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Application of modifying systems based on phenol-formaldehyde resins and products of processing of fat-containing raw materials in elastomeric compositions

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One of the ways to improve the quality of elastomeric materials is to modify them with active compounds. As modifying additives in this work, the systems were considered which are based on sulfur-containing phenol-formaldehyde resins Octofor 10S and 101K together with an amine-containing compound produced by the interaction of ethanol amines with the methyl ester of fatty acids, the raw material being chicken fat. Study was carried out using model polymer compositions based on butadiene and styrene copolymer. Analysis of the complex of properties showed that it is effective to use 3–4 parts by weight of additives at the ratio of components 2:1. Mechanical mixture of components is inferior to the additive produced by the components fusion in this respect. Moreover, the dynamic strength of rubber is significantly increased. A significant reduction in the mechanical loss tangent was established in the composition with the use of Octofor 10S resin. That allows reducing heat formation at dynamic loads and increasing the service life of large articles, such as tyres. The patterns of the influence of modifiers on the complex of properties of elastomeric compositions are useful when choosing the direction of industrial application of the additives under consideration in the composition of rubber compounds. The addition of crushed vulcanizate treated with modifying additives was found to reduce the viscosity of rubber compounds as compared with the use of untreated vulcanizate; this decreases the energy consumption in the manufacture and processing of elastomeric compositions.

Keywords: modifier, phenol-formaldehyde resin, chicken fat, elastomer, composition, resource saving.

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Introduction

The development of modern industries requires continuous improvement of the basic performance characteristics of rubber components and above all their durability. Modification of elastomeric materials with active compounds is known to be one of the reserves for improving their quality. It is carried out under the conditions of a generally accepted technological process of rubber product manufacture. Elastomer composites modification was started in the 50s and 60s of the 20th century mainly with the aim of increasing adhesion in rubber-cord systems [1]. The aim of these works is the introduction of adhesive-active additive into the composition of rubber mixtures and their subsequent interaction with the functional groups of cord or latex, with the formation of strong adhesive-rubber chemical bonds.

At present, the polymer material industry uses various modifying techniques: chemical, structural, physical, radiation, electromagnetic and others. They all have the common goal of improving the properties of polymers and composite materials based on them by slightly changing the molecular structure of macromolecules or transforming the supramolecular structures of polymers.

Oligomeric modifiers are the most effective for increasing the vulcanizate elasticity and durability. The oligomeric modifiers include epoxy and phenol-formaldehyde resins of different structures [2,3]. Resins and oligomers are used both individually and as a part of modifying systems [4].

In addition to phenol-formaldehyde oligomers, aliphatic polyamines of different structures [5] are effective modifiers as well. However, polyamine modifying systems with phenol-formaldehyde resins have a greater impact on the improvement of elastomer material properties [6]. It is proposed to carry out preliminary interaction between the components of the system at elevated temperatures. The resulting compounds have a positive influence on the complex properties of vulcanizates.

Previous work [7] showed that the use of modifying systems based on a sulfur-containing phenol-formaldehyde oligomer, resin Octofor 10S

with aliphatic polyamines of various types, is highly effective in rubber mixtures.
The aim of the work is to find new components of modifying systems based on phenol-formaldehyde resins to expand the range of ingredients of elastomer materials and to improve the quality of rubber products.

Experimental

Modifying systems based on phenol-formaldehyde resins were studied. The object of study was sulfur-containing phenol-formaldehyde resin Octofor 10S of 10–12% as well as resin 101K. Salt as an amine-containing component, obtained by the interaction of triethanolamine with a fatty acid methyl ester (TEA-FAME), chicken fat as raw material, was studied. The presence of active functional groups and a sufficiently high surface energy of this compound allow providing the possibility of its use as a co-former in modifying systems based on phenol-formaldehyde oligomers.
The influence of composite additives in formulations of model rubber mixtures based on butadiene-styrene rubber SBR-1705 HI-AR of the following composition, parts by weight, was investigated: Rubber SBR-1705 HI-AR 100.0; filler (technical carbon N339) 40.0; stearic acid 2.0; zinc white 5.0; sulfur technical 2.0; and Altax 3.0.
According to the results of previous studies [8], the ratio between the selected components of the modifying systems ranged from 2:1 to 1:2. The additives were prepared by mixing the components at temperatures above the melting point of the resins by 10–150^{0}C for 10–15 minutes. The characteristics of the modifying systems are given in Table 1. The additives obtained were added to the rubber mixtures together with the free-flowing ingredients.
The improvement of properties of the rubber composition as well as kinetics of their vulcanization was characterized by "Alpha Tecnologes" rotor-free rheometer MDR-2000 with a lower-shaped oscillation amplitude of 0.5 degrees. The parameters were calculated using a computer program. The mechanical loss tangent of both the rubber mixture at minimum torsion torque and the vulcanizate at maximum torsion torque were determined as well. The characteristics of the rubber modifiers were analyzed according to the results of the determination of the elastic-strength characteristics under static and dynamic load.

Results and discussion

The best rubber elastic-strength properties were achieved at the concentration of additives of 3-4 parts per 100 parts of rubber, as was shown by determining the dependence of the rubber strength properties on the additive content. This conclusion coincides with the data [8].
Table 2 shows the results of studied properties of model rubbers based on SBR-1705 HI-AR rubber, which contains 4.0 parts additives based on phenol-formaldehyde resin Octofor 10S.
In order to assess the feasibility of preparing a component alloy, a comparison was made between the effect of the mechanical mixture of the components and the additive produced by their fusion.
Analysis of the data summarized in Table 2 showed that the use of additives, both as a mixture and as an alloy, did not change either the viscosity of the rubber mixtures (minimum torque) or the degree of crosslinking (maximum torque). The application of additives under study increases the rate of vulcanization, probably due to the presence of sulfur-containing fragments in the resin that may influence the vulcanization kinetics.

Table 1. Characteristics of additives based on phenol-formaldehyde oligomer Octofor 10S

Code	Components	Manufacturing temperature, ^{0}C	Manufacturing time, min	Melting point, ^{0}C	Appearance
Octofor-FAME	Octofor 10S+salt, the product of interaction of monoethanolamine with methyl ester of fatty acids, (ratio 2:1)	90–120	15–20	80–95	Resinous mass of dark brown color
Octofor-FAME-S	Resin 101K+salt, the product of interaction of monoethanolamine with methyl ester of fatty acids+sulfur	90–120	15–20	70–85	Resinous mass of dark brown color

Table 2. Properties of model elastomeric compositions containing 4 parts additives based on Octophor 10S

Indexes	No additives	A mixture of Octofore 10S with TEA-FAME (1:1)	Alloy of Octofore 10S with TEA-FAME at a ratio		
			2:1	1:1	1:2
Rheometer characteristics at 175^0C:					
Minimum torque M_{min}, dN m	1.38	1.30	1.43	1.36	1.28
Maximum torque M_{max}, dN·m	10.56	11.31	11.40	11.28	11.02
The difference in torque ΔM, dN m	9.18	10.01	9.97	9.92	
Time of the beginning of vulcanization t_{S2}, min	0.87	0.79	0.79		0.72
Optimal vulcanization time t_{90}, min	2.79	2.77	2.74	2.67	2.55
Vulcanization rate R_V, min^{-1}	5.12	6.03	6.04	6.19	
Conditional stress at 300% elongation, MPa	13.2	13.8	14.0	13.6	13.1
Conditional tensile strength, MPa:					
at 25^0C	19.5	17.4	21.6	21.0	20.8
after aging at 100^0C for 48 hours	16.5	13.2	17.2	17.0	16.6
Relative elongation at break, %:					
at 25^0C	430	390	420	400	400
after aging at 100^0C for 48 hours	290	230	305	300	290
Tear resistance, kN m^{-1}	26.5	25.4	27.3	27.0	26.9
Resistance to multiple deformations, thousand cycles:					
at 25^0C	12.0	14.6	29.8	19.2	15.5
after aging at 100^0C for 48 hours	5.9	9.5	15.0	11.5	10.0

The results of the determination of mechanical properties show that it is advisable to use an alloy, since the conditional strength of rubber with alloy is higher than that of rubber without researched additives and rubber containing a mixture of resin and salt. This can be explained by the fact that, when an alloy is obtained in the system, the interaction between the components with the formation of more active substances of sulfonamide character is reliably observed, in part by the presence of a thermal effect on the derivatives.

It is interesting to observe the results of determining the mechanical loss tangent (tgδ) of rubber compositions (Fig. 1). The addition of a modifying system was shown to result in a significant reduction of tgδ for both rubber mixtures and vulcanizates. In the first case, this shows that it is possible to reduce energy consumption in the manufacture and processing of rubber mixtures on different equipment by reducing the level of interphase interaction in compositions; and for vulcanizates, the decrease of tgδ results in less heat formation in the mass of products, especially large ones, such as large and superlarge tyres, under dynamic load. The latter will reduce the negative impact of heat on the performance of articles under dynamic load, which will ensure greater durability, for example, of tyres in use.

The decrease in tgδ is probably due to the fact that modifiers based on phenol-formaldehyde resin are able to be distributed in the matrix of the elastomer in the form of microdroplets and perform the function of internal lubrication, which increases the degree of microheterogeneity of the system and significantly increases the mobility considered in work [9]. The result of this action of modifiers is a significant increase in the dynamic characteristics of tyres, as shown by their increasing resistance to repeated deformations (Table 2). Thus, the obtained results are a prerequisite for the use of resin-based compositions of Octofor 10S in the composition of industrial rubber, wherein the best properties are achieved at the ratio of phenol formaldehyde oligomers: TEA-FAME=2:1 (Octofor-FAME modifier).

The influence on the model resin properties of the composition based on phenol-formaldehyde resin 101K was also determined in order to extend the range of modifying additives. Taking into account the results of work [7] during the manufacture of resin with TEA-FAME additives, additional 10–12% sulfur was added (Octofor-FAME-S modifier).

The obtained results are summarized in Table 3 and Fig. 2. The analysis of the rheometric data showed that the addition of this type of additive reduces the minimum torque of rubber mixtures, which may indicate the plasticizing effect of 101K resin-based additives. In terms of influence on vulcanization processes, the addition of 101K-based alloys is found to be safer than Octofor 10S-based alloys given the processes of sub-vulcanization, but the rate of vulcanization is lower. The results are a basis for adjusting the processing and vulcanization of rubber mixtures.

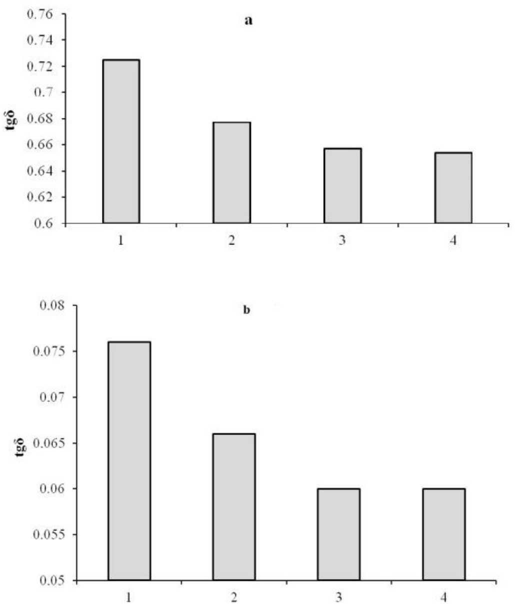

Fig. 1. Mechanical loss tangent (tgδ) of rubber mixtures (a) based on rubber SBR-1705 HI-AR and vulcanizates based thereon, and (b) containing modifying systems based on resin Octofor 10S. (Reference: Table 2)

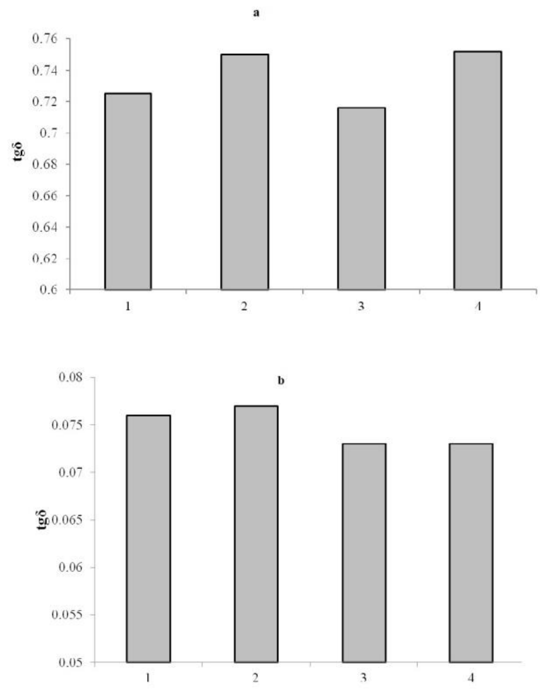

Fig. 2. Mechanical loss tangent (tgδ) of rubber mixtures (a) based on rubber SBR-1705 HI-AR and vulcanizates based thereon, and (b) containing modifying systems based on 101K resin. (Reference: Table 3)

Table 3. Properties of model elastomeric compositions containing 4 parts by weight additives based on 101K resin

Indexes	No additives	A mixture of resin with TEA-FAME (1:1)	The alloy of resin 101K with TEA-FAME in the ratio 2:1	
			without sulfur	in the presence of sulfur
Rheometer characteristics at 175^{0}C:				
Minimum torque M_{min}, dN m	1.38	1.16	1.16	1.09
Maximum torque M_{max}, dN m	10.56	9.83	9.69	10.24
The difference in torque ΔM, dN m	9.18	8.67	8.53	9.15
Time of the beginning of vulcanization t_{S2}, min	0.87	0.96	0.97	0.94
Optimal vulcanization time t_{90}, min	2.79	3.46	3.16	3.35
Vulcanization rate R_V, min^{-1}	5.12	3.8	4.19	4.41
Conditional stress at 300% elongation, MPa	13.2	12.8	12,9	13,2
Conditional tensile strength, MPa:				
at 25^{0}C	19.5	17.0	21.0	22,4
after aging at 100^{0}C for 48 hours	16.5	14.1	17.0	19,5
Relative elongation at break, %:				
at 25^{0}C	430	405	440	420
after aging at 100^{0}C for 48 hours	290	295	330	310
Tear resistance, kN m^{-1}	26.5	25.4	27.3	27.0
Resistance to multiple deformations, thousand cycles:				
at 25^{0}C	12.0	13.5	21.5	20.5
after aging at 100^{0}C for 48 hours	5.9	10.0	13.2	12.0

The addition of composites based on 101K resin improves the complex of physical-mechanical and dynamic properties of vulcanizates as well. A sulfur additive is more effective.

It should be noted that the influence of the 101K resin-based additive practically does not change the mechanical loss tangent of elastomeric compositions, which may indicate a smaller influence of the said additives on the mobility of macromolecular structures compared to Octofor 10S resin-based additives.

Based on the data obtained, modifying additives were used to treat the surface of crushed vulcanizate (CV). The crushed vulcanizate prepared by mechanical grinding of worn tires and rubber products was studied at positive temperatures.

Tyre crushed vulcanizate (rubber crumb) met the technical requirements of TS 38 108035-92 and TS 6-25521987.010.2000. The particle size of the crushed vulcanizate ranged from 0.8 to 5–8 mm. Thermomechanic reclaimed rubber of RSHT, RSHTN brands, according to TS 6-25521987.009-2000, was used for comparison. The surface was processed on rolled equipment in accordance with the modes specified in work [10].

The size distribution of crushed volcanic particles was carried out using the filler dispersion analyzer of the same firm and the analysis was carried out according to the methods specified in study [11].

The introduction of various ingredients into elastomer materials causes changes in the morphological structure of the matrix. These changes when using crushed vulcanizates were determined by the rheometric curves in accordance with recommendation given elsewhere [12].

The efficiency of the surface treatment process was evaluated based on the results of analysis of physical-mechanical properties of vulcanizates containing processed CV 25 and 50 parts per 100 parts of rubber, these being chosen as the most appropriate concentration of the crushed vulcanizate. This choice is due to the fact that depending on the content of the CV, there is a change in the fracturing mechanism of the composition: with a small rubber crumb content, the strength of the system will be mainly determined by the strength of the elastomeric matrix, and then there is the cohesiveness of the material. When the dosage is increased to more than 25 parts by weight, CV particles are capable of forming a continuous phase and the nature of the fracture is close to adhesive and depends on the strength of the bond on the interphase boundary. CV processed on modifier-free roller equipment was applied for comparison. The surface treatment of crushed vulcanizates carried out on laboratory rolls in the presence of research modifiers revealed no technical difficulties. Given

Table 4. Rheometer performance at 175^{0}C for elastomeric compositions containing crushed vulcanizates processed with various resins

Indexes	Without CV	Regenerate RSHT, parts by weight		Type of modifier and content of crushed vulcanizate, parts by weight per 100 parts of rubber					
				unprocessed		alloy Octofore 10S with TEA-FAME (1:1)		alloy of resin 101K with TEA-FAME (2:1) in the presence of sulfur	
		20	50	20	50	20	50	20	50
Rheometer characteristics at 175^{0}C:									
Minimum torque, M_{min}, dN m	2.60	3.13	3.44	3.25	3.56	3.15	3.48	3.08	3.58
Maximum torque, M_{max}, dN m	18.35	15.93	14.9	14.71	14.00	17.31	15.37	17.46	16.28
Time of the beginning of vulcanization T_2, min	0.72	0.82	0.77	0.86	0.88	0.85	0.87	0.85	0.88
Optimal vulcanization time t_{90}, min	3.11	3.01	2.92	3.32	2.76	3.21	2.86	3.21	2.98
Vulcanization rate R_V, min^{-1}	41.84	45.66	46.51	40.65	53.19	42.37	50.25	42.37	47.62
Mechanical loss tangent at M_{min}, $tg\delta_{min}$	0.927	0.856	0.779	0.866	0.798	0.832	0.724	0.828	0.740
Mechanical loss tangent at M_{max}, $tg\delta_{max}$	0.160	0.179	0.180	0.165	0.160	0.157	0.154	0.156	0.155

the appearance and influence of processed CV on the properties of model systems, 4–5 parts composites per 100 parts of crushed vulcanizate can be considered as an optimal content of additives. In order to address the practical application of treated CV, it has been added to rubber mixtures that are intended for the manufacture of molded rubber products, such as shock absorbers.

Table 4 shows the results concerning the studies of elastomeric compositions performed by a rheometer. It has been found that the addition of treated CV reduces the viscosity of rubber compounds as compared with the untreated CV, and this makes it possible to reduce energy consumption in the manufacture and processing of elastomeric compositions. There is also an increase in the maximum torque, which indicates an increase in the degree of crosslinking of vulcanizates with processed vulcanizate in comparison with both unprocessed CV and regenerated CV.

Changes in the physical-mechanical properties of elastomer compositions containing processed crushed vulcanizates as compared with the unprocessed compositions are shown in Table 5.

The analysis of mechanical losses with repeated deformation (Fig. 3) showed that the mechanical loss tangent is reduced when the processed CV is used in comparison with the unprocessed one. As a consequence, heat formation will be significantly reduced during dynamic loading, which will ensure high operational reliability of rubber products, such as shock absorbers.

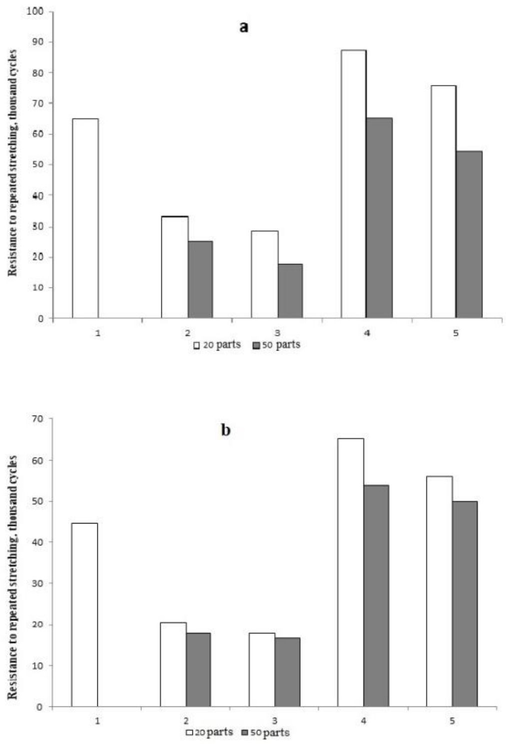

Fig. 3. Resistance to repeated stretching of tyres containing 20 parts and 50 parts crushed vulcanizate processed with the investigated modifying additives. (Reference: Table 4)

Conclusions

Thus, the influence of modifying systems based on phenol-formaldehyde resins with an amine-containing composition on the properties of

Table 5. Changes of the properties of elastomer compositions containing processed crushed vulcanizates as compared with unprocessed vulcanizates

Indexes	Type of modifier for processing crushed vulcanizate and CV content, parts by weight			
	alloy Octofore 10S with TEA-FAME (1:1)		alloy of resin 101K with TEA-FAME (2:1) in the presence of sulfur	
	20 parts CV	50 parts CV	20 parts CV	50 parts by weight CV
Changing the tensile strength, %:				
at 25^0C	+40.0	+39.0	+36.1	+35.0
after aging 100^0C×72 hours	+26.6	+54.1	+28.1	+58.3
Changing the elongation at break, %				
at 25^0C	+18.1	+21.6	+11.0	+14.5
after aging 100^0C×72 hours	+38.1	+36.2	+32.3	+34.8

model elastomeric compositions based on styrene-butadiene rubber SBR-1705 HI-AR was considered. An improvement in the complex of technological properties of rubber compounds and mechanical properties of vulcanizates using research alloys has been established. The addition of this type of additive reduces the minimum torque of rubber mixtures by 20%, which indicates the plasticizing effect of 101K resin-based additives. There is a 15% increase in the conditional tensile strength both under normal conditions and after aging as compared with the control mixture.

The established influence of modifiers on the complex properties of elastomeric compositions can be further used when choosing the direction of the industrial application of experimental additives in the rubber mixture compositions.

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Застосування модифікуючих систем на основі фенолоформальдегідних смол і продуктів переробки жировмісної сировини в еластомерних композиціях

Л.С. Голуб, Ю.М. Ващенко, М.В. Андріянова

Одним з напрямків підвищення якості еластомерних матеріалів є їх модифікація активними сполуками. Як модифікуючі добавки розглянуті системи з використанням сірковмісної фенолоформальдегідної смоли октофор 10S та смоли 101K разом з аміновмісною сполукою, що отримана взаємодією етаноламінів із метиловим естером жирних кислот, сировиною був курячий жир. Дослідження проводили в модельних полімерних композиціях на основі сополімеру бутадієну та стиролу. Аналіз комплексу властивостей матеріалів показав, що ефективним є застосування 3–4 мас.ч. добавок при співвідношенні компонентів 2:1. Використання механічної суміші компонентів за ефективністю дії значно поступається добавці, яка отримана шляхом сплавлення компонентів. Показано, що при цьому суттєво підвищується динамічна витривалість гум. Встановлено значне зниження тангенсу кута механічних втрат при застосуванні композиції з використанням смоли октофор 10S, що дає змогу зменшити теплоутворення при динамічних навантаженнях та збільшити ресурс великогабаритних виробів, наприклад, шин. Встановлені закономірності впливу модифікаторів на комплекс властивостей еластомерних композицій можуть бути застосовані при виборі напрямку використання дослідних добавок у складі гумових сумішей промислового призначення. Встановлено, що додавання подрібненого вулканізату, обробленого модифікуючими добавками, знижує в'язкість гумових сумішей у порівнянні з використанням необробленого вулканізату; це дає змогу знизити енергоспоживання при виготовленні та переробці еластомерних композицій.

Ключові слова: модифікатор, фенолоформальдегідна смола, курячий жир, еластомер, композиція, ресурсозбереження.

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One of the ways to improve the quality of elastomeric materials is to modify them with active compounds. As modifying additives in this work, the systems were considered which are based on sulfur-containing phenol-formaldehyde resins Octofor 10S and 101K together with an amine-containing compound produced by the interaction of ethanol amines with the methyl ester of fatty acids, the raw material being chicken fat. Study was carried out using model polymer compositions based on butadiene and styrene copolymer. Analysis of the complex of properties showed that it is effective to use 3–4 parts by weight of additives at the ratio of components 2:1. Mechanical mixture of components is inferior to the additive produced by the components fusion in this respect. Moreover, the dynamic strength of rubber is significantly increased. A significant reduction in the mechanical loss tangent was established in the composition with the use of Octofor 10S resin. That allows reducing heat formation at dynamic loads and increasing the service life of large articles, such as tyres. The patterns of the influence of modifiers on the complex of properties of elastomeric compositions are useful when choosing the direction of industrial application of the additives under consideration in the composition of rubber compounds. The addition of crushed vulcanizate treated with modifying additives was found to reduce the viscosity of rubber compounds as compared with the use of untreated vulcanizate; this decreases the energy consumption in the manufacture and processing of elastomeric compositions.

Keywords: modifier; phenol-formaldehyde resin; chicken fat; elastomer; composition; resource saving.

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