loss of bitumen properties are the high temperatures of the mentioned processes and the contact of hot bitumen with the oxygen of the air.

Unlike technological aging, operational aging of a thin bitumen film is affected by atmospheric oxygen, ultraviolet radiation, high temperature, and water. As a result, bitumen continues to lose its binding properties. However, it is worth noting that, since the bitumen aging index is five times higher at the technological stage than at the operational stage, the impact of the above-mentioned factors should be minimized and bitumen with enhanced resistance to technological aging should be produced.

Today, there are already several compounds used as aging inhibitors, in particular, antioxidants and plasticizers. The plasticizers reduce the viscosity of the dispersion medium (oil and resins) and the number of structural elements (asphaltenes) per unit of bitumen volume. Taking into account that plasticizers are mainly designed to "dilute" the binder, they include tar, oil fuel, oil extracts, industrial oils, *etc*.

Antioxidants are used to slow down or block oxidation reactions that occur while heating the binder and contacting with the oxygen of the air. Depending on the antioxidant action mode they are divided into primary antioxidants capable of "scaring off" radicals and secondary antioxidants which are designed to inhibit the formation of peroxides, thereby preventing the beginning of the oxidation process.[1] Primary antioxidants include vitamin E, furfural, butylated hydroxytoluene, "protective" phenolic antioxidants, and stabilizers. The secondary ones are dilauryl thiodipropionate (DLTDP), aminogenic surfactants, N-phenyl-2-naphthylamine, zinc dibutyldithiocarbamate (ZDBC), thin-crystalline aliphatic hydrocarbons with long chains, polyfunctional additives based on fatty amine surfactants and polyethylene, C18 fatty unsaturated acids, derivatives of fatty amines, *etc*.[2-9] Ouyang *et al.*,[7] as well as Martin,[10] considered primary antioxidants to be more effective, but it is also possible to combine primary and secondary antioxidants into complexes, which in some cases allows to ensure their higher thermo-oxidative resistance.[3]

At the same time, four main groups of compounds capable of slowing down the oxidation of hydrocarbons in bitumen are identified:[11]

– phenols that break the chain reaction with peroxyl radicals;

– inhibitors that slow down aging by reacting with alkyl radicals;

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– agents that break peroxides without the formation of free radicals;

– agents that consume oxygen molecules faster than they enter into oxidation reactions.

Among the compositions that include these additives, the most widely used are amines, "preventing" phenols (with one or more bulky functional groups, *e.g.*, *tert*-butyl), phosphites, and organic zinc compounds.[3,7,12] However, the use of organic zinc compounds requires significant economic costs, which is a serious obstacle to their widespread use.[13,14]

Apart from antioxidants and plasticizers, some polymers and adhesive additives also have a positive effect on slowing down the technological aging processes. In particular, this applies to terpolymers, waxes, and adhesive additives belonging to cationic surfactants. At the same time, none of the above modifiers in their pure form is an antioxidant that could prevent aging from oxygen radicals.[15-19]

Another area of research that is gaining popularity is the use of simple and natural substances as antiaging agents, which can also be found directly in the environment. Thus, in recent years, the attention of researchers has increased significantly to such "natural" antioxidants as phospholipids, ascorbic acid, diatomaceous earth, lignin from rice husks and wood, *etc*.[13,20]

The experimental results demonstrated a positive effect on the slowing down of bitumen aging processes, which gives a boost to the study of other natural compounds as aging inhibitors for petroleum bitumen.[21,22]

From this point of view, the class of compounds to which humic substances belong is of great interest. As it is known today, humic substances are a complex mixture of natural organic compounds formed during the biochemical decomposition of dead plants and their subsequent humification. This class of compounds contains a large set of different functional groups, in particular carboxyl, carbonyl, and amine groups, which allows to use the corresponding compounds as modifiers of petroleum bitumen.

Among a number of humic substances, which include humic and hymatomelic acids, fulvic acids, and humic acids, the most valuable are humic acids or their salts, in particular, potassium humate. Depending on the raw material, this salt can be extracted using a weak alkali solution from peat, lignite or leonardite. Moreover, depending on the raw material type, the content of humic acids in the finished product will vary. The lowest amount of humic acids (up to 25 wt. %) is contained in brown coal, 20–70 wt. % are in peat, and the highest amount (95 wt. %) is in leonardite.[23]

In addition to the highest content of humic acids among all the samples, leonardite is also a natural mineral complex of phenolic hydrocarbons, which suggests the high antioxidant properties of potassium humate obtained from this type of raw material. Based on the foregoing, it can be assumed that potassium humate can be used as an inhibitor of technological aging of paving bitumen.

Therefore, the research goal was formulated, which was to obtain petroleum bitumen resistant to technological aging. At the same time, among the tasks that were planned to be solved to achieve this goal, the following were identified:

– to establish the possibility of using potassium humate as an inhibitor of technological aging of oxidized petroleum bitumen;

– to select potassium humate with an optimal content of humic acids for further use as an aging inhibitor of paving bitumen;

– to determine the operational properties of hot mix asphalt based on a binder modified with potassium humate.

2. Experimental

2.1. Materials

The modification was carried out using the following materials:

– oxidized petroleum bitumen of BND 60/90 grade, obtained from PJSC Ukrtatnafta (Kremenchuk, Ukraine);

– potassium humate with different amounts of humic acids, depending on types of raw materials (peat and leonardite).

The characteristics of the initial materials are shown in Tables 1 and 2

To determine the preparation of stone mastic asphalt (SMA), the following materials were used:

- crushed stone parts and crushed stone from natural stone, fr. 0–5, 5–10, 10–15 mm from LLC Novograd-Volyn Stone Crushing Plant;
- limestone mineral powder brand MP I;
- stabilizing additive Antrocel-G.

To determine the adhesion to gravel (AG) the fraction of 20–40 mm of crushed stone was used. The fraction was obtained from natural stone supplied by JSC "Mokriansky Stone Quarry No.3" (Ukraine).

2.2. Experimental Procedure

Before the modification, granular samples of potassium humate were ground into powder to improve their interaction with bitumen. Modification of bitumen was carried out in a metal container placed on a laboratory hotplate and equipped with a mechanical stirrer and a thermometer. A certain amount of bitumen BND 60/90 was heated up to the appropriate temperature (393 K), and then the appropriate modifier was added. The amount of potassium humate as a modifier (1.0, 3.0, and 5.0 wt. %) was investigated experimentally. The mixture was stirred for 1 h at 1000 rpm.

Table 1. Physical and mechanical characteristics of BND 60/90 bitumen

Index	Unit of measurement	Value
Penetration at 298 K	dmm	63
Softening point	K	321
Ductility at 298 K	cm	62.3
Adhesion to gravel	point	3.0
Solubility in organic solvent	%	99.9
Elastic recovery at 298 K	%	15.0
Resistance to hardening at 436 K (RTFOT method):		
Mass change	wt. %	0.06
Softening point after RTFOT	K	327.2
Penetration at 298 K after RTFOT	dmm	38
Softening point change	K	6.2
Retained penetration	%	60.3

Table 2. Physical and chemical characteristics of potassium humate (PH)

Index	Unit of measurement	Value		
		PH-1	PH-2	PH-3
Raw material	–	peat	leonardite	leonardite
Appearance	–	granules	powder	granules
Color	–	black	black	black
Amount of humic acids	wt. %	30.0	80.0	85.0
Amount of K_2O	wt. %	10.0	12.0	15.0
Solubility in water	%	99.0	99.0	98.0

2.3. Methods of Analysis

The main physico-chemical characteristics of the original and potassium humate-modified bitumen were determined according to standardized methods. In particular, the following were determined: softening temperature;[24] penetration;[25] ductility;[18] and adhesion to gravel.[26] To study the resistance of bitumen to technological aging, the RTFOT method was used,[27] which involves samples heating in a furnace at a temperature of 436 K and a constant air supply for 75 min. Under appropriate conditions, the film of bituminous binder, which is scrolled in cylindrical flasks, constantly overflows, which allows activating the processes of technological aging evenly for the entire bitumen sample.

To show the effect of aging on the properties of bitumen the value of retained penetration was calculated using Eq. (1):

$$Retained\ penetration = \frac{P^{298}_{AGED}}{P^{298}_{UNAGED}} \cdot 100\ \% \qquad (1)$$

where P^{298}_{AGED} is the needle penetration depth determined at 298 K for the sample after aging, 0.1 mm; P^{298}_{UNAGED} is the needle penetration depth determined at 298 K for the sample before aging, 0.1 mm.

In order to determine the influence of the introduced modifier on slowing down technological aging processes, the aging coefficients were calculated for the original and modified bitumen samples with the optimal amount of potassium humate. In particular, the penetration depth coefficients of the needle at 298 K, K^A_P, and the softening temperature coefficients K^A_{SP}, were calculated according to Eqs. (2) and (3), respectively:

$$K^A_P = \frac{P^{298}_{AGED} - P^{298}_{UNAGED}}{P^{298}_{UNAGED}} \cdot 100\ \% \qquad (2)$$

$$K^A_{SP} = \frac{SP_{AGED} - SP_{UNAGED}}{SP_{UNAGED}} \cdot 100\ \% \qquad (3)$$

where SP_{UNAGED} is the softening point of the sample before aging, K; SP_{AGED} is the softening point of the sample after aging, K.

3. Results and Discussion

The addition of potassium humate to oxidized petroleum bitumen with different content of humic acids in the samples made it possible to obtain modified bitumen with the characteristics given in Table 3.

Table 3. Physical and mechanical properties of bitumen modified with potassium humate before RTFOT aging

Bitumen sample	Softening point, K	Penetration at 298 K, dmm	Ductility at 298 K, cm	Adhesion to gravel, points
BND 60/90	321.0	63.0	62.3	3.0
3 wt. % PH-1	322.8	52.0	54.7	3.0
5 wt. % PH-1	322.4	53.0	56.4	3.5
3 wt. % PH-2	325.0	61.0	59.4	3.5
5 wt. % PH-1	323.8	63.0	72.2	4.0
1 wt. % PH-3	324.6	59.0	54.8	3.5
3 wt. % PH-3	324.4	62.0	62.6	4.0
5 wt. % PH-3	324.0	64.0	75.6	4.0

Table 4. Physico-mechanical characteristics of modified bitumen after RTFOT aging

Index	Unit of measurement	Value							
		BND 60/90	BND 60/90 + PH-1		BND 60/90 + PH-2		BND 60/90 + PH-3		
			3 %	5 %	3 %	5 %	1 %	3 %	5 %
Softening point	K	327.2	327.0	327.2	327.4	327.0	327.4	329.2	330.0
Penetration at 298 K	dmm	38.0	36	39	43	55	46	52	54
Change of mass	%	0.06	0.09	0.10	0.11	0.21	0.23	0.31	0.46

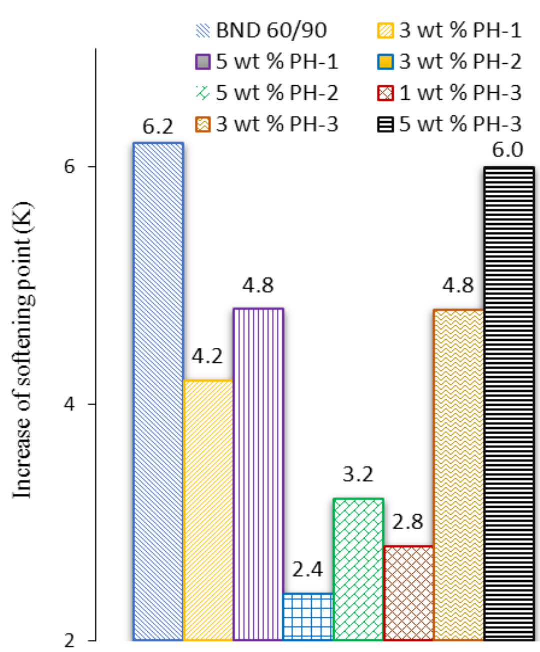

Fig. 1. Influence of potassium humate addition on the increase of bitumen softening point after RTFOT aging

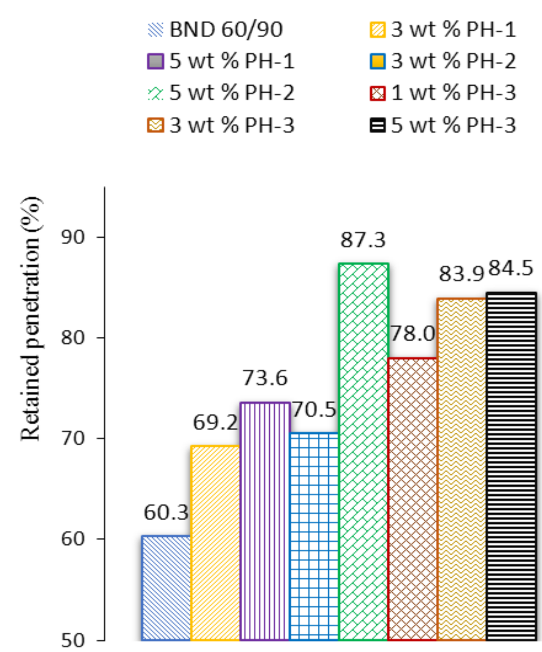

Fig. 2. Influence of potassium humate addition on the bitumen retained penetration fter RTFOT aging

The analysis of the obtained results shows the following:

– according to the needle penetration depth, the best values were found for the samples with 5 wt. % PH-2 (63.0 dmm); 3 wt. % PH-3 (62.0 dmm) and 5 wt. % PH-3 (64.0 dmm);

– according to the softening temperature, the most optimal results were obtained for 3 wt. % PH-2 (325.0 K), as well as 1wt. % PH-3 (324.6 K) and 3 wt. % PH-3 (324.4 K);

– addition of 5 wt. % PH-2 made it possible to increase the elongation to 72.2 cm, while the addition of 3 wt. % PH-3 and 5 wt. % PH-3 increased the value to 62.6 cm and 75.6 cm, respectively, to compare with the original bitumen;

– the samples modified with 5 wt. % PH-2, 3 wt. % PH-3 and 5 wt. % PH-3 also had a high level of adhesion to crushed stone (4.0 points).

Thus, it can be stated that the best results are demonstrated by the samples with 5 wt. % PH-2, 3 wt. % PH-

3, and 5 wt. % PH-3; at the same time they have necessary heat resistance and plasticity.

The change in the properties of bitumen modified with potassium humate after technological aging according to the RTFOT method is shown in Figs. 1, 2 and Table 4.

Comparing the data given in Table 4 and Figs. 2 and 3, the following conclusions can be drawn. The introduction of potassium humate with the lowest content of humic acids (PH-1) and a further increase in the amount of the acids (PH-2 and PH-3) result in the slowing down aging processes in bitumen samples. The best results are observed with PH-2 in the amount of 3 and 5 wt. %, as well as with PH-3 in the amount of 1 and 3 wt. %. For all these samples, the residual penetration is at a high level, which indicates a slight increase in hardness as a result of heating, which is also confirmed by a small increase in the softening point.

Taking into account the obtained results and the initial characteristics of the modified samples, the potassium humate may be recommended as an aging inhibitor of oxidized petroleum bitumen. The best results are observed for the samples with 5 wt. % PH-2 and 3 wt. % PH-3 but the difference in the resulting indices is insignificant. Since PN-3 requires a smaller amount of humate (3 wt. %), it was accepted as the optimal aging inhibitor and is recommended for further research.

To determine the influence of the introduced modifier on slowing down technological aging processes, the aging coefficients were calculated (Table 5) for the original bitumen and bitumen modified with the optimal amount of potassium humate.

The data given in Table 5 demonstrate the rapid slowing down of aging processes when potassium humate is added to the oxidized bitumen. In particular, the introduction of the modifier in the amount of 3 wt. % shows a decrease in the needle penetration depth by 2.46 times, and a decrease in softening point by 1.39 times. It can be assumed that, in proportion to the decrease in the calculated aging indicators, the resistance of oxidized bitumen to technological aging will increase with the introduction of the optimal amount of the corresponding aging inhibitor.

The next stage of research was the design of SMA-15 based on BND 60/90 and BND 60/90 + 3 wt. % PH-3. The composition of SMA-15 (Table 6) included a stabilizing additive (Antrocel-G). Physico-mechanical properties are shown in Fig. 3. The amount of the bitumen binder for all SMA-15 compositions did not exceed 0.20 wt % according to the DSTU B V.2.7-127:2015 requirements.

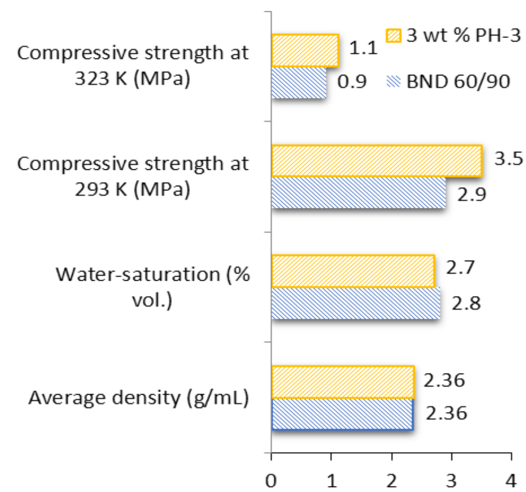

Fig. 3. Physical and mechanical properties of SMA-15

Table 5. Aging coefficients of oxidized bitumen before and after the addition of potassium humate

Bitumen sample	Value	
	K_P^A, %	K_{SP}^A, %
BND 60/90	39.7	12.9
BND 60/90 + 3 wt. % PH-3	16.1	9.3
Ratio unmodified/modified	2.46	1.39

Table 6. Composition of SMA-15

No.	Name of material	Content of material in asphalt concrete, wt. %
1	Aggregates, including fractions:	
	10/15 mm	50
	5/10 mm	20
	0.071/5 mm	15
2	Mineral powder (≤ 0.071 mm)	15
	Total amount	**100**
3	Antrocel-G	0.4
4	Bitumen binder	6.5

Analyzing data from Fig. 3, we observe that the introduction of 3 wt. % PH-3 to bitumen does not actually change the SMA water-saturation indicator. At the same time, SMA with PH-3 has higher compressive strength at 293 K and 323 K (compared to SMA based on unmodified bitumen), which can be explained by improved adhesion of modified bitumen to the surface of mineral material (crushed stone). These results are agreed with those from Table 3. The cohesive strength of SMA increases as well. High value of SMA heat resistance is achieved due to the high softening point of modified bitumen. This is evidenced by the higher value of compressive strength at 323 K compared to SMA based on unmodified bitumen.

4. Conclusions

As a result of oxidized bitumen modification, starting from the introduction of potassium humate with the lowest content of humic acids (PH-1, which is obtained from peat) and with a further increase in the amount of the humic acids (PH-2 and PH-3, which are obtained from leonardite), a slowdown in the technological aging processes is observed in bitumen samples. For the samples with 5 wt. % of PH-2 as well as 3 wt. % and 5 wt. % of PH-3 there are the highest values of residual penetration after the technological aging process, which is 87.3 wt. %; 83.9 wt. % and 84.5 wt. %, respectively. For the samples with 5 wt. % of PH-2 and 3 wt. % of PH-3 the increase in softening points 3.2 K and 4.8 K, respectively. The obtained results allow us to state that the inmentioneddicated amounts of humic acids show the best resistance properties of modified bitumen to technological aging processes. Given the initial characteristics of the samples and the smaller amount of the modifier with a higher concentration of humic acids, the amount of 3 wt. % PH-3 was chosen as an optimal one. The given assumptions were confirmed by calculations using aging coefficients. In particular, the introduction of the PH-3 modifier in the amount of 3 wt. % decreases the needle penetration depth at 298 K by 2.46 times, while the softening point decreases by 1.39. Thus, this concentration of the modifier, namely 3 wt. % PH-3 can be considered optimal for slowing down the technological aging of oxidized petroleum bitumen.

A comparison of the operational properties of SMA based on a binder modified with potassium humate and SMA based on unmodified bitumen also indicates the feasibility of using potassium humate in asphalt concrete pavements.

Abbreviations

P penetration at 298 K (dmm)
PH potassium humate
PH-1 potassium humate from peat (30 wt. % humic acids)
PH-2 potassium humate from leonardite (80 wt. % humic acids)
PH-3 potassium humate from leonardite (85 wt. % humic acids)
RTFOT rolling thin film oven test
SMA stone mastic asphalt
SP softening point (K)

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ВПЛИВ ГУМАТУ КАЛІЮ НА ПРОЦЕСИ ТЕХНОЛОГІЧНОГО СТАРІННЯ ОКИСНЕНОГО НАФТОВОГО БІТУМУ

Анотація. *Досліджено можливість використання гумату калію як інгібітора технологічного старіння окисненого нафтового бітуму. Для порівняння відібрано зразки гумату калію, що одержували з різної сировини, зокрема торфу та леонардиту. Проведено розрахунок коефіцієнтів старіння та досліджено експлуатаційні властивості модифікованого бітуму як в'яжучого матеріалу для гарячих асфальтобетонних сумішей. Встановлено, що додавання до окисненого нафтового бітуму 3.0 % мас. PH-3 дозволяє сповільнювати процеси технологічного старіння та одержувати асфальтове покриття із кращими характеристиками в порівнянні з покриттям, виготовленим на основі немодифікованого бітуму.*

Ключові слова: *бітум, старіння бітуму, модифікація, окиснений бітум, гумат калію, покриття.*