SYNTHESIS AND PROCESSING OF POLYMERS AND POLYMERIC COMPOSITES

СИНТЕЗ И ПЕРЕРАБОТКА ПОЛИМЕРОВ И КОМПОЗИТОВ НА ИХ ОСНОВЕ

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RESEARCH ARTICLE

Change of electrical characteristics of rubber in the process of "swelling–deswelling"

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Objectives. *The main indicator that determines electrical conductivity of rubbers is specific volumetric electrical resistance (ρ_v). The purpose of this work is to investigate changes in this indicator during swelling and deswelling of electrically conductive rubbers. When considering the swelling process of rubbers in liquid media at a molecular level, an analogy of this process with mechanical deformation of the material is drawn and common features and differences of these processes are revealed.*

Methods. *For rubber compositions based on paraffinate and alkyl sulfonate nitrile butadiene rubbers, the degree of their swelling and the change in linear dimensions in heptane and in gasoline grades 80, 92, and 95 were determined. The ρ_v value was determined by a potentiometric method: the initial value was measured after temperature control of rubbers for 1 h at 120°C, and the second measurement was carried out after these rubbers were swollen in the solvents for 48 h, followed by drying at 20°C to a constant weight and repeated temperature control under the same conditions. Using an IR Fourier spectrometer, spectra of the solvents used were obtained before and after identification of the investigated rubber samples in them.*

Results. *It was shown that the type of rubber and solvent used influence the degree of rubber swelling. Rubber compositions based on natural rubbers with a large amount of attached acrylonitrile, obtained in the presence of an alkyl sulfonate emulsifier, have the highest resistance to swelling. The effect of the used solvent on the change in the degree of swelling is determined by its affinity for rubber and the presence of polar additives that increase the octane number of gasoline. It was established that the linear change of the samples upon swelling in the indicated solvents varies according to the length and thickness of the samples. Results show that depending on the type of rubber used and the degree of its filling, the described rubber processing technology leads to a decrease in the ρ_v value by 2 to 20 times. The greatest effect of ρ_v reduction is observed in low-filled rubber compositions based on paraffinate nitrile rubbers. The spectra of the frustrated total internal reflection of the solvents after their interaction with the studied rubbers show that particulate extraction of dibutyl phthalate, which was used as a plasticizer in rubber compounding, takes place as a result of rubber swelling.*

Conclusions. *The proposed method of rubber processing reduces the ρ_v value by removing dibutyl phthalate from the studied rubbers and forming a more developed carbon–elastomer structure. Furthermore, it solves the problem of the negative effect of the plasticizer on the ρ_v value of rubber without excluding it from the rubber composition.*

Keywords: *rubber, specific volume electrical resistance, deformation, degree of swelling, linear change, reduction factor, dibutyl phthalate, extraction, developed carbon–elastomer structure*

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НАУЧНАЯ СТАТЬЯ

Изменение электрических характеристик резин в процессе «набухание-отбухание»

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Цели. *Основным показателем, определяющим электропроводность резин, является удельное объемное электросопротивление. Цель работы – исследовать изменения данного показателя при набухании и отбухании электропроводящих резин. При рассмотрении процесса набухания резин в жидких средах на молекулярном уровне проведена аналогия этого процесса с механической деформацией материала, выявлены общие черты и различия этих процессов.*

Методы. *Для резин на основе парафинатных и алкилсульфонатных бутадиен-нитрильных каучуков были определены степень их набухания и изменение линейных размеров в гептане, бензинах марок АИ-80, АИ-92, АИ-95. Удельное объемное электросопротивление определено потенциометрическим методом: исходное значение измерено после термостатирования резин в течение 1 ч при 120 °С, а повторное измерение проведено после набухания этих резин в растворителях в течение 48 ч с последующей сушкой при 20 °С до постоянной массы и повторного термостатирования при тех же условиях. С помощью ИК-спектрометра Фурье были получены спектры растворителей до и после нахождения в них исследованных резин.*

Результаты. *Показано, что на степень набухания резин оказывает влияние тип используемого каучука и растворителя. Наибольшей стойкостью к набуханию обладают резины на основе каучуков с большим количеством присоединенного НАК, полученные в присутствии алкилсульфонатного эмульгатора. Влияние используемого растворителя на изменение степени набухания определяется его сродством к каучуку и наличием полярных добавок, повышающих октановое число бензина. Установлено, что изменение линейных размеров образцов при набухании в указанных растворителях различно по длине и толщине образца. Результаты измерения удельного объемного электросопротивления показали, что в зависимости от типа используемого каучука и степени его наполнения описанная технология обработки резины приводит к снижению данного показателя от 2 до 20 раз. Наибольший эффект снижения удельного объемного электросопротивления наблюдается у малонаполненных резин на основе парафинатных бутадиен-нитрильных каучуков. Представленные в работе спектры нарушенного полного внутреннего отражения растворителей после их взаимодействия с исследованными резинами показали, что в результате набухания резин происходит частичная экстракция из них дибутилфталата, использованного в рецептуре в качестве пластификатора.*

Выводы. *Предложенный способ обработки резины позволяет снизить ее удельное объемное электросопротивление за счет удаления из нее дибутилфталата и формирования более развитой углерод-эластомерной структуры и решить проблему отрицательного влияния пластификатора на удельное объемное электросопротивление резин без исключения его из состава резиновой смеси.*

Ключевые слова: *резина, удельное объемное электросопротивление, деформация, степень набухания, изменение линейных размеров, кратность снижения, дибутилфталат, экстракция, развитая углерод-эластомерная структура*

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INTRODUCTION

Rubbers, like the vast majority of currently known polymers, are dielectrics. However, it is possible to make electrically conductive rubber compositions, which are widely used in various industries and in everyday life, and their range of applications is constantly expanding. To create such materials, special grades of carbon black are added into elastomers, which have high specific adsorption surface, dispersion, structure, and roughness [1–3]. These indicators play a decisive role in the formation of a developed spatial carbon–elastomer structure, which ensures charge transfer during direct contact of filler particles or through thin rubber layers by a tunneling mechanism [4–7].

The level of electrical characteristics of electrically conductive rubbers determines their specific volumetric electrical resistance, ρ_v. A decrease in the ρ_v value results from increasing the content of electrically conductive filler or using filler with a higher level of specific adsorption surface, dispersity, and structure [8, 9]. However, after reaching the optimum filling, a further increase in the content of carbon black causes a sharp increase in the viscosity of the rubber compound and, as a consequence, a deterioration in the processability of the composition, a decrease in the elastic-strength properties of rubbers, and an increase in their cost [10]. The gain in electrical characteristics of the material when filled above the optimum is relatively small.

The current level of development of science, technology, and medicine requires finding new approaches to improving the electrical characteristics of rubbers, for example, using the influence of external factors on the carbon–elastomer structure already formed in the process of rubber production.

It is known from the literature [11] that the specific volumetric electrical resistance of rubber is influenced not only by various technological factors, such as the order of addition of ingredients and vulcanization, or prescription factors (introduction of plasticizers and fillers into the rubber mixture) but also by the conditions of its operation. These include the effect of elevated temperature, the presence of deformation, the deformation rate and magnitude, and the degree of swelling of rubber in solvents.

The effect of elevated temperature on rubber leads to a noticeable decrease in the ρ_v value of the vulcanizate [12], which, according to many researchers, is associated with the desorption of the elastomer from the surface of carbon black particles and the formation of larger extended structures—clusters, which facilitates charge transfer. In this case, the intensity of the decrease in the ρ_v value is determined by the temperature acting on rubber.

When a sample of an electrically conductive rubber is deformed by stretching, compression, or shear, a number of complex processes occur in it, leading to a nonmonotonic change in the initial specific volumetric electrical resistance: an increase in the ρ_v value upon deformation of up to 50% due to destruction of the carbon–elastomer structure, a subsequent decrease in the ρ_v value due to orientation of the elements of the carbon–elastomer structure along the direction of deformation at its significant value, and a sharp increase in the ρ_v value upon removal of the deforming force and reduction of sample due to disorientation of the carbon–elastomer structure [11]. Thus, any deformation of tension, compression, and shear of a sample of an electrically conductive rubber will ultimately lead to a significant increase in the ρ_v value, making it more electrically conductive.

According to the general definition of the concept of “deformation,” swelling of rubbers in liquid media can also be considered as their deformation. When considering the process of swelling at a molecular level, direct contact of rubber and liquid leads to diffusion of the latter into the sample volume due to the presence of free space between elastomer macromolecules. Due to this, liquid molecules easily and quickly

penetrate between macromolecules, pushing and deforming molecular chains. The amount of solvent absorbed by rubbers is different and is determined by the affinity of the elastomer and the solvent, the time of their interaction, as well as the density of the spatial vulcanization network. However, when rubber swells, in contrast to tensile, compression, and shear deformation, the sample volume increases and all of its linear dimensions simultaneously increase. This, naturally, is accompanied by an increase in the distance between filler particles, which complicates the charge transfer and leads to a noticeable increase in the ρ_v value.

However, in the process of solvent evaporation—"deswelling" of rubber—the initial electrical conductivity is restored. Thus, the nature of the effect on the electrical conductivity of rubbers is one of the main differences in the deformation process during swelling and deswelling from, for example, stretching the sample under mechanical action and returning it to its original state, and it requires more careful attention and study.

In this regard, the purpose of this work is to study the change in the ρ_v value of electrically conductive rubbers during the swelling and deswelling process.

MATERIALS AND METHODS

Elastomeric compositions based on nitrile butadiene rubbers (NBRs) were manufactured and investigated: NBR18 and NBR26 (alkyl sulfonate), and NBR1845 and NBR2645 (paraffinic) (*Krasnoyarsk Synthetic Rubber Plant*, Russia). These brands of rubbers differ not only in the content of bound acrylic acid nitrile but also in the type of emulsifier used during the polymerization process, which has a decisive effect on both the vulcanization properties and the physicomechanical and operational properties of rubbers [13–16]. In order to impart electrically conductive properties into rubbers, mixed compositions [17] were used, consisting of conductive carbon black grade UM76 (*KHIMPLAST*, Russia), the content of which varied from 25 to 50 mass fractions in combination with 50 mass fractions of low-activity carbon black P803 (*Tuymazytehuglerod*, Russia) and 10 mass fractions of graphite GK-1 (*Zavalivskiy Graphite*, Ukraine). Mixture composition of fillers provides the rubber compound with satisfactory processing properties even with a total filler content of 110 mass fractions per 100 mass fractions of natural rubber, and rubbers have high and stable electrical characteristics. For vulcanization, a standard vulcanizing group was used, consisting of zinc oxide, stearic acid, sulfenamide C, and sulfur (*VitaKhim Group*, Russia). In addition, to facilitate processing operations, dibutyl phthalate (DBP) (*RosKhim Group*, Russia) was used.

Elastomeric compositions were made on LB 320 160/160 rollers (*Metallist*, Russia), and the electrically conductive filler was introduced into the rubber mixture already containing carbon black P803 [9]. After curing, the rubber compounds were vulcanized at the optimum vulcanization time determined on an RPA-2000 rheometer (*Alpha Technologies*, USA). Degree of swelling of rubbers after 48 h of exposure to organic solvents (heptane (*ORGKHIM*, Russia) and gasoline grades AI-80, AI-92, and AI-95 (*Gazpromneft-MNPZ*, Russia)) was determined in accordance with GOST 9.030-74[1].

The main characteristic of electrically conductive rubbers— the indicator of the specific volumetric electrical resistance—was determined by a potentiometric method (ISO 1853:1998[2]). Before measurements, the rubber samples were thermostated for 1 h at 120°C. After measuring the initial ρ_v value, they were placed in the organic solvents for 48 h. After that, the solvent was completely removed by drying the swollen samples to a constant weight at a temperature of 20°C and thermostated them again in an air thermostat for 1 h at 120°C, and then the ρ_v value was measured.

To determine the nature of substances extracted from the rubber samples as a result of exposure to the solvents, spectra of the solvents used were obtained by an IR Fourier spectrometer (*Bruker*, Germany) before and after identification of the investigated rubbers in them.

RESULTS AND DISCUSSION

The values of the degree of swelling of the manufactured rubbers are given in Table 1.

The data in Table 1 show that the degree of swelling is determined by the type of rubber and solvent used. More resistant to swelling are rubber compositions based on rubbers with a high content of acrylonitrile (ACN), regardless of the type of emulsifier used. Rubber compositions based on paraffinic rubbers are inferior in resistance to swelling compared to those that are based on alkyl sulfonate rubbers. This is especially pronounced for rubbers based on rubbers containing 18% ACN.

The type of solvent used has a significant influence on the degree of swelling of rubbers. The smallest

[1] GOST 9.030-74. Unified system of corrosion and ageing protection. Vulcanized rubbers. Method of testing resistance to attack by corrosive media in limp state. Moscow: Standartinform; 2008.

[2] ISO 1853:1998. Conducting and dissipative rubbers, vulcanized or thermoplastic – Measurement of resistivity.

Table 1. Degree of swelling of rubbers in the organic solvents (for example, rubber containing 35.0 mass fractions of carbon black UM76)

Elastomeric rubber base	Degree of swelling after 48 h, %			
	Heptane	AI-80	AI-92	AI-95
NBR18	1.6	30.7	18.7	36.1
NBR26	−0.8	15.1	11.4	17.7
NBR1845	5.0	42.6	39.2	60.0
NBR2645	−0.8	14.9	14.0	20.3

degree of swelling was observed in heptane due to a significant difference in the solubility parameters of the rubber samples and the solvent. For rubbers based on NBR26 and NBR2645, there was even a decrease in the mass of samples after swelling compared to the initial values. This is probably due to leaching of some of the ingredients from the sample during its swelling. The studied rubbers swelled more intensively in gasoline grades AI-80, AI-92, and AI-95 than in heptane, which is due to the presence of polar additives in their composition that increase the octane number. By increasing the degree of swelling, the gasolines used in this work can be arranged in the following sequence: AI-95 > AI-80 > AI-92. An increase in the content of ACN in rubber leads to a noticeable decrease in the influence of the gasoline brand on the degree of swelling of rubbers.

Table 2 shows the change in the linear dimensions of the rubber samples based on NBR18, containing 30.0 mass fractions of conductive carbon black UM76, 50.0 mass fractions of low-activity carbon black P803, and 10.0 mass fractions of graphite GK-1, before and after their swelling in the selected solvents.

The data presented in Table 2 clearly demonstrate a significant change in the linear dimensions of the samples after prolonged (48 h) exposure to the solvents, which is evident of the occurrence of deformation processes that affect the carbon–elastomer structure formed in the rubber compounds. It can be noted that the degree of deformation of the samples along the length and thickness was different under the action of any of the considered solvents, i.e., the sample was unevenly deformed upon swelling. The amount of deformation along the thickness of the samples was noticeably greater than the deformation along the length for all the samples and media.

Thus, the solvents used in this work interacted to varying degrees with the studied rubbers from the point of view of the intensity of their absorption by the test material and, consequently, the change in the size of the swollen samples. This allows a more complete assessment of the effect of deformation process during swelling on specific volumetric electrical resistance.

Figures 1–4 show the dependence of the ρ_v value on the content of technical carbon in rubber, as well as the reduction factor of the ρ_v value, which was determined as the ratio between the values of this indicator before and after the above-described exposure.

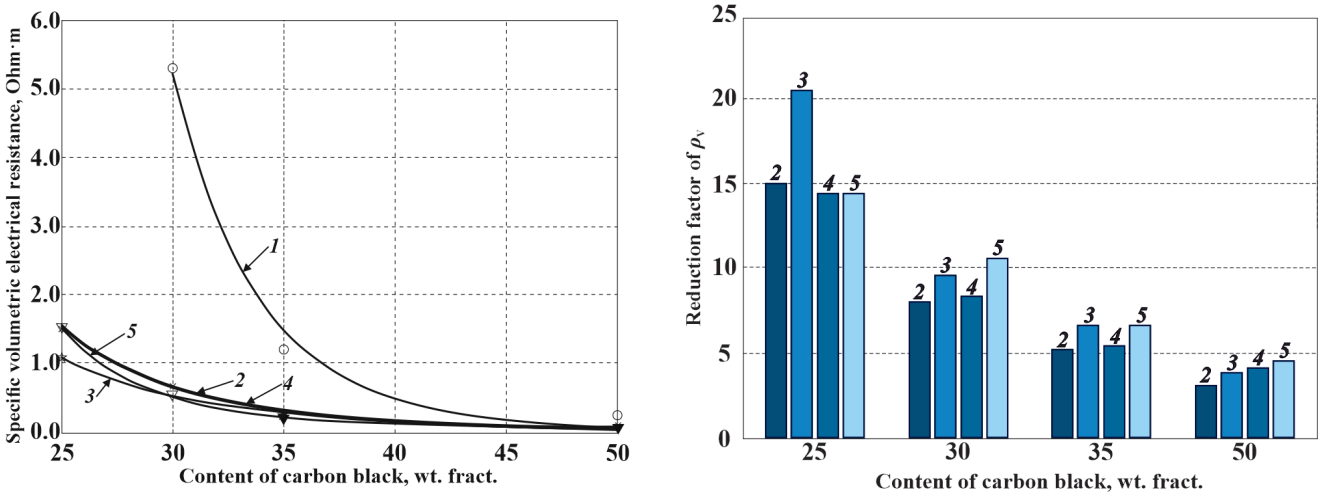
As seen from Figs. 1 to 4, swelling of the rubber samples in any of the considered solvents followed by the complete removal of the solvent according to the scheme described above made it possible to obtain a material with a lower specific volumetric electrical resistivity as compared to the initial value. The reduction factor of the ρ_v value varied over a wide range of values. From the presented data, it can be seen that the greatest effect of reducing the ρ_v value was observed for rubbers containing the minimum of the considered dosages of electrical conductive carbon black UM76 (25 mass fractions). The reduction factor of the specific volumetric electrical resistance for these rubbers was 15.0–20.5. For rubbers with carbon black UM76 content of 30 mass fractions and more, the effect of reducing the ρ_v value was less pronounced; the reduction factor was in the range of 1.1–5.0.

It should be noted that the type of elastomeric binder used to obtain electrically conductive rubbers [14] affects the ρ_v value of the material obtained by the method described above. The increase in the reduction factor of the ρ_v value was most pronounced for rubbers based on paraffinic NBR.

When comparing the electrical properties of equally filled rubbers based on the studied rubbers, it can be noted that the reduction factor of the ρ_v value for most rubbers increased with an increase in the degree of swelling and, accordingly, an increase in their deformation during the swelling process.

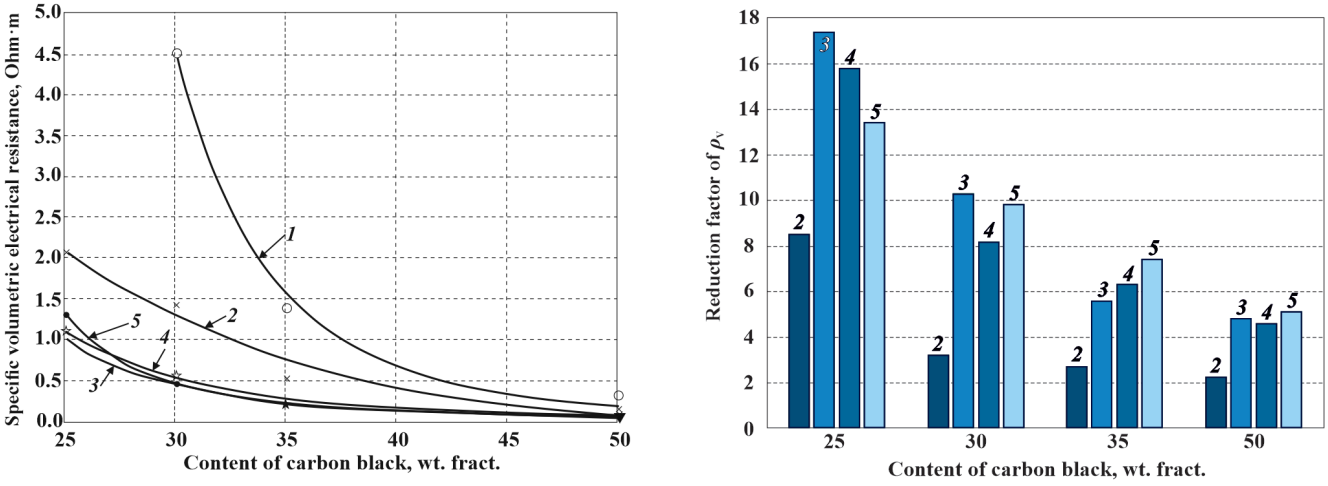
Table 2. Change in the linear dimensions of the rubber samples based on NBR18 before and after swelling in various solvents

Linear dimension and mass of sample	Sample linear dimension value		Change, %
	Before swelling	After swelling	
Heptane			
Width, mm	1.87	1.94	3.74
Length, mm	60.0	61.0	1.67
Mass, g	1.926	1.963	1.92
Gasoline grade AI-80			
Width, mm	1.87	2.29	22.5
Length, mm	60.0	68.0	13.3
Mass, g	1.930	2.580	33.7
Gasoline grade AI-92			
Width, mm	1.87	2.21	18,2
Length, mm	60.0	67.0	11.7
Mass, g	1.912	2.408	25.9
Gasoline grade AI-95			
Width, mm	2.08	2.56	23.1
Length, mm	60.0	69.0	15.0
Mass, g	2.139	2.934	37.2



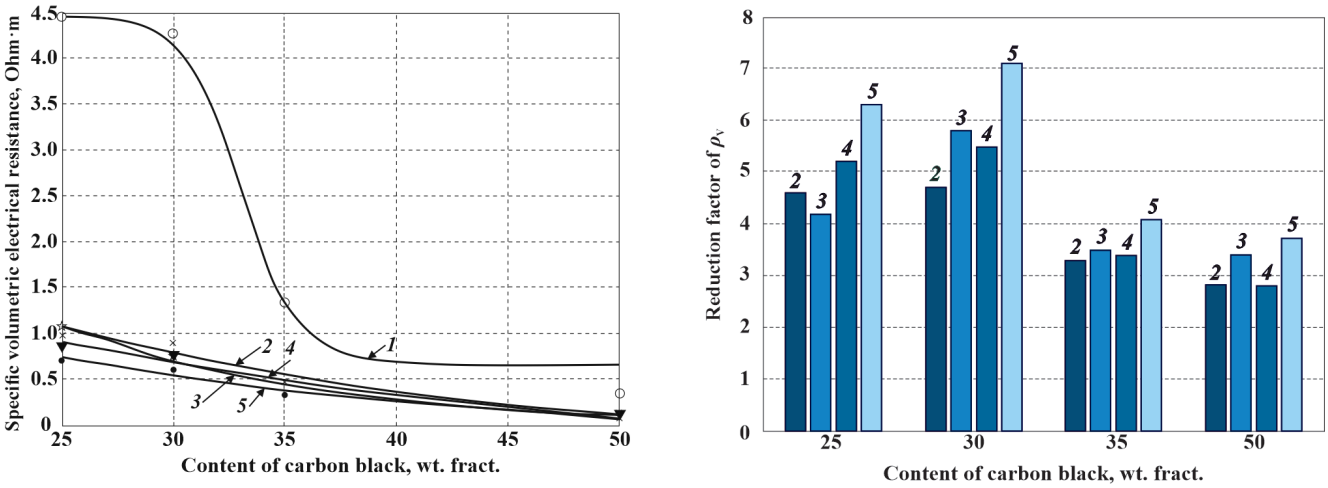
a b

Fig. 1. Dependence of the ρ_v value on the content of carbon black UM76 (a) and reduction factor of the ρ_v value (b) for electrically conductive rubbers based on NBR18:
(*1*) before interaction, (*2*) heptane, (*3*) AI-80 gasoline, (*4*) AI-92 gasoline, and (*5*) AI-95 gasoline.



a b

Fig. 2. Dependence of the ρ_v value on the content of carbon black UM76 (a) and reduction factor of the ρ_v value (b) for electrically conductive rubbers based on NBR26:
(*1*) before interaction, (*2*) heptane, (*3*) AI-80 gasoline, (*4*) AI-92 gasoline, and (*5*) AI-95 gasoline.



a b

Fig. 3. Dependence of the ρ_v value on the content of carbon black UM76 (a) and reduction factor of the ρ_v value (b) for electrically conductive rubbers based on NBR1845:
(*1*) before interaction, (*2*) heptane, (*3*) AI-80 gasoline, (*4*) AI-92 gasoline, and (*5*) AI-95 gasoline.

The results shown in Figs. 1–4 show that an increase in the content of carbon black UM76 in the mixed filler in the dosage range of 25–50 mass fractions, in the case of rubber processing according to the proposed technology, changed the value of the ρ_v value within the same order. Thus, the considered method of rubber processing allows reducing the content of the expensive filler in the composition, which will lead to a decrease in the cost of the material and an increase in the complex of technological and physical and mechanical properties of rubbers.

When rubber swells, solvent molecules increase the distance between macromolecules, which can facilitate the transition of the rubber ingredients into the solvent. Therefore, it is natural to assume that the effect of reducing the ρ_v value of rubbers upon contact with the organic solvents is due to the processes of extracting ingredients from rubber that affect the formation of the carbon–elastomer structure and the formation of a more developed carbon–elastomer structure during the removal of the solvent.

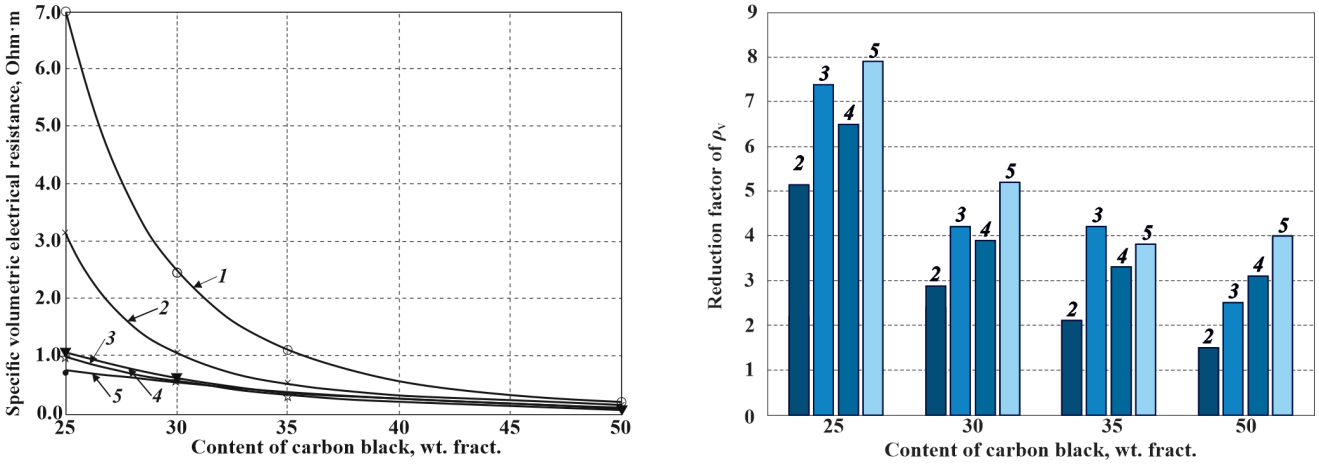
Figures 5 and 6 both show the spectra of the used solvents before and after identification of the studied rubbers in them, which were obtained using an IR Fourier spectrometer.

When comparing the IR spectra of the solvent (AI-95 gasoline) before and after its interaction with the rubber compositions based on NBR18

and NBR1845 (Figs. 5 and 6), one can note the appearance of bands, indicating that low-molecular substances are extracted as a result of the swelling process of the samples with the solvent. For all the considered solvents, the presence of characteristic absorption bands at 1284 and 1121 cm^{-1} can be noted. According to [18], such bands are characteristic of compounds containing an ether group. In the rubber samples investigated in the framework of this work, of all the ingredients in their composition, the ether group is contained only in the plasticizer dibutyl phthalate.

The negative effect of a plasticizer on the electrical conductivity of rubbers while maintaining a constant content of carbon black is understandable and has been known for a long time. However, its exclusion from the rubber composition in order to reduce the ρ_v value is not always possible. In the case of rubber compounds based on NBR, due to their high polarity and, as a result, high viscosity, the absence of a plasticizer complicates the production and processing on mixing and forming equipment.

In this regard, in the case of electrically conductive rubbers based on NBR, a choice arises between making a more technologically advanced rubber compound and obtaining a finished product with a higher level of electrical characteristics. The processing method considered above makes it possible to eliminate the negative effect of the plasticizer on the ρ_v value of the material. After selecting the



a b

Fig. 4. Dependence of the ρ_v value on the content of carbon black UM76 (a) and reduction factor of the ρ_v value (b) for electrically conductive rubbers based on NBR2645:
(*1*) before interaction, (*2*) heptane, (*3*) AI-80 gasoline, (*4*) AI-92 gasoline, and (*5*) AI-95 gasoline.

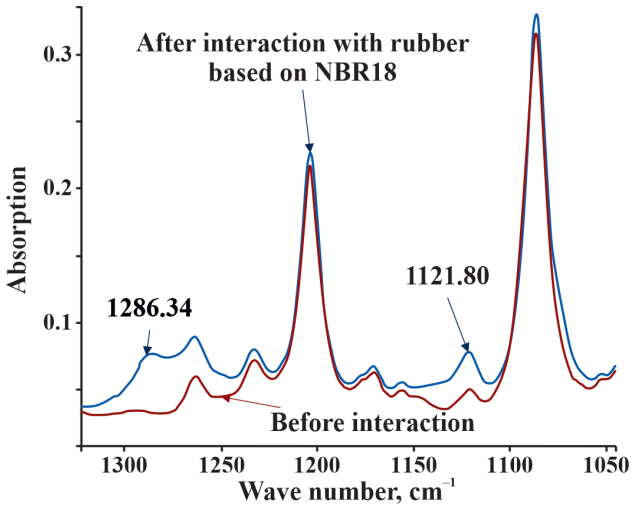


Fig. 5. Attenuated total reflection spectrum of AI-95 gasoline before and after interaction with the rubber compositions based on NBR18.

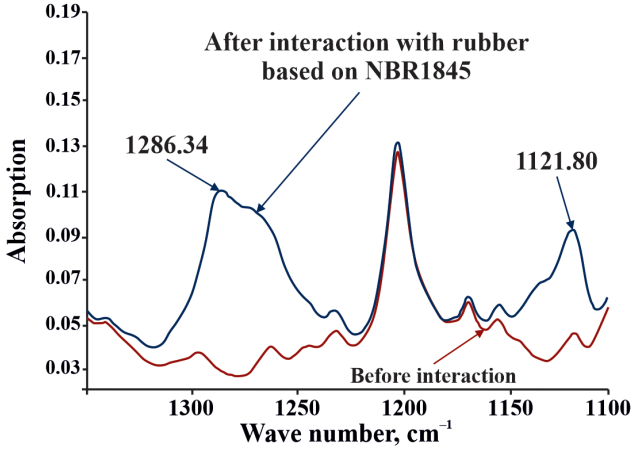


Fig. 6. Attenuated total reflection spectrum of AI-95 gasoline before and after interaction with the rubber compositions based on NBR1845.

appropriate solvent, the described method can also be applied to rubbers for general applications [19].

CONCLUSIONS

Thus, the paper considers a method of directed physical action on the carbon–elastomer structure formed in rubber during its manufacture, which provides a 2- to 20-fold decrease in the ρ_v value of electrically conductive rubbers. A decrease in the ρ_v value upon contact of an elastomeric material with a certain solvent occurs due to the formation of a more developed carbon–elastomer structure during the swelling–deswelling process of rubber and partial removal of a plasticizer from it. The proposed method of directed physical action on the carbon–elastomer structure allows one to solve the problem of the negative effect of a plasticizer on the ρ_v without excluding it from the rubber composition.

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Authors' contribution

All authors equally contributed to the research work.

The authors declare no conflicts of interest.

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