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Research Article

Modified Bitumen Materials from Kazakhstani Oilfield

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The oil bitumen BND 90/130, produced at the “LLP SP Caspi Bitum” with the modifier, which consists of copolymer of ethylene with butyl acrylate and glycidyl methacrylate taken in an amount of 0.5–1.6 wt%, and the tire reclaim (4–20 wt%), which is the destructate of mesh elastomers of different chemical nature, was modified; possibility of using the developed bitumen-elastomer binders in road asphalt concrete was justified. Modification of bitumen with a copolymer of ethylene with butyl acrylate and glycidyl methacrylate leads to an improvement in the properties of road bitumen: the softening point, hardness, deformability at low temperatures, elasticity, and adhesion to metal and mineral filler increase. It was shown that ethylene with butyl acrylate and glycidyl methacrylate chemically interacts with the functional groups of bitumen asphaltenes through the epoxy group of glycidyl methacrylate. Analysis of the spectra and group composition indicates an increased content of high molecular weight asphaltenes in the modified bitumen with a slight increase in structuring resins. It has been established that bitumen modified with rubber crumbs of 0.6–1.0 mm in size has high elasticity. The most effective composition of a bitumen-regenerated composite material based on tire reclaim has been determined. In terms of the totality of physicochemical and operational characteristics and comparative cost, the most acceptable is the bitumen-regenerated composition (with a regenerate content of 20%) and is superior in the complex of properties to bitumen modified with an optimal content of ethylene with butyl acrylate and glycidyl methacrylate (1.6%). The technology for modifying bitumen with tire reclaim is less time-consuming, more economically profitable, and environmentally effective, since it utilizes large-tonnage waste of worn-out tires. The resulting bitumen-polymer compositions have a high positive set of properties: softening point, hardness, elasticity, frost resistance, and low-temperature characteristics.

1. Introduction

From the world practice of bitumen production, it is known that the nature and composition of raw materials are the fundamental factors for ensuring high-quality characteristics of the obtained bitumen. For this reason, at the leading foreign enterprises producing bitumen products, the correct choice of the source oil is of paramount importance. Oils intended for the production of bitumen are not mixed with oils intended for the production of fuels [1].

Currently, there are new increased requirements for materials used in road and industrial civil construction, housing, and communal services. Therefore, it is important to find reliable raw materials for the production of bitumen [2, 3] to improve the production technologies for bitumen and materials of improved quality based on them.

However, even with the correct selection of raw materials, bitumen obtained using any currently available technologies in terms of quality indicators does not always meet the requirements for modern road bitumen. Unmodified bitumen has a number of disadvantages that negatively affect the quality of asphalt concrete materials and, accordingly, road surfaces: high thermal sensitivity (increased plastic deformation at summer temperatures and brittleness at winter temperatures), poor mechanical characteristics, and low elasticity, susceptibility to aging.

From this point of view and taking into account economic factors in the road industry, bitumen binders are increasingly used, modified by-products and wastes of the chemical and petrochemical industries: sulfur, organo-manganese compounds, rubber crumbs, fibrous fillers, petroleum polymers, indene-coumarone, and other resins, liquid rubbers such as divinyl acrylonitrile, divinyl carboxylate, and others, various high molecular weight polymers [4–7], microencapsulated materials with a phase transition, synthesized with various shell materials using the emulsion polymerization method [8].

Today, the modification of bitumen with polymers is recognized as a necessary technological method for obtaining highly efficient road construction materials based on this main organic binder [9]. To achieve this goal, a lot of research have been conducted [10–16]. The first mention of the use of polymers (natural rubber) to improve the properties of bitumen dates back to the last century [17].

To date, almost all types of polymers (linear thermoplastics, elastomers, thermoplastic elastomers, and a number of oligomers (epoxy, furan, and other resins)) have been tested and often successfully used as oil bitumen modifiers. Of greatest interest is the use of elastomers and synthetic thermoplastic elastomers, since they have high deformation characteristics at low temperatures and transfer these properties to bitumen [18].

Nowadays, there are two main approaches to bitumen modification. Until recently, rubbers and thermoplastic elastomers were considered the best bitumen modifiers, which, without chemically interacting with bitumen, dissolved in it, giving the properties of a polymer. However, even the best bitumen-polymer compositions of this type have some disadvantages. For example, the most popular bitumen modifiers—SBS block copolymers, when introduced into bitumen, do not solve the problem of its susceptibility to atmospheric aging due to the large number of double bonds in the main chain. A common disadvantage of such compositions is their delamination under the influence of differences in the densities of bitumen and polymers.

The second promising approach is the modification of bitumen with reactive additives. Such modifiers can be introduced in smaller quantities, and the resulting compositions do not separate.

The high relative cost of thermoplastic elastomers and rubbers hinders their widespread use to improve the properties of bitumens. In this regard, of great practical interest is the waste of rubber products, the vast majority of which are worn-out car tires [19]. Tires fail during operation due to mechanical wear of the tread, delamination, and rupture of parts. At the same time, rubber, as a structural material, undergoes only minor changes in the topological and chemical structures by the time the products are out of service, which is facilitated by the presence of an inhibitor in it that inhibits the development of the oxidation process, which underlies the aging of rubber [20].

Direct modification of bitumen with rubber crumb is ineffective, since it behaves in bitumen mainly as a filler, i.e., swells, forming separate centers of elasticity, but does not create a polymer network in bitumen; that is, it does not fully show its polymer properties in the latter. Therefore, in order to realize the properties of the polymer in bitumen, crumb rubber must be devulcanized, turning the network polymer into a linear one and, therefore, having the ability to dissolve. For this purpose, we used tire reclaim as a component of the bitumen modifier [21].

The study was carried out on the modification of petroleum bitumen grade BND 90/130, produced at the LPP SP "Caspi Bitum" with the modifier "EBG," which is a copolymer of ethylene with butyl acrylate and glycidyl methacrylate, rubber crumb and tire reclaim, which is the destructor of network elastomers of different chemical nature, substantiation of the possibility of use developed bitumen-elastomer binders in road asphalt concrete [22–25].

In this work, petroleum bitumen grade BND 90/130, produced at the LPP SP "Caspi Bitum," was modified with the modifier "EBG," which is a copolymer of ethylene with butyl acrylate and glycidyl methacrylate, rubber crumb, and tire reclaim (a destructor of network elastomers of different chemical natures). A comparative characteristic was carried out between studied modifiers on the impact on the quality indicators of the resulting bitumen and availability, substantiation of the possibility of using the developed bitumen-elastomer binders in road asphalt concrete.

The cheapest and perhaps most promising elastomeric bitumen modifiers are crumb rubber and tire reclaim. Crumb rubber is a collection of particles of crushed rubber of various dispersion and various shapes, which are characterized primarily by the fact that they fundamentally retain the molecular structure and elastomeric properties of the original rubber, and the surface of the particles can be activated to impart special properties to the crumb rubber, or by partial devulcanization at the surface layer of particles, or modification of the surface of particles by chemical or physicochemical treatment [26].

Crumb rubber is one of the products of processing secondary rubber raw materials (rubber waste, including old tires). The main raw material for producing crumb rubber should be considered worn tires, since more than half of the rubber produced in the world is used in the production of tires [27]. It was noted [28] that partial swelling and dissolution of old regenerated rubber begins after 2 hr of heating with continuous stirring. Surmeli et al. [29], based on microstructural analysis data, combine reclaim and bitumen at a temperature of 200°C for 0.5 hr. It also showed that adding old vulcanized rubber with a high sulfur content can lead to the opposite result, i.e., hardening and significantly increasing the brittleness of the bitumen. Prolonged heating at high temperatures negatively affects the properties of bitumen.

Unlike polymer-bitumen composites [30], with prolonged mixing, the uniform distribution of rubber particles in the bitumen-regenerated composition is not the resulting thermodynamically equilibrium stage of structure formation in the material [31].

2. Materials and Methods

The object of study is the oil bitumen BND 90/130 (ST RK 1373-2005) "LLP SP Caspi Bitum," Aktau, Kazakhstan.

Table 1: Group composition of bitumen.

Bitumen grade	Asphaltenes (%)	Resins (%)	Oils (%)	Saturated aromatics (%)
BND-90/130	21.9	30.5	7.18	40.42

Table 2: Characteristics of bitumen.

Bitumen grade BND-90/130	Softening temperature (°C)	Penetration, 0.1 mm		Ductility (cm)		Elasticity at 25°C (%)
		25°C	0°C	25°C	0°C	
Sample	44	97	50	95	0	13
According to the norms of ST RK 1373–2005; GOST 22245-90	No less than 43	91–130	No less than 28	No less than 65	4.0	—

Figure 1: Tire reclaim.

Figure 2: EBG.

The composition of oil bitumen was determined using the adsorption liquid chromatography method on a Jatroscan MK-5 instrument. The component composition was determined according to the standard method [32].

The compositions and main technical characteristics of bitumen are given in Tables 1 and 2.

IR spectral analysis of the original bitumen and bitumen modified with 2% EBG (a copolymer of ethylene with butyl acrylate and glycidyl methacrylate) was carried out on a Shimadzu IR Prestige-21FT-IR spectrometer with a miracle attenuated total internal reflection attachment from Pike Technologie (Figure 1).

The temperature of softening (T_s) is determined by the Ring and Ball Apparatus (GOST 11506). The essence of the method is to determine the temperature at which bitumen, located in a ring of given dimensions, softens under test conditions and, moving under the action of a steel ball, reaches the bottom plate.

Penetration (P) is determined according to GOST 11501, characterizes the hardness of bitumen and is defined as the depth of immersion (penetration) of a calibrated needle with a diameter of 1 mm into a bitumen sample under the action of a certain load for a given time at a fixed temperature. It is measured in tenths of mm.

Ductility (D) is the ability of bitumen to stretch into a thread. It is defined as the length of the thread formed by the moment of rupture at fixed loads and a temperature of 25°C (D_{25}), 0°C (D_0) (GOST 11505).

The sample is a double spatula; the initial thickness of the neck is 1 cm, and the length is 3 cm.

Elasticity (E) characterizes the ability of bitumen to undergo reversible deformations and is determined by reducing the length of the sample, prestretched to rupture, after determining ductility.

Asphaltenes were precipitated from bitumen with a 40-fold excess of petroleum ether (35–75°C). The separation of the deasphalted oil into oils and resins was carried out by column chromatography using solvents: petroleum ether + carbon tetrachloride, benzene, and alcohol-benzene.

2.1. Modifying Additives

(1) EBG bitumen modifier is a colorless transparent granule and is a copolymer of ethylene with butyl acrylate and glycidyl methacrylate (Figure 2).

(2) Tire reclaim (Figure 1), which is a destructor of network elastomers of different chemical nature; the

Figure 3: Crumb rubber.

process of obtaining tire reclaim from tire crumbs includes the operations of mixing ingredients in a mixer of bulk components, devulcanization of rubber in a cam extruder and on rollers at a temperature not exceeding 100°C. To obtain tire reclaim, tire crumbs up to 2.0 mm in size are used, obtained by barodestruction grinding. A distinctive feature of such crumbs is its developed surface, which increases the susceptibility of the crumbs to the effects of devulcanizing factors [18].

(3) Rubber crumbs with dimensions of 0.6–1.0 mm (Figure 3).

When obtaining tire reclaim on roller equipment, crumb rubber was pre-mixed with a softener. Soapstock waste from the oil and fat industry was used as a softener [21].

Bitumen modification was carried out as follows: a certain volume of BND 90/130-grade bitumen (100 pts.wt.) was loaded into a metal glass (1) and heated to a temperature of 140–175°C. When the bitumen was melted, the required volume of tire reclaim or crumb rubber was added to it and, with the mixer (4), was mixed for 2 hr at a rotor speed of 100 rpm.

To prepare the bitumen modification, an installation was assembled, the diagram of which is shown in Figure 4.

3. Results and Discussion

In this paper, the possibility and effectiveness of the modification of petroleum bitumen with the complex additive EBG, obtained at the Department of "Technology of inorganic and petrochemical industries," was studied. EBG copolymer expands the operating temperature range, gives bitumen elasticity, and increases bitumen adhesion (Figure 5).

IR spectroscopy was used for a comparative analysis of the changes that occur in the group and the chemical composition of bitumen when it is modified with EBG.

The results of IR spectroscopy of the original bitumen and modified bitumen with 2% EBG are shown in Figure 6 and Table 3. In accordance with the procedure for conducting a comparative analysis of the chemical composition of compounds using IR spectroscopy, the obtained spectra were superimposed with scaling over CH_2-groups, the content of which does not depend on the conditions of the experiment.

The analysis of the obtained results of BND 90/130 bitumen studies showed the presence of intense bands is typical for bitumen in the region of 3,000–2,800 cm^{-1} (stretching vibrations of ν (CH) and CH_2-groups), 1,458 cm^{-1} (deformation vibrations of δ (CH_2)) and 1,377 cm^{-1} (deformation vibrations of δ (CH_3)). These bands are always present in the spectra of saturated hydrocarbons, paraffins, and oils [33]. In the spectra of the components, a transmission band at 721 cm^{-1} is clearly visible, which corresponds to the bending vibrations of δ (CH_2) groups in free paraffin chains. The characteristic triplet 744, 810, and 871 cm^{-1} is clearly manifested as a sign of the presence of aromatic structures.

The transmission bands in the region of 1,660–1,580 cm^{-1} are characterized by a much greater intensity, indicating the presence of oxygen-containing compounds. The band at about 1,585 cm^{-1} characterizes stretching vibrations of unsaturated C=C bonds, mainly of cyclic structure and, first of all, benzene rings. The large half-width and complex structure of this band indicates a wide composition distribution of aromatic compounds (asphaltenes) in bitumen. In the region of 1,685 cm^{-1}, there are bands of carbonyl and carboxyl C=O groups that appear during the oxidation of organic compounds.

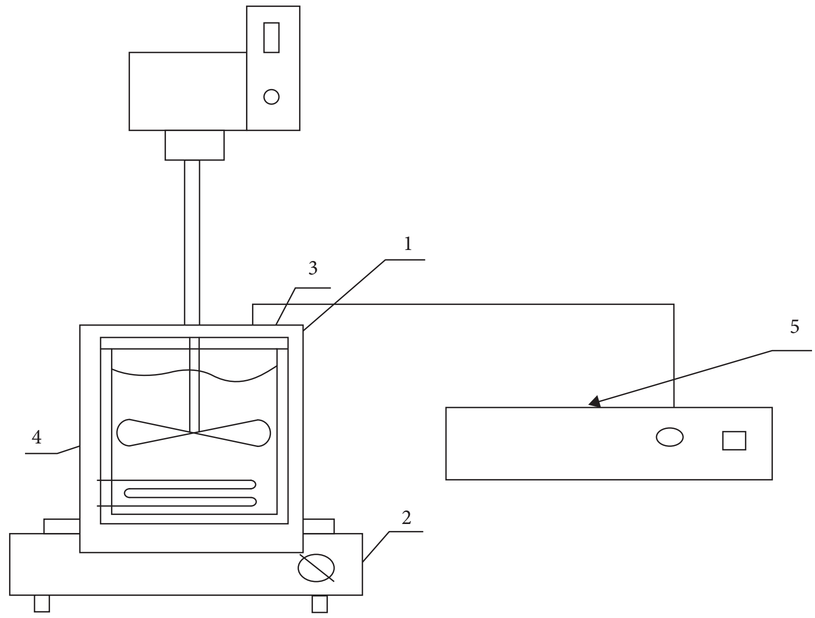
The most informative from the point of view of comparative analysis are as follows:

(1) High-frequency peak 3,344 cm^{-1} (in the original bitumen). The frequency and small half-width of this peak unambiguously allow us to attribute it to the stretching vibrations of OH hydroxyl groups that are not associated with any type of hydrogen bond [34]. Modification of bitumen [35] by EBG leads to changes in the frequency of valence ν (OH) in the high-frequency part: the intensity increases, and the maximum shifts from 3,344 cm^{-1} (bitumen spectrum) to 3,440 cm^{-1} (modified bitumen). This change is associated with some rearrangement in the structure of the hydrogen bonds of bitumen when EBG is introduced into it.

(2) A characteristic absorption band of the carbonyl group, which in the spectrum of the modified bitumen has a greater intensity than in the spectrum of the original bitumen and shifts from 1,689 to 1,695 cm^{-1}.

The analysis of the given spectra indicates an increased content of high-molecular-weight asphaltenes in the modified bitumen with a slight increase in structuring resins, since there is an increase in the absorption bands of the carbonyl group at 1,685 cm^{-1} (shift in the modified bitumen up to 1,695 cm^{-1}) and aromatic rings at 1,602 cm^{-1}. In addition, when interacting with the modifier in bitumen, the content of the oil fraction decreases, in particular, paraffin-naphthenic hydrocarbons, characterized by paraffin chains with a spectral band at 720 cm^{-1}

The group composition of modified bitumen is given in Table 4.



1—metal glass; 2—electric heater; 3—thermometer; 4—mixer; 5—temperature regulator

Figure 4: Installation diagram for preparing bitumen modification.

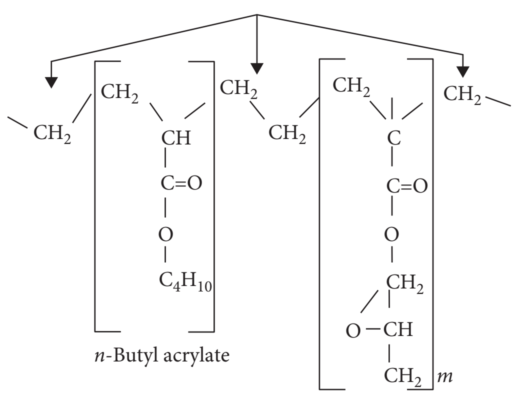


Figure 5: Structural formula of EBG.

A decrease in the concentration of paraffin-naphthenic hydrocarbons in bitumen leads to an increase in the lyophilicity of asphaltenes, which solvate and swell in aromatic hydrocarbons and are insoluble in paraffin-naphthenic hydrocarbons. Such bitumen differs in that asphaltenes can interact with their polar (lyophobic) surface areas, forming aggregates and nuclei of the coagulation structure, and resins are oriented adsorbed on the lyophilic outer side of asphaltenes.

Thus, when bitumen is modified with a copolymer of ethylene with butyl acrylate and glycidyl methacrylate, a chemical interaction of the carboxyl groups of bitumen asphaltenes with the epoxy groups of glycidyl methacrylate actually takes place, which leads to the production of a homogeneous material.

The physical and mechanical properties of BND 90/130 bitumen were studied, depending on the concentration of the EBG modifier in them: 0.5%, 0.8%, 1%, and 1.6%. The components were mixed for 2 hr at a temperature of 175°C, after which the mixture was kept at the same temperature for another 4 hr for a more complete chemical interaction of the bitumen with the polymer, after which a homogeneous mass was obtained.

Figure 7 shows the dependence of the temperature of softening (T_s) of road bitumen (1) on the concentration of the EBG modifier in it, as well as road bitumen on the concentration of the tire reclaim (TR) in it (2) and crumb rubber (CR) [21, 36].

As can be seen, curve 1 increases equidistantly, which indicates the similarity of the mechanism of action of the EBG additive in bitumen. The softening temperature of road bitumen increases by 20°C. The maximum possible concentration of EBG is regulated by the manufacturer at 1.6%, which is explained by the significantly increasing viscosity of the composition and the high price of the modifier. Comparing curves 1, 2, and 3, we can conclude that at the low concentrations of the modifier (1.6%), EBG is a more effective additive, and the optimal composition with reclaim and crumb rubber (20%) has a softening temperature of 12°C higher and, given the low-cost reclaim and crumb rubber, is economically more profitable.

Penetration at 25°C (P_{25}) of modified bitumens (Figure 8) decreases in all cases, which indicates an increase in the hardness of the compositions with an increase in the additive content in them. Here, the road bitumen curve (1) is from 98 to 59. Modification of bitumen with the resulting tire reclaim and devulcanized rubber crumb also leads to an increase in the hardness of the binder.

From the presented data on T_s and penetration, there is a clear correlation between these indicators of the properties of the obtained bitumen-polymer compositions, i.e., as the hardness of bitumen increases, its softening point increases.

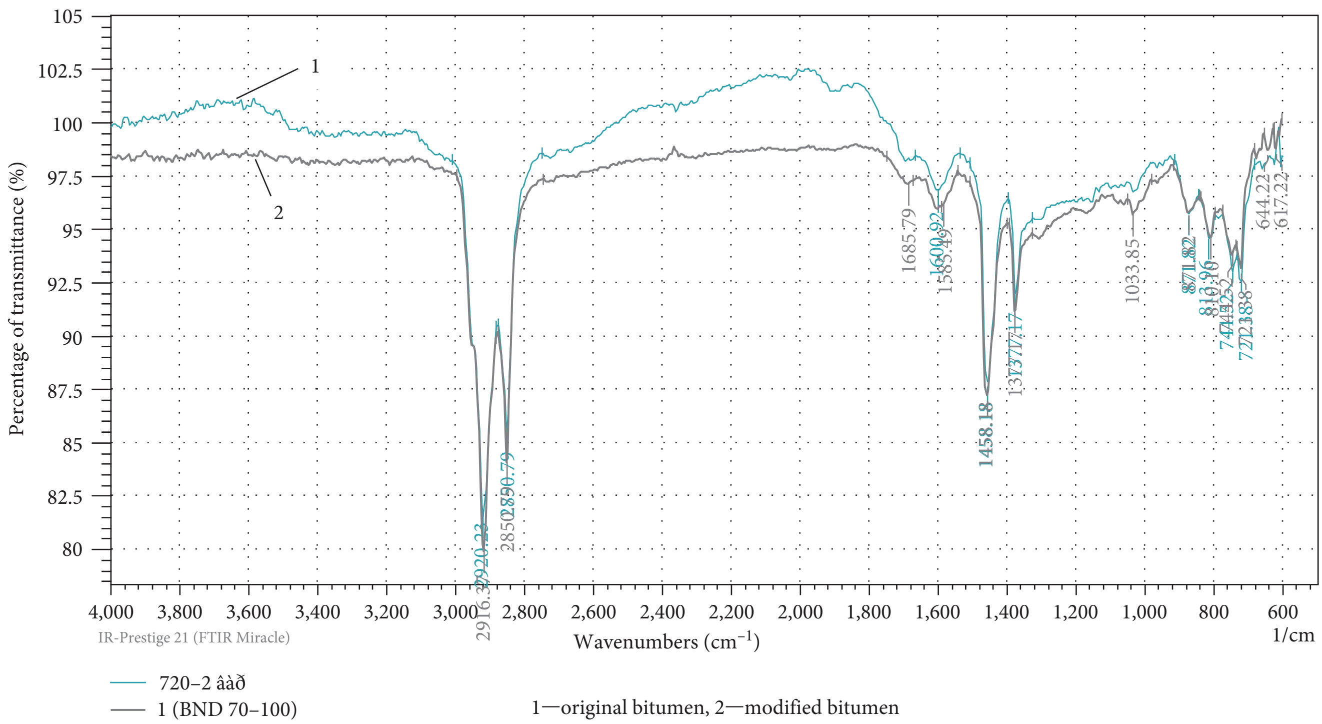


Figure 6: IR spectra of the original bitumen and bitumen modified with 2% EBG.

Table 3: Experimental frequencies ν (cm^{-1}) and optical densities of some bands in the IR spectra in the region of 500–4,000 cm^{-1}.

Frequencies ν (cm^{-1})	Optical density	Assignment
	BND 90/130	
1,688	20	ν (C═O)
1,592	57	ν (CCar)
	BND 90/130 + 2% EBG	
1,685	30	ν (C═O)
1,585	52	ν (CCar)

Table 4: Group composition of modified bitumen.

Composition	Asphaltenes (%)	Resins (%)	Oils (%)	Saturated aromatics (%)
BND-90/130 + 2% EBG	29.5	33.1	5.24	32.16

The decrease in ductility (D_{25}) (Figure 9) of road bitumen (1, 2, 3) with the introduction of polymers into them is a well-known natural effect for low-viscosity bitumen.

The elasticity of bitumen at 25°C (Figure 10) with an increase in the concentration of modifiers in them in all cases increases up to 51% for road bitumen (1), with an EBG concentration of 1.6%. The elasticity of the optimal bitumen-reclaimed tire composition (2) is significantly higher and amounts to 83% when it contains 20% reclaimed material, and the optimal bitumen-crumb rubber composition (3) is 80%.

The given data show that the modification of road bitumen with a copolymer of ethylene with butyl acrylate and glycidyl methacrylate is effective at its low concentrations, which is explained by its chemical interaction with bitumen asphaltenes. However, the optimal bitumen-reclaimed compositions in terms of a set of properties (softening temperature, hardness, elasticity) are significantly superior to binders modified with EBG. In addition, the technology of modifying bitumen with the obtained tire reclaim and crumb rubber [21] is less time-consuming, economically more profitable, and environmentally efficient, since the large-tonnage waste of worn-out tires is utilized in this case. The results of the conducted studies are presented in Table 5.

Nevertheless, the possibility of chemical interaction of EBG with bitumen asphaltenes makes it possible to effectively modify high-viscosity bitumens with a high content of asphaltenes with this polymer, where elastomers are ineffective and are practically not used. In addition to the

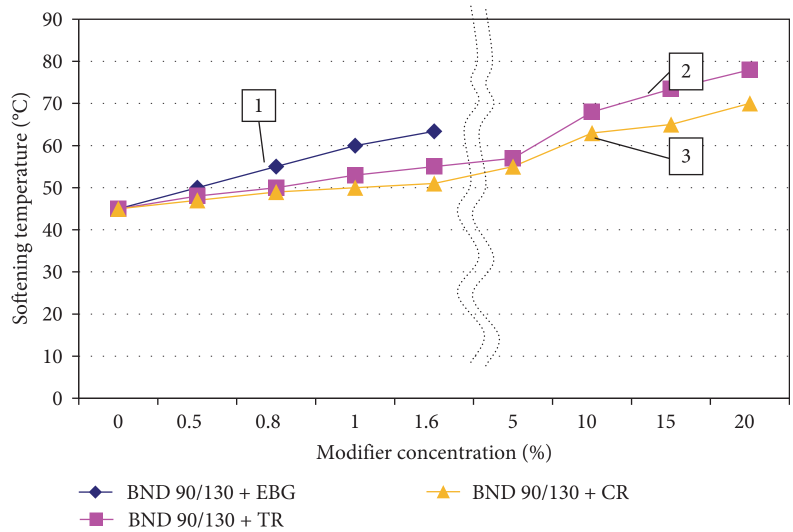


Figure 7: Dependence of the softening temperature of bitumen-polymer compositions on the concentration of modifiers.

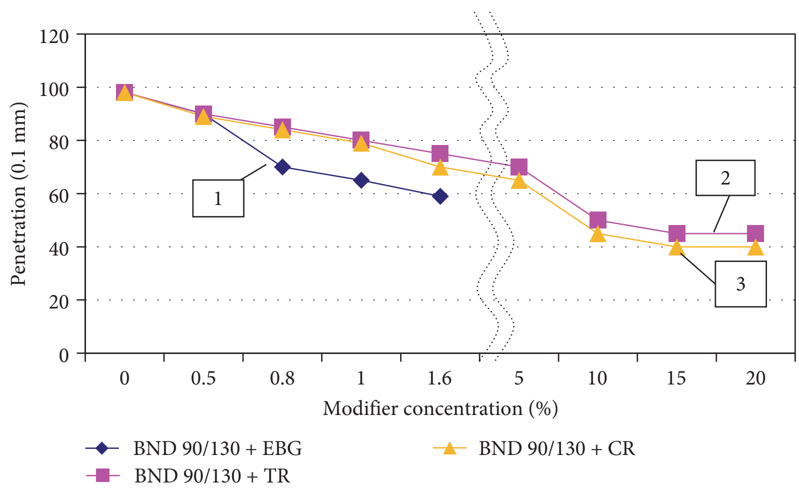


Figure 8: Dependence of the penetration of bitumen-polymer compositions at 25°C on the concentration of modifiers.

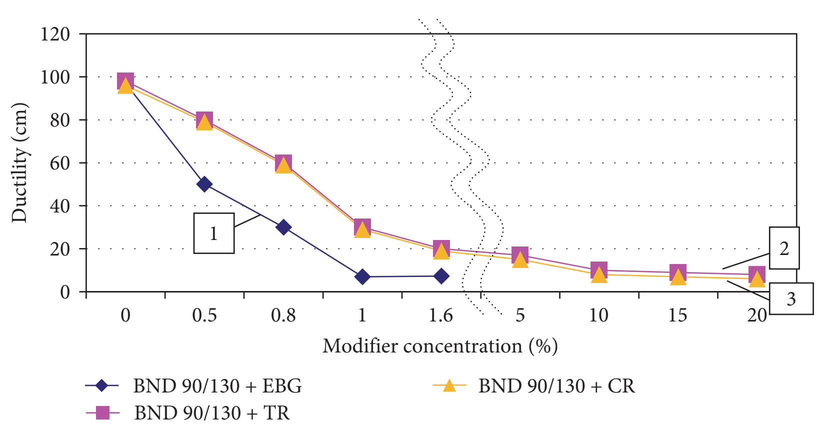


Figure 9: Ductility dependence of bitumen-polymer compositions at 25°C from the concentration of modifiers.

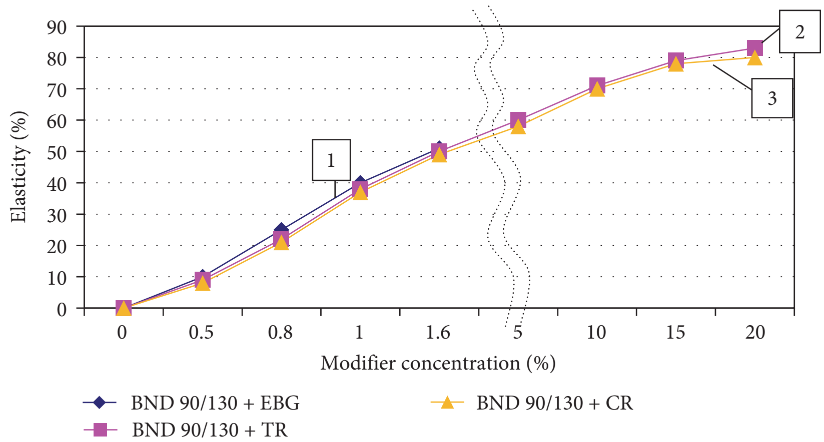


Figure 10: Elasticity dependence of bitumen-polymer compositions at 25°C on the concentration of modifiers.

Table 5: Properties of modified bituminous binders.

Composition	Penetration at 0°C (0.1 mm)	Ductility at 0°C (cm)	Elasticity at 0°C (%)
BND 90/130	50	0	0
BND 90/130 + 1,6% EBG	42	5, 9	45
BND 90/130 + 20% TR	36	5	75
BND 90/130 + 20% crumb rubber	35	4	74

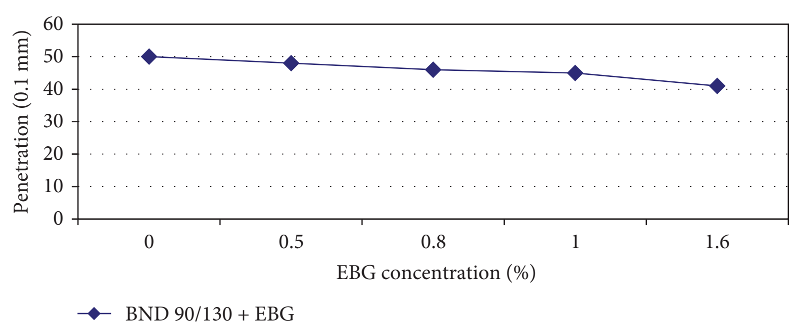


Figure 11: Dependence of the penetration of bitumen-polymer compositions at 0°C on the concentration of the modifier.

traditional effects of modifying bitumen with EBG, an increase in the softening point, hardness, and elasticity of bitumen, unusual effects were found, an increase in ductility at 25°C (Figure 9).

Penetration at low temperature (P_0) (Figure 11) decreases (1, 2) for BND from 50 to 42, respectively.

Reducing the penetration, i.e., increasing the hardness of modified bitumen compositions, a significant improvement in low-temperature and elastic properties, is a very positive factor for low-viscosity bitumen.

It has been established that high technical indicators (T_s, P_0, P_{25}, D_{25}, D_0, E_{25}, E_0) are achieved at an EBG concentration of 0.8%–1.6%, i.e., at a low concentration, which to some extent justifies the price of EBG.

Thus, the modification of bitumen with a copolymer of ethylene with butyl acrylate and glycidyl methacrylate leads to an improvement in the properties of road bitumen and increases the softening point, hardness, deformability at low temperature, elasticity, and adhesion to metal and mineral filler.

4. Conclusions

Based on the results of the analyses carried out, we would like to note the most basic conclusions that we have drawn:

(1) It has been shown that EBG chemically interacts with the functional groups of bitumen asphaltenes through the epoxy group of glycidyl methacrylate.

(2) It has been established that the optimal bitumen—reclaimed composition (with a tire reclaim content of 20%) is superior in terms of the complex of properties to bitumen modified with the optimal content of EBG (1.6%).

(3) The bitumen, modified with rubber crumbs of 0.6–1.0 mm in size, has high elasticity.
(4) The technology for modifying bitumen with reclaim is less time-consuming, more economically profitable, and environmentally effective, since it utilizes large-tonnage waste of worn-out tires.
(5) The resulting bitumen-polymer compositions have a high positive set of properties: softening point, hardness, elasticity, frost resistance, and low-temperature characteristics.

Data Availability

Data will be available upon request to guzalita.f1978@mail.ru.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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