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COMPARATIVE EVALUATION OF BND 50/70 ROAD BITUMEN MODIFIED WITH SKEPT-60 AND SKN-26 ELASTOMERS**E.A.Guseinova¹, V.A.Mammadova², X.Ch.Abiyev¹**¹*Azerbaijan State Oil and Industry University*²*Heydar Aliyev Oil Refinery*

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The article provides information on the physical and mechanical properties of polymer-bitumen compositions obtained by modifying road bitumen BND 50/70 with elastomers SKEPT-60 and SKN-26 and determination of the obtained polymer-bitumen composites. The properties of composite samples of various concentrations (2.5, 3.0, 5.0%, 6.5 and 7.0%) were determined: needle depth, softening, brittleness and flash point, stretching and adhesion. It was determined that the physical and mechanical characteristics of polymer-bitumen composite materials obtained at high concentrations (6.5, 7.0%) are improved.

Keywords: *modification, bitumen, elastomer, road bitumen, kieselgur, solvent, polymer-bitumen binder, physical and mechanical properties, penetration index.*

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Introduction

Every year, a large amount of material is used around the world for the construction of new roads and repair of existing road surfaces, among which the leading place is asphalt concrete based on bituminous binders. For example, statistical analysis shows that the service life of bitumen-mineral pavements is only 50–70% of the standard.

Different approaches to road bitumen replacement [1, 2] have been considered to improve road surface performance. The most effective is the modification of bitumen with thermoplastic elastomers [3, 27].

The method of replacing petroleum bitumen with polymers has been used for a long time. As early as the 70s and 80s of the last century, a method of replacing bitumen with rubber chips [4] was proposed and the use of the prepared composite ("Brizol" waterproofing material) began. In the last 25–30 years, polymer bitumen composite materials [5] have become widespread. First, because synthetic polymer materials [6] produce hundreds of thousands of tons per year and are more accessible, and second, a number of valuable properties inherent in polymers with such a change (high temperature range, elasticity, low temperature crack resistance, tensile strength,

adhesion, brittleness and s.) passes through bitumen. The properties of bitumen are significantly improved by the incorporation of elastomeric crumbs into the mass or their replacement by polymers. As a rule, polymer additives do not react chemically with bitumen. It dissolves or disintegrates in bitumen, helping to strengthen its structure. As a result, the polymer-bitumen mixture [7–11] acquires a number of valuable physical and mechanical properties specific to the applied polymers and resists aging. Based on them, the road surface can provide cracking resistance, high adhesive resistance, heat resistance, shear resistance and long-term durability at the required temperature, and is therefore more reliable and safe for transport. Thus, the use of polymer additives in road construction reduces the maintenance and repair costs of highways by 30%. It also expands the area of recycling of polymer waste [12]. To date, almost all major industrial polymer types – linear thermoplastics, elastomers (rubbers), thermoplastic elastomers and a number of copolymers have been tested as substitutes for petroleum bitumen. The results are unequal, although in most cases they are positive in terms of improving individual technical characteristics. On average, polymer additives improve the basic properties of bitumen by 1.5–2.0 times.

The task of this work is to determine the technological and physical-mechanical properties of composites based on BND 50/70 road bitumen modified with elastomers SKEPT-60 (ethylene-propylene triple copolymer) and SKN-26 (synthetic butadiene nitrile rubber), as well as the interaction with mineral fillers is to obtain quantitative dependencies.

Experimental part

The following materials were used to make polymer-bitumen composites:

1. BND 50/70 road bitumen meeting the requirements of TC AZ 3536601.242-2015 [13] (H.Aliyev Oil Refinery, Baku, Azerbaijan);
2. SKEPT-60 (TC 2294-022-05766801-2002). Ethylene-propylene rubbers have excellent air and ozone resistance, high heat, oil and abrasion resistance, as well as high air permeability, elasticity, resistance to aggressive environments, good dielectric properties [14–15];
3. SKN-26 (GOST 7738-79). This elastomer has good mechanical properties, high abrasion resistance, high stable friction coefficient and good dynamic properties [14–15];
4. Crushed (medium-sized fraction) soil kieselgur rocks [16, 19] (physical and mechanical indicators: appearance – yellow, medium-sized particles; mass density – 1.56 g/m³; average particle size – 1.5–2.5 mm; special surface area – 1500–2000 sm²/g).

The technology of preparation of bituminous composition (modified bitumen) treated with modifiers is as follows:

1. In this work, BND 50/70 road bitumen was modified with SKEPT-60 elastomer. First, the SKEPT-60 elastomer is cut into small particles and dissolved in benzene or toluene solvent. The dissolution process takes 15–20 hours. During this time, the elastomer is converted into a solid homogeneous solution as a solvent.

The road bitumen to be used is heated to 180–200⁰C to make it fluid. Then polymer-bitumen mixtures of different concentrations (3.0, 6.5%) are prepared using the prepared polymer solution and heated road bitumen. To ensure complete mixing, the process is carried out in a mixing apparatus for 30 min under continuous heating and mixing conditions. The prepared

polymer-bitumen samples are cooled to room temperature.

2. In this work, BND 50/70 road bitumen was modified with SKN-26 elastomer. First, the elastomer is crushed and dissolved in acetone solvent. The dissolving process takes 12–15 hours. During this time, a solid homogeneous solution is obtained as a result of dissolution. The road bitumen to be used is heated to 180–200⁰C to make it fluid. Then polymer-bitumen mixtures of different concentrations (2.5, 5.0, 7.0%) are prepared using the prepared polymer solution and heated road bitumen. As in the previous method, the process is carried out in a mixing apparatus for 30 minutes under continuous heating and mixing conditions to ensure complete mixing. In this case, the solvent (acetone) in the polymer solution evaporates. The prepared polymer-bitumen samples are cooled to room temperature.

The physical and mechanical properties of the prepared bituminous binder samples are appropriate was determined in the laboratory according to the standards. The main quality indicators identified [17] are listed below:

- GOST 11506-73: Petroleum bitumen. The method of the determination of softening point by ring and ball;
- GOST 11501-78: Petroleum bitumen. Method for determination of depth of penetrometer needle penetration;
- GOST 11507-78: Petroleum bitumen. Method for determination of brittle point;
- GOST 11505-75: Petroleum bitumen. Method for determination of ductility;
- GOST 11508-74: Petroleum bitumens. Methods for determination of bitumen adhesion to marble and sand;
- GOST 4333-2014: Petroleum products. Methods for determination of flash and ignition points in open crucible.

Discussion of results

According to the purpose of the research, the physical and mechanical properties of polymer bitumen compositions were studied and the effect of elastomeric components on the properties of bitumen was studied. In the first stage, the effect of application of SEKPT-60

and SKN-26 on the softening temperature of BND 50/70 road bitumen was studied. It is known that the softening point of bitumen is the temperature at which bitumen changes from a solid to a liquid. Softening temperature characterizes the heat resistance of bitumen. High heat-resistant bitumen can be obtained by selecting the appropriate raw material, technological method and mode of production, as well as by adding polymeric materials to bitumen [18, 25].

The results of the addition of elastomers SEKPT-60 and SKN-26 to bitumen are shown in Figure 1.

The softening temperature of the polymer bitumen composite material modified with these elastomers gave a positive result. The softening temperature of bitumen taken for modification is 46–54°C. During modification with SKN-26, the softening temperature reached 49.5°C in a 2.5% sample. These data show an increase in the softening temperature after modification of bitumen with elastomers, especially SKN-26, which is consistent with the known literature [20, 21]. The softening temperature of the composite obtained by modification with SEKPT-60 dropped to 45°C. This is an indicator of the elasticity of the elastomer used.

Another parameter determined in the prepared samples is the penetration depth of the needle. It is known that penetration is an indicator that characterizes the depth of penetration of a standard object into semi-liquid and semi-solid products under a certain regime, which determines the ability of that object to penetrate the product and the resistance of the product to the body. Penetration is a conventional quantity that characterizes both the viscosity and hardness of bitumen.

During the research work, the depth of needle immersion of both bitumen and prepared composite samples was determined at 0 and 25°C. The effect of the composition of the composite on the needle sinking depth is shown in Table 1. Due to the high elastic properties of the SEKPT-60 elastomer used, the elasticity of the prepared composite increases when 6.5% is added to the bitumen, resulting in a significant increase in the needle sinking depth of the com-

posite. This can be clearly seen in Table 1. The table also shows the changes in the needle sinking depth of bitumen as a result of the addition of SKN-26 elastomer to bitumen. The addition of SKN-26 to the bitumen does not cause significant changes in needle sinking. When SKN-26 is added up to 5.0%, the penetration rate of the composite decreases slightly, but the penetration rate increases above it. It should be noted that this increase is very small compared to the sample studied with SEKPT-60. The expected differences can only be due to the different dispersed structure of the compositions: it is assumed that when SEKPT-60 is applied, the volume of the dispersed phase is reduced relative to the original. In general, an increase in the penetration of modified bitumen indicates a slower dissolution of the asphalt mix to be prepared.

One of the standard indicators of the quality of road bitumen is the penetration index, which characterizes their thermal sensitivity. This value characterizes the temperature sensitivity of bituminous binders and is determined by the degree of change in the dispersed state depending on the standard quality indicators – softening temperature and penetration depth of the needle at 25°C. In the first half of the last century, various researchers have proposed a number of methods to calculate the thermal sensitivity of bitumen, which allows to assess the properties of bitumen with temperature changes [22, 23]. The proposed thermal sensitivity index has been widely used, added to regulatory documents specifying bitumen quality indicators and currently remains unchanged in the Ukrainian standard (DSTU 4044-2001) and the CIS interstate standard [17]. It is possible to find the penetration index of bitumen using the penetration index at 0 and 25°C. The temperature sensitivity of bitumen was also determined during this study and is shown in Table 1.

The tensile properties and brittleness of the prepared composites were determined in devices based on modern technology. The corresponding performance of BND 50/70 road bitumen modified with both elastomers is shown in Figure 2 and Table 2.

Fig. 1. Dependence of softening temperature of modified BND 50/70 road bitumen on elastomer concentration.

Table 1. Depth of needle insertion, depending on the composition of the elastomer

The amount of elastomer, % by weight	Needle depth of the composite, 0.1 mm		
	0°C	25°C	Temperature sensitivity, 0°C/25°C
<i>BND 50/70+SKEPT-60</i>			
0	57	11	19.3
3.0	57	6	10.5
6.5	95	8	8.4
<i>BND 50/70+SKN-26</i>			
0	57	11	19.3
2.5	53	1	1.9
5.0	56	4	7.1
7.0	64	7	10.9

a)

b)

Fig. 2. Dependence of tensile properties of modified bitumen on the composition of elastomers SKN-26 (a) and SKEPT-60 (b).

Tension depends on the composition of the bitumen. As a result of the addition of elastomers to bitumen, the tension increases, the bitumen becomes more elastic, which is of great importance in the use of road construction. Tensile condition is a technical indicator, which indicates the degree of softness of bitumen, along with the softening temperature and needle penetration. The longer and thinner the ropes during the determination of tensile strength, the better the bitumen withstands mechanical stress, has better elastic properties, adhesion and bending ability. Picture 2 shows that the addition of SKEPT-60 elastomer at a concentration of 6.5% to bitumen further increases the tensile properties of modified road bitumen, it is explained that under the given conditions, elastomer forms a new "gel-structure" [24] that stabilizes asphalt-tenes. As a result, it allows to obtain road bitumen with high elastic properties.

Brittleness temperature characterizes the behavior of bitumen on the road surface. The lower the brittleness temperature, the higher the quality of the road bitumen. Oxidized bitumen has a lower brittle point than other bitumen. The brittle temperature of elastomer-modified road bitumen is shown in Table 2.

Table 2. The effect of elastomers SKN-26 and SKEPT-60 on the brittleness temperature of bitumen

The amount of elastomer, % by weight	The brittleness temperature of the composite, °C
0*	Negative 30.0
2.5	Negative 30.4
3.0	Negative 30.5
5.0	Negative 30.7
6.5	Negative 30.9
7.0	Negative 31.0

0*- Initial brittleness temperature of BND 50/70 road bitumen, °C.

It can be seen in Table 2 that in this research method, as a result of the addition of a small amount of elastomer to the road bitumen, there was a certain change in the brittleness temperature of the bitumen. As a result, the brittleness temperature of bitumen increased in the range of 0.5–1°C.

Figure 3 shows the determination of the flash point of the modified road bitumen. The flash point for road bitumen is not a key indicator, it does not determine quality, but it has been standardized to characterize fire hazard characteristics.

The flash point of modified road bitumen is slightly lower than the standards (BND 50/70 road bitumen flash point [13] should not be less than 230°C), but it provides safe conditions for the application of bitumen in road construction.

Fig. 3. Dependence of ignition temperature of modified BND 50/70 road bitumen on elastomer concentration.

The determination of the flash point is related to the fire safety requirements when using bituminous binders [26] and allows to determine the content of flammable, volatile substances. In the next step, the adhesion properties of the modified road bitumen samples with different elastomer composition were investigated. The chemical composition of the prepared composite samples is shown in Table 3.

Table 3. Composition of modified road bitumen

Components name	Sample number					
	1	2	3	4	5	6
Bitumen BND 50/70, %	100	97.5	95.0	93.0	97.0	93.5
SKN-26, %	–	2.5	5.0	7.0	–	–
SKEPT-60, %	–	–	–	–	3.0	6.5

Adhesion of road bitumen to the surface of mineral particles is of great importance to ensure the basic properties of asphalt concrete. For this reason, along with the improvement of quality indicators, the preparation of bituminous binders

that increase the adhesion properties of the mineral material should be the basis of the work.

Figure 4 shows the adhesion of BND 50/70 road bitumen modified with elastomers to the mineral material. It is known that quantitative and qualitative methods are considered and their comparative evaluation is carried out to assess the adhesion of road bitumen to mineral materials. Interactions are taken into account during the formation of the bitumen-mineral material interface.

In the study, the indicators of adhesion of road bitumen with crushed soil kieselgur rocks and sand additives were determined. The adhesion indices determined by the quality method were compared according to GOST 11508-74. As a result, the conformity of the modified road bitumen sample to "Control sample №2" (at least $\frac{3}{4}$ surface coating with mineral material) was determined.

a) Adhesion of road bitumen modified with SKN-26 elastomer to mineral material.

b) Adhesion of road bitumen modified with SKEPT-60 elastomer to mineral material.

Fig. 4. Adhesion of the modified bitumen composition to the mineral material.

Conclusion

Although bitumen of the same brand was taken as part of the initial composition, the added elastomers had a different effect, and the resulting samples differed in their performance

characteristics. As a result of the research it was determined that;

- Made on the basis of BND 50/70 road bitumen and SKN-26 elastomer the softening and brittleness temperature of the composition, the depth of needle sinking increased. This in-

crease is more pronounced in the 7.0% sample. The softening temperature of the composite prepared at this concentration increased to 49.5°C. On the contrary, a decrease in the tensile property was observed. Future research will include a more in-depth study and explanatory study of this property.

– With the addition of SKEPT-60 elastomer to BND 50/70 road bitumen, the elasticity was further increased and as a result, the main quality indicators such as needle sinking depth, brittleness temperature and tensile properties were increased. As a result of the addition of 6.5% SKEPT-60 elastomer to bitumen, the depth of needle penetration of the composite was even doubled. The softening temperature of the composite, on the other hand, decreased to 45°C. This is due to the high elastic properties of the elastomer.

The adhesion of each composite to the mineral material was determined in accordance with GOST 11508-74. It was found that at least $\frac{3}{4}$ part of the prepared composites was covered with mineral material.

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**SKEPT-60 VƏ SKN-26 ELASTOMERLƏRİ İLƏ MODİFİKASIYA OLUNMUŞ BND 50/70 YOL
BİTUMUNUN GÖSTƏRİCİLƏRİNİN MÜQAYİSƏLİ QIYMƏTLƏNDİRİLMƏSİ****E.Ə.Hüseynova, V.A.Məmmədova, X.Ç.Abiyev**

Məqalədə BND 50/70 yol bitumunun SKEPT-60 və SKN-26 elastomerləri ilə modifikasiyası və hazırlanmış polimer bitum kompozitlərin fiziki-mexaniki xassələrinin təyini haqqında məlumat verilmişdir. Müxtəlif qatılıqlı kompozit nümunələrin bir sıra xüsusiyyətləri (2.5, 3.0, 5.0, 6.5 və 7.0%) müəyyən edilmişdir: iynə batma dərinliyi, yumşalma, kövrəklik və alışma temperaturu, dartılma və yapışma qabiliyyəti. Müəyyən olunmuşdur ki, yüksək qatılıqlarda (6.5, 7.0%) hazırlanmış polimer bitum kompozisiya materiallarının fiziki-mexaniki göstəriciləri yüksəlmişdir.

Açar sözlər: *modifikasiya, bitum, elastomer, yol bitumu, kizelqur, həlledici, polimer-bitum bağlayıcı, fiziki və mexaniki xassələr, penetrasiya indeksi.*

**СРАВНИТЕЛЬНАЯ ОЦЕНКА МОДИФИЦИРОВАННОГО
ЭЛАСТОМЕРАМИ СКЭПТ-60 И СКН-26 ДОРОЖНОГО БИТУМА БНД 50/70****Э.А.Гусейнова, В.А.Мамедова, Х.Ч.Абиев**

В статье представлены данные о физико-механических свойствах полимер-битумных композиций, полученных модифицированием дорожного битума БНД 50/70 эластомерами СКЭПТ-60 и СКН-26. Были определены свойства композитных образцов различной концентрации (2.5, 3.0, 5.0, 6.5 и 7.0%): глубина проникновения иглы, температура размягчения, температура хрупкости и воспламенения, показатели растяжения и адгезия. Было определено, что физико-механические характеристики полимерно-битумных композиционных материалов, полученных при высоких концентрациях (6.5, 7.0%) улучшаются.

Ключевые слова: *модификация, битум, эластомер, дорожный битум, кизельгур, растворитель, полимерно-битумное вяжущее, физико-механические свойства, индекс пенетрации.*