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USE OF CARBON BLACK AS A MODIFIER FOR PETROLEUM BITUMEN



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The problem of recycling worn-out tires and recycling of solid carbon-containing residue from the pyrolysis of tires is considered. The possibility of processing carbon black from the pyrolysis of tires into a high-quality concentrate, which can serve as a raw material for modifying petroleum bitumen, has been shown. The nature of the interactions occurring between the components of carbon black and the matrix of petroleum road bitumens has been studied. The chemical interaction of carbon black with the bitumen matrix through unsaturated C=C bonds has been proven. The introduction of carbon black as a modifying additive leads to a significant change in the physical, mechanical and rheological properties of the resulting bitumen binder systems. Based on spectrometric studies, an assessment is made of the structural changes occurring in the original and modified bitumen during the production of modified bitumen binders. It has been established that with such modification, diffusion of aromatic hydrocarbons occurs from the bitumen component into the modifier particles. During the modification process, six-membered cyclic aromatic compounds are formed in the bitumen part, the composition of paraffin-naphthenic hydrocarbons changes, and sulfur compounds are formed that "transfer" from the modifier particles to the bitumen. Thus, in this study, the phenomenon of mutual diffusion between bitumen and CBWT modifier was confirmed using IR spectroscopy.

KEY WORDS: *bitumen, carbon black from worn tires (CBWT), IR spectroscopy, modifier, bitumen binder, pyrolysis.*

ТЕХНИКАЛЫҚ КӨМІРТЕКТІ МҰНАЙ БИТУМЫНЫҢ МОДИФИКАТОРЫ РЕТІНДЕ ҚОЛДАНУ

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Мақалада авторлар тозған шиналардың қайта өңделуі және де олардың пиролизінен алынған қатты көміртегі бар қалдықтардың утилизациялану мәселелерін қарастырыған. Шиналардың пиролизінен алынған техникалық көміртекті мұнай битумын модификациялау үшін шикізат бола алатындай етіп жоғары сапалы концентратқа өңдеу мүмкіндігі көрсетілген. Техникалық көміртектің құрамдас бөліктері мен мұнай жол битумдарының матрицасы арасындағы өзара әрекеттесулердің табиғаты зерттелді. Техникалық көміртектің битум матрицасымен қанықпаған С=С байланыстары арқылы химиялық әрекеттесуі дәлелденді. Модификациялық қоспа ретінде қолданылған техникалық көміртекті қосқанда пайда болатын битумды байланыстырғыш жүйелердің физикалық, механикалық және реологиялық қасиеттері айтарлықтай өзгеріске ұшырайды. Спектрометриялық зерттеулер негізінде модификацияланған битум байланыстырғыштарын өндіру кезінде бастапқы және модификацияланған битумда болатын құрылымдық өзгерістерге баға берілді. Мұндай модификация кезінде битум компонентінен модификатор бөлшектеріне ароматты көмірсутектердің диффузиясы жүретіні анықталды. Модификация процесі барысында битум бөлігінде алты мүшелі циклді ароматты қосылыстардың түзілуі орын алады, ал парафиндік-нафтенді көмірсутектердің құрамы өзгереді, сонымен қатар, модификатор бөлшектерінен битумға «берілетін» күкірт қосылыстары түзіледі. Осылайша, бұл зерттеуде битум мен ТАТК модификаторы арасындағы өзара диффузия құбылысы ИҚ-спектроскопия көмегімен расталды.

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ИСПОЛЬЗОВАНИЕ ТЕХНИЧЕСКОГО УГЛЕРОДА В КАЧЕСТВЕ МОДИФИКАТОРА НЕФТЯНОГО БИТУМА

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Рассмотрена проблема переработки изношенных автошин, утилизации твердого углеродсодержащего остатка пиролиза автошин. Показана возможность переработки технического углерода пиролиза автошин в высококачественный концентрат, который может служить сырьем для модификации нефтяного битума. Исследована природа взаимодействий, происходящих между компонентами технического углерода и матрицей битумов нефтяных дорожных. Доказано химическое взаимодействие технического углерода с матрицей битума по непредельным С=С связям. Введение технического углерода в качестве модифицирующих добавок приводит к значительному изменению физико-меха-

нических и реологических свойств полученных битумных вяжущих систем. На основании спектрометрических исследований дана оценка структурным изменениям, происходящим в исходном и модифицированном битуме в процессе получения модифицированных битумных вяжущих. Установлено, что при такой модификации происходит диффузия ароматических углеводородов из битумной составляющей в частицы модификаторов. В процессе модификации в битумной части формируются шестичленные циклические ароматические соединения, изменяется состав парафинонафтеновых углеводородов, образуются соединения серы, «переходящие» из частиц модификаторов в битум. Таким образом, в данном исследовании с помощью ИК-спектроскопии было подтверждено явление взаимной диффузии между битумом и модификатором ТУИА.

КЛЮЧЕВЫЕ СЛОВА: *битум, технический углерод из изношенных автошин (ТУИА), ИК-спектроскопия, модификатор, битумное вяжущее, пиролиз*

Introduction. Asphalt concrete pavements are widely used in Kazakhstan, Russia and many other countries. The quality of their construction is largely determined not only by convenience and comfort in operation, but also by the durability of the entire highway. The most important component of asphalt concrete, which determines many of its indicators, is the binding material – bitumen [1].

The properties of bitumen determine the resistance of the coating to weather conditions, strength, durability, etc. By specifically regulating the properties of bitumen, a significant improvement in the service life of coatings can be achieved.

In Kazakhstan, various grades of bitumen are used depending on the specific requirements and conditions of the project. Some of the popular grades of bitumen that can be used in Kazakhstan include BND 50/70, BND 70/100, BND 90/130, BND 40/60, etc. The specific choice of bitumen grade depends on the quality requirements of road construction or other infrastructure projects.

The main reason for the destruction of coatings, provided that construction technology is followed, is the disruption of the structural bonds between the components of asphalt concrete as a result of changes in the properties of bitumen during operation. Under the influence of loads, oxidation, polymerization and other processes on the surface of bitumen, its aging and structure destruction occur.

During the aging process, the chemical and group composition of bitumen changes, and its ability to relax stress decreases. The adhesion forces between the mineral filler and the binder weaken, and under the influence of loads, various defects begin to appear in the asphalt concrete pavement – cracks, peeling, chipping of crushed stone, etc. Thus, we can say that of all the components of asphalt concrete, it is bitumen that determines the durability of the pavement.

Observations of the condition of asphalt concrete pavements show that in recent decades their service life has been significantly reduced [2]. It is possible to restore the balance between the increased load on roads and the quality of bitumen produced by modifying bitumen. To do this, it is necessary to create a structure of the binder material that will be more resistant to external loads and more durable (less prone to aging).

The properties of bitumen are improved in two main directions: compounding at the production stage and modification at the stage of production of commercial products [3, 4].

Compounding is a secondary process for processing bitumen, most often carried out in oil refineries.

Modified bitumens are those improved by the addition of certain substances (polymers, crumb rubber, sulfur, adhesive additives, etc.). The purpose of the modification is not only to ensure that bitumen meets the requirements of GOST, but also to improve its properties by reducing the temperature sensitivity of the binder, i.e. increasing its hardness in summer and decreasing in winter, as well as imparting elasticity to the binder i.e. the ability to undergo reversible deformations over the entire range of operating temperatures. As a result of binder modification, asphalt concrete acquires increased shear resistance, low-temperature crack resistance and fatigue life.

To modify bitumen, a variety of additives are used: thinning, plasticizing, structuring-plasticizing, adhesive, adhesive-structuring, structuring, emulsifiers, etc.

An urgent problem in developed countries of the world can be considered the re-involvement of waste rubber products into industrial production [5,6]. The volume of generation and accumulation of used tires in the world reaches enormous proportions. Tires thrown into landfills or buried decompose under natural conditions for at least 100 years. Contact of tires with rainfall and groundwater is accompanied by the leaching of a number of toxic organic compounds: diphenylamine, dibutyl phthalate, phenanthrene, etc., which enter the soil. In addition, even if rubber is not used, it releases a certain amount of chemicals (up to 100) [7].

At the same time, worn out car tires are a valuable source of secondary raw materials: rubber, carbon black, etc. Worn out tires are valuable secondary raw materials containing around 50% rubber, circa 20% carbon black, etc. [8]. The economic importance of using waste tires is determined by the fact that the extraction of natural resources is becoming increasingly expensive and, in some cases, limited. Recycling used tires will significantly reduce the consumption of some scarce natural resources. Therefore, the use of waste tires is becoming increasingly important. The work of a number of authors is devoted to the method of tire recycling [9-11].

The most environmentally friendly way of disposal is pyrolysis of used tires. Pyrolysis is promising due to the possibility of processing whole tires. In the reactor, the raw material undergoes decomposition at a temperature of approximately 450^0 C, during which the product carbon black is obtained.

Many works have recently been devoted to the issues of pyrolysis of tires and the study of pyrolysis products [5, 12-15].

Of the pyrolysis products suitable for further use, carbon black is of greatest interest. The use of carbon black is promising in various industries.

Our research has identified the possibility of processing carbon black, a powdery residue from the pyrolysis of tires, as a modifying additive for petroleum bitumen.

The purpose of this work was to establish the nature of interactions occurring between the components of carbon black from worn tires and petroleum road bitumen (PRB).

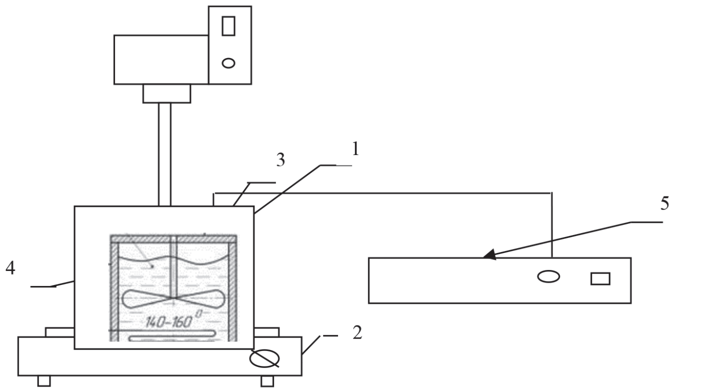
Materials and methods. Petroleum bitumen BND 70/100 of "Kazakhbitum" LLP, Shymkent and BND 50/70 of "Pavlodar Petrochemical Plant" LLP were selected as objects of study.

Carbon black was used as a modifying additive in the work (recycling of old tires by pyrolysis) of "Eco-Shina" LLP, Shymkent.

Identification of the resulting reaction products was carried out using IR spectroscopy on a Shimadzu IR Prestige-21 Fourier transform infrared spectrometer in the wavenumber range 4000-500 cm^{-1}, with a Miracle attenuated total internal reflection (ATR) attachment from Pike Technologies.

Bitumen modification was carried out as follows. A certain volume of bitumen was loaded into a metal glass (1) and heated to a temperature of 140–160°C. When the bitumen melted, the required volume of carbon black from worn tires was added to it, and mixed with a mixer (4) for 40 minutes. The content of the modifying additive (CBWT) in the compositions was 1.0-5.0% by weight (*Figure 7*).

To prepare the bitumen modification, an installation was assembled, the diagram of which is shown in *Figure 1*.



1- metal glass; 2- electric heater; 3- thermometer; 4- mixer; 5- temperature controller

Figure 1 – **Installation diagram for preparing bitumen modification**

IR spectral analysis of the original bitumen and bitumen modified with carbon black was carried out on a Shimadzu IR Prestige-21 IR-Fourier spectrometer with a Miracle attenuated total internal reflection (ATR) attachment from Pike Technologie (*Fig. 2-5*).

After this, the main characteristics were determined according to GOST 33133–2014. The results of the experiments are presented in *tables 2* and *3*.

The main characteristics of the prepared samples of bitumen binder were determined in the work: the heat resistance of bitumen was assessed by the "Ring and Ball" method, the low-temperature properties of bitumen – by the method of determining the brittleness temperature according to Fraas, the hardness index – by the method of determining the depth of penetration of a needle, plasticity was assessed by the method of determining ductility, elastic restoration – by the method of determining elasticity.

Results and discussion. The introduction of carbon black (CBWT) as a modifying additive (*Figure 6*) leads to a significant change in the physical and mechanical properties of bitumen binders (BB). To further study the effect of modification, modified bitumen was obtained by authors. During the research, authors modified bitumen of different grades (BND 70/100 and BND 50/70) with 1-5% carbon black in different contents (*Table 1*):

99%(BND70/100)+1% CBWT
98%(BND70/100)+2% CBWT
97%(BND70/100)+3% CBWT

96%(BND70/100)+4% CBWT
95%(BND70/100)+5% CBWT
99%(BND50/70)+1% CBWT
98%(BND50/70)+2% CBWT
97%(BND50/70)+3% CBWT
96%(BND50/70)+2% CBWT
95%(BND50/70)+1% CBWT

Table 1 – **Composition of modified bitumen (BND 70/100, BND 50/70) with carbon black from worn tires**

No.	Bitumen composition, wt%		Composition of carbon black from worn tires, wt%
	BND 70/100	BND 50/70	
1	100	100	-
2	99	99	1
3	98	98	2
4	97	97	3
5	96	96	4
6	95	95	5

For a comparative analysis of the changes occurring in the group and chemical composition of bitumen during its modification with CBWT, the method of IR spectroscopy was used in this work.

The results of IR spectroscopy of the original (BND 70/100; BND 50/70) and modified with 1-5% CBWT bitumens are presented in *Figures 2-5*. At the same time, in accordance with the methodology for conducting a comparative analysis of the chemical composition of compounds using IR spectroscopy, the obtained spectra were superimposed with scaling by CH_2 groups, the content of which does not depend on the experimental conditions.

It is known that bitumen is a complex organo-mineral system consisting of a large number of saturated aliphatic and aromatic hydrocarbons. *Figure 1* shows the IR spectrum of the original BND 70/100 bitumen without modifying additives. The spectrum is characterized by the presence of bands corresponding to stretching (2916, 2850 cm^{-1}) and bending (1454, 1377 and 721 cm^{-1}) vibrations of CH_2 and CH_3 groups.

Aromatic structures correspond to a band at 1454 cm^{-1} and peaks in the low-frequency region at 867, 810 and 721 cm^{-1}

Figure 3 shows the IR spectrum of the original BND 50/70 bitumen without modifying additives. The spectrum is characterized by the presence of bands corresponding to stretching (2920, 2850 cm^{-1}) and bending (1458, 1377 and 721 cm^{-1}) vibrations of CH_2 and CH_3 groups.

Aromatic structures correspond to a band at 1600, 1458 cm^{-1} and peaks in the low-frequency region at 871, 813 and 721 cm^{-1}

Figure 4 shows the IR spectra of the original and modified with CBWT bitumens: Initial bitumen BND70/100 (curve 1) and 99% (BND70/100) + 1% TUIA (curve 2); 98% (BND70/100) + 2% CBWT (curve 3); 97% (BND70/100) +3% CBWT (curve 4); 96% (BND70/100) + 4% CBWT (curve 5) 95% (BND70/100) + 5% CBWT.

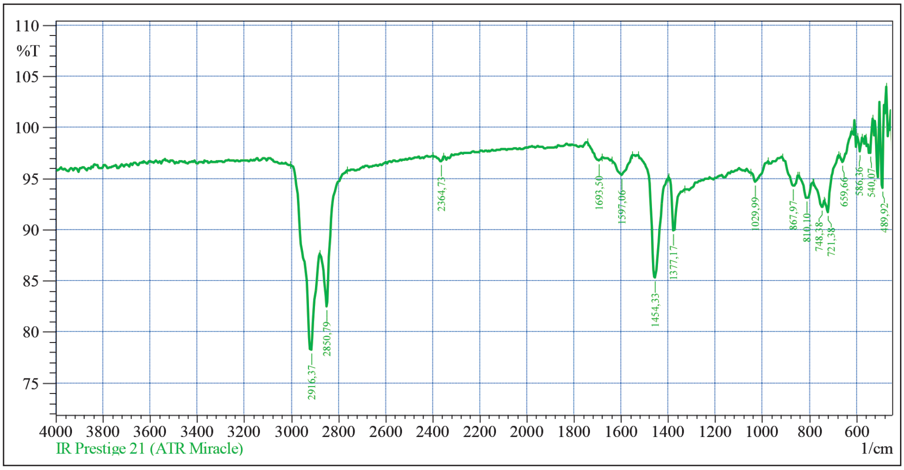


Figure 2 – **IR spectra of the original bitumen BND 70/100**

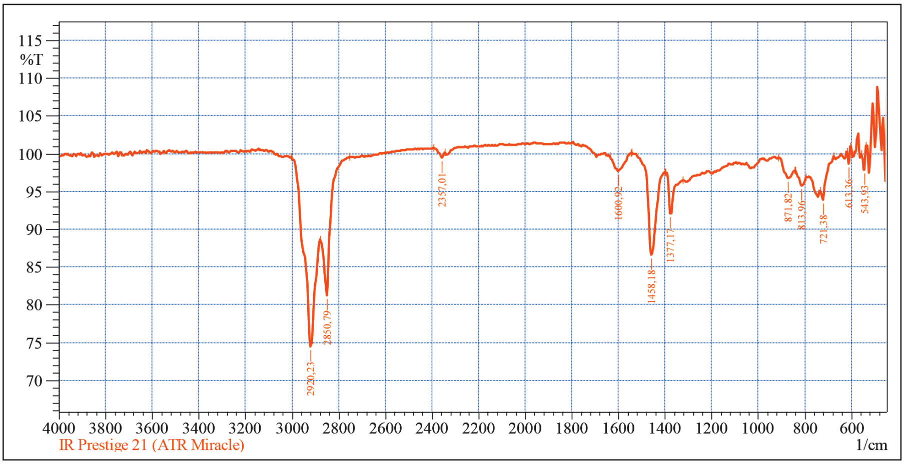


Figure 3 – **IR spectra of the original bitumen BND 50/70**

In the bitumen component of modified binders, both in the case of BND 70/100-CBWT and BND 50/70+CBWT of different contents, a band of 1029 cm^{-1} is distinguished, which corresponds to compounds containing the S=O group (compounds of the R–SO3–type).

2850, 2920 cm^{-1} is the region of stretching vibrations of the CH group. The highest frequency of stretching vibrations νC-H 3300 cm^{-1} belongs to -C≡C-H. Aliphatic hydrocarbons are absorbed in the range of 2800-3000 cm^{-1}, aromatic and unsaturated – about 3100 cm^{-1}. 1458, 1597 cm^{-1} is the region of vibrations of double bonds. The most common and characteristic are vibrations of the carbonyl group. Below 900 cm^{-1}, the region is especially useful for identifying aromatic compounds; it contains bands of C-H bending vibrations in alkenes and benzene derivatives, as well as C-Cl stretching vibrations [16].

Qualitative analysis of the spectra given in *Fig. 4 and 5* demonstrates that the structure of the bitumen component changes during the formation of binders. We can note a significant change in the group composition of aromatic compounds in bitumen. After modifying bitumen grade BND 70/100 with CBWT (*Fig. 4*), peaks appear in the low-frequency region corresponding to an aromatic compound (864, 806, 721, 667) [17].

In the spectrum of the bitumen component, obtained by subtracting the CBWT spectrum from the RBW spectrum, absorption bands related to the paraffin-naphthenic fraction are not observed (for example, at an intensity of 1029 cm^{-1}) [18]).

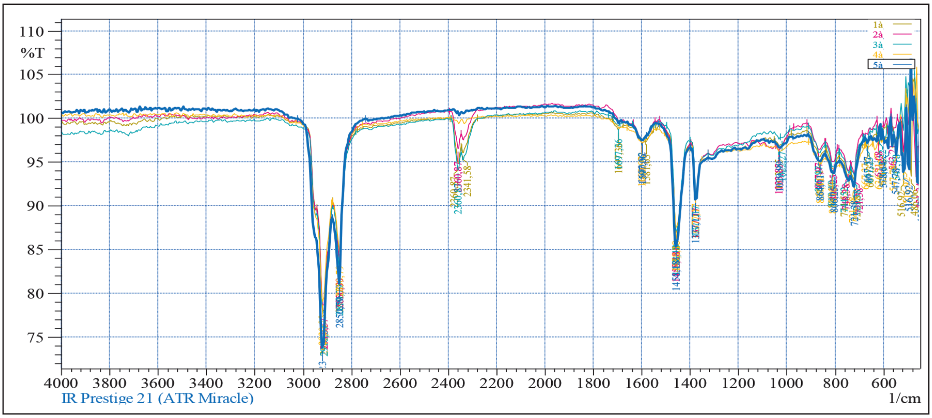


Figure 4 – **IR spectra of the original and modified with CBWT bitumens: Initial bitumen BND 70/100 (curve 1) and 99%(BND70/100)+1% CBWT (curve 2); 98%(BND70/100)+2%CBWT (curve 3); 97%(BND70/100)+3%CBWT (curve 4); 96% (BND70/100) + 4% CBWT (curve 5) 95% (BND70/100) + 5% CBWT (curve 6)**

Figure 5 shows the IR spectra of the original and modified with CBWT bitumen: Initial bitumen BND 50/70 (curve 1) and 99%(BND50/70) + 1%CBWT (curve 2); 98%(BND50/70) + 2%CBWT (curve 3); 97%(BND50/70) + 3%CBWT (curve 4); 96%(BND50/70) + 4%CBWT (curve 5); 95%(BND50/70) + 5%CBWT (curve 6).

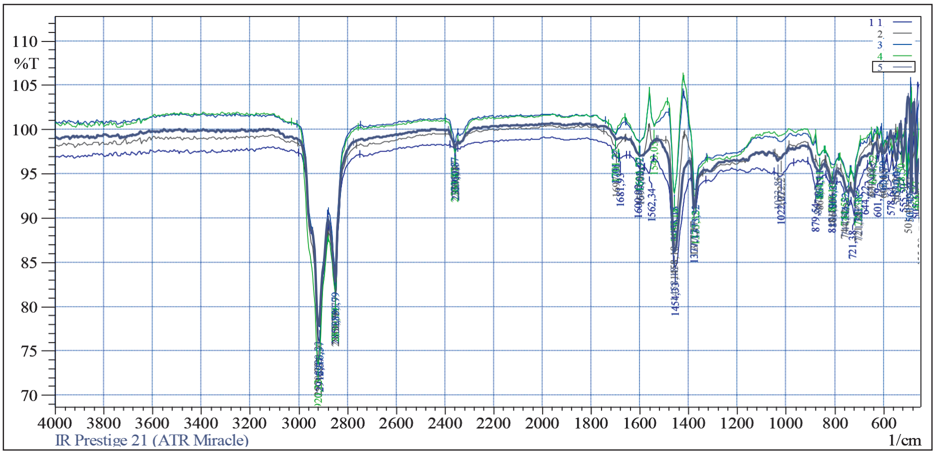


Figure 5 – **IR spectra of the original and modified with CBWT bitumen: Original bitumen BND500/70 (curve 1) and 99%(BND50/70)+1%CBWT (curve 2); 98%(BND50/70)+2%CBWT (curve 3); 97%(BND50/70)+3%CBWT (curve 4); 96%(BND50/70)+4%CBWT (curve 5); 95%(BND50/70)+5%CBWT (curve 6)**

The nature of changes in the structure of BND 50/70 bitumen with CBWT (*Fig. 5*) is similar to the changes caused by the introduction of CBWT. The group composition of paraffin-naphthenic hydrocarbons changes in the region of 1030 cm^{-1}, when this absorption band, present in the spectrum of the original bitumen, degenerates in the spectrum of the bitumen component after modification [18].

When using CBWT, changes are observed in the region of 900–600 cm^{-1}, which relates to aromatic compounds. There is degeneration of the bands of individual aromatic compounds (721, 744, 810 cm^{-1}), as well as bands at intensities of 1604 and 1701 cm^{-1}, characterizing five- and six-membered rings of aromatic compounds [18]. Modification with CBWT leads to the appearance of an absorption band in the region of 1373 cm^{-1} (acetates, phenols).

Studies of the physical and mechanical properties of the original bitumen and the resulting modified with CBWT bitumen were carried out in the laboratory of the Department of Technology of Inorganic and Petrochemical Productions of the M. Auezov SKRU, as well as in the laboratories of the St. Petersburg State Marine Technical University, St. Petersburg, Russian Federation.

The main properties of samples of petroleum bitumen modified with carbon black from worn tires are given in *Tables 2* and *3*.

Here are samples that modified petroleum bitumen BND70/100 with carbon black from worn tires (*Figure 6*): 1-1% CBWT; 2-2%CBWT; 3-3%CBWT; 4-4%CBWT; 5%CBWT, as well as a sample of petroleum bitumen BND 50/70 modified with carbon black from worn tires: 1/-1% CBWT; 2/-2% CBWT; 3/-3% CBWT; 4/-4% CBWT; 5/% CBWT.

Penetration studies were carried out at 25 and 0°C.

Figure 6 – **Pyrolysis carbon black from worn tires**

Figure 7 – **Modified bitumen with carbon black from worn tires**

Table 2 – **Main properties of bitumen binder samples obtained from BND 70/100 bitumen with the addition of carbon black from the crushed mass of worn tires**

No.	Property	BND 70/100	Example No.				
			1	2	3	4	5
1	Softening temperature according to KiSh, °C	not lower than 48	49	50	51	50	59
2	Brittleness temperature, °C	not higher than -20	-21,3	-22	-23,1	-23,2	-24,1
3	Penetration at 25 °C	70/100	65	63	59	55	53
4	Penetration at 0 °C	not lower than 22	21	20	20,1	20,2	20,3
5	Ductility at 25 °C, cm	not less than 75	68	67	65	58	53
6	Ductility at 0 °C, cm	not less than 3,8	3,8	3,6	3,5	3,5	3,4
7	Flash point, °C	not lower than 240	250	240	250	255	256

Table 3 – **Main properties of bitumen binder samples obtained from BND 50/70 bitumen with the addition of carbon black from worn tires**

No.	Property	BND 50/70	Example No.				
			1/	2/	3/	4/	5/
1	Softening temperature according to KiSh, °C	not lower than 50	48	49	50	51	50
2	Brittleness temperature, °C	not higher than -18	-19,3	-20,7	-21,1	-21,6	-22,4
3	Penetration at 25 °C	51-70	66	67	65,8	65,9	66
4	Penetration at 0 °C	not lower than 18	21	21	22	22,1	21,3
5	Ductility at 25 °C, cm	not less than 65	46	47	47	38	26
6	Ductility at 0 °C, cm	not less than 3,5	3,4	3,2	3,0	2,9	2,1
7	Flash point, °C	not lower than 230	243	244	240	241	243

In accordance with the data given in *Tables 2* and *3*, for all samples of bitumen compositions, the softening temperature according to KiSh is higher than that of the original bitumens. Only samples No.1/, 2/ in the amount of additives 1% and 2% CBWT reduces the softening point according to KiSh. One of the most important indicators of low-temperature properties of bitumen is the brittleness temperature. All samples No.1-No.5 are not higher than -20°C, samples No.1/-No.5/ are not higher than -18°C. An increase in the brittleness temperature is observed compared to the original bitumen. Another important indicator of bitumen quality is the level of penetration. The penetration rate at 25°C and 0°C for samples No.1-No.5 decreases, and for samples No.1/-No.5/ increases. Similar dynamics are observed in relation to the ductility indicator. All samples of bitumen binder show a decrease in the elasticity index at 25°C and 0°C compared to the original bitumen, and sample No.1 with the modifying additive CBWT 1% remains stable compared to the original bitumen.

Thus, according to the analysis of the data in *tables 2* and *3*, when carbon black from worn tires is added to the composition of bitumen, the decrease in penetration is observed to a greater extent for bitumen (BND 70/100). The softening temperature increases for bitumen (BND 70/100) and for bitumen (BND 50/70) samples No.3/-5/.

A study of the properties of bitumen has shown that bitumen with high ductility values may not maintain it during operation. Conversely, bitumens that had a ductility value lower than that required by GOST maintained it during the service life of the asphalt concrete pavement; it was also shown that the maximum possible introduction of carbon black from worn tires into BND 50/70 bitumen is less (4% wt.), whereas in the grade BND 70/100 has the ability to introduce carbon black from worn tires up to 5% by weight.

Conclusions. The use of carbon black from worn tires as a modifier of petroleum bitumen reduces the softening point, improves the elasticity of the bitumen binder, and reduces penetration based on BND 70/100 bitumen. In addition, it increases these indicators based on BND 70/100 bitumen, which leads to a change in the grade of the original bitumen, and also improves an important indicator of low-temperature properties i.e. the brittleness temperature.

The introduction of carbon black from worn tires has different effects on different bitumens.

A reasonable amount of additive should be considered to be 4% carbon black from worn tires for BND 50/70, for BND 70/100 up to 5% CBWT.

Thus, it can be stated that it is possible to use carbon black from worn tires as a modifier of petroleum bitumen, which can be very effective in producing asphalt concrete for road surfaces with the best operational and technical characteristics.

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