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Study of the influence of dispersed fillers on properties of rubber for gaskets of rail fastening

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Abstract. The effect of dispersed fillers (NDP-D-400 and NDP-230 diatomites, microquartz and magnesium hydrosilicate) on the rheometric, physical-mechanical, operational and dynamic (sound-absorbing) properties of rubber based on general and special purpose caoutchoucs used for the manufacture of rail fastening gaskets has been studied. The rubber mixture was prepared on laboratory rollers LB 320 160/160, and then vulcanized in a P-V-100-3RT-2-PCD press. For the rubber compound, the vulcanization characteristics were studied, and for the vulcanizates, the physical and mechanical properties, their changes after exposure to aggressive environments and dynamic performance. It has been established that dispersed fillers improve the technological properties of the rubber mixture, increase the physical and mechanical, dynamic performance and resistance of rubber to aggressive media. It is shown that vulcanizates containing diatomites NDP-230 and NDP-D-400 in an amount of 5.0 phr, are characterized by improved physical, mechanical, operational and dynamic properties.
Keywords: diatomites; caoutchoucs; rubber; rheometric; physical-mechanical; operational and dynamic properties

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Introduction

Railways during operation create increased levels of vibration and noise caused by the interaction of the rolling stock and the railway track [1–7]. The noise generated during the operation of railway transport has its negative impact on human health [8-14]. To solve this problem, rubber gaskets for rail fastenings are used [15–20]. These gaskets are made from noise-absorbing rubbers based on a combination of general and special purpose rubbers, as well as directional functional ingredients [21–26]. In [27,28], trans-polynorbornene and silica filler Silica 1165 were used as such ingredients. It is known [29–33] that dispersed fillers also improve the noise-absorbing properties of composite materials. In this regard, in this article, the effect of dispersed fillers (NDP-D-400 and NDP-230 diatomites, microquartz and magnesium hydrosilicate) on the properties of rubber used for the manufacture of rail fasteners was studied.

Materials and Method

The studied rubber mixture included the following caoutchoucs and ingredients: butadiene-methylstyrene SKMS-30ARK, butadiene-nitrile SKN-2655 and *cis*-isoprene SKI-3, vulcanization accelerator - N-cyclohexyl-2-benzothiazolesulfenamide; vulcanizing agents -

sulfur, tetramethylthiuram disulfide and N,N'-dithiodimorpholine; vulcanization activators - zinc oxide, stearic acid; stabilizers - acetonanil N, protective wax ZV-P, N-phenyl-N'- isopropyl-p-phenylenediamine; dispersant - Hepsol HKP; softeners - rosin, industrial oil I-12A; scorch retarder - N-nitrosodiphenylamine; fillers - kaolin, carbons black N 220 and P 514, silicon dioxide Zeosil 1165 MP and *trans*-polynorbornene. As dispersed fillers, diatomites NDP-D-400 and NDP-230 were used (contain 84-87 % SiO_2, 5.5-6.0 % Al_2O_3, 2.5-3.0 % Fe_2O_3 and 0.6 % CaO), microquartz and magnesium hydrosilicate. Diatomite NDP-D-400 is a beige powder with a pink tint, particle size 25-30 μm, density 2.14 g/cm^3, porosity 82.7 %, specific surface area 0.8-1.1 m^2/g. Diatomite NDP-230 is a beige powder, particle size 5 μm, density 2.06 g/cm^3, porosity 89.3 %, specific surface area 3.0-3.3 m^2/g. Microquartz is a gray powder, mass fraction of silicon dioxide is not less than 98 %, specific surface area is 16.8 m^2/g, density is 2.55 g/cm^3. Magnesium hydrosilicate composition $3MgO \cdot 4SiO_2 \cdot H_2O$ is a dark gray powder with a scaly particle size of 5-20 μm, density 2.60-2.80 g/cm^3. Kaolin was a beige powder with a particle size of 10–20 μm, a density of 2.5–2.63 g/cm^3, and a specific surface area of 10–24 m^2/g.

The rubber mixture was prepared on an LB 320 160/160 laboratory roller at 60–70 °C for 25 min. The vulcanization characteristics of the rubber compound were studied on a Mon Tech MDR 3000 Basic rheometer at 143 °C for 40 min in accordance with ASTM D2084-79. Standard samples for determining physical and mechanical properties were vulcanized at a temperature of 143 °C for 20 min in a P-V-100-3RT-2-PCD type vulcanizing press. The main characteristics of the vulcanizates were determined according to the standards in force in the rubber industry: elastic-strength properties were determined according to GOST 270-75; hardness - according to GOST 263-75; tear resistance - according to GOST 262-79; change in conditional tensile strength, relative elongation at break and hardness after aging in air - according to GOST 9.024-74; change in conditional tensile strength, relative elongation at break and hardness after exposure to aggressive hydrocarbon media - according to GOST 9.030-74 (method B); change in mass after exposure to aggressive media - according to GOST 9.030-74 (method A). The dynamic parameters of the vulcanizates of the rubber compound were studied at 30 °C on a Metravib VHF 104 dynamic mechanical analyzer with a degree of deformation of 0.01 %, a frequency of 1000 Hz in the "tension-compression" deformation mode.

Results and Discussion

The possibility of partial and complete replacement of the fillers (kaolin, carbons black N 220 and P 514 and Zeosil 1165 MP silicon dioxide) included in the composition of the rubber mixture with dispersed fillers: NDP-D-400 and NDP-230 diatomites, microquartz and magnesium hydrosilicate was previously studied. As a result, the efficiency of the equal weight replacement of kaolin with the listed particulate fillers in the amount of 5.0 phr (parts per hundred parts of rubber), which was taken as the basis for further research. Table 1 shows the investigated options for the rubber mixture.

The first variant of the rubber mixture was made using kaolin. In the second-fifth variants of the rubber compound, an equal mass replacement of kaolin was carried out for diatomites NDP-D-400 and NDP-230, microquartz and magnesium hydrosilicate, respectively.

To establish the rheometric characteristics of the rubber mixture, the vulcanization kinetics of its various variants was studied. Figure 1 shows the resulting vulcanization curves.

Based on these curves, the rheometric properties were determined, which are given in Table 1. From the data in Table 1 it follows that the equal-mass replacement of kaolin with the above fillers leads to an increase in the maximum torque, the vulcanization start time and a decrease in the minimum torque, the optimal vulcanization time of the rubber mixture. The rubber compound of the third variant has improved technological properties, which is characterized by the lowest value of the minimum torque and the longest vulcanization start time. The results of studies of the physical and mechanical characteristics of vulcanizates of various options for the rubber mixture are given in Table. 2.

Table 1. Variants of the studied rubber mixture

Fillers	Variants of the rubber mixture				
	1	2	3	4	5
Kaolin, phr	5.0	–	–	–	–
Diatomite NDP-D-400, phr	–	5.0	–	–	–
Diatomite NDP-230, phr	–	–	5.0	–	–
Microquartz, phr	–	–	–	5.0	–
Magnesium hydrosilicate, phr	–	–	–	–	5.0
Rheometric properties of the rubber mixture					
S_{max}, dN·m	18.30	18.43	18.53	18.48	19.03
S_{min}, dN·m	3.62	3.47	3.44	3.61	3.50
t_s, min	5.03	5.59	5.68	5.64	5.51
t_{90}, min	16.26	15.89	15.69	15.47	15.52
Note: S_{max} – maximum torque; S_{min} – minimum torque; t_s – start time of vulcanization; t_{90} – optimum vulcanization time.					

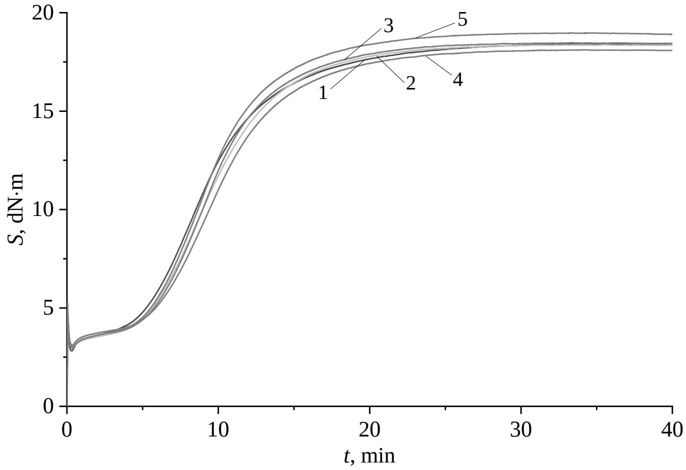

Fig. 1. Vulcanization curves of various variants of the rubber compound at 143 °C using various fillers: 1 – kaolin, 2 – diatomite NDP-D-400, 3 – diatomite NDP-230, 4 – microquartz, 5 – magnesium hydrosilicate

Table 2. Physical and mechanical properties of vulcanizates

Parameter	Variants of the rubber mixture				
	1	2	3	4	5
f_{100}, MPa	3.6	3.8	4.1	3.9	3.8
f_p, MPa	12.8	14.4	14.8	14.4	13.1
ε_p, %	350	400	380	370	400
H, Shore A units	70	70	70	68	70
B, kN/m	43	48	51	47	45
Note: f_{100} – conditional stress at 100 % stretching; f_p – conditional tensile strength; ε_p – elongation at break; H – hardness; B – tear resistance.					

Based on these curves, the rheometric properties were determined, which are given in Table 1. From the data in Table 1 it follows that the equal-mass replacement of kaolin with the above fillers leads to an increase in the maximum torque, the vulcanization start time and a decrease in the minimum torque, the optimal vulcanization time of the rubber mixture. The rubber compound of the third variant has improved technological properties, which is characterized by the lowest value of the minimum torque and the longest vulcanization start time. The results of studies of the physical and mechanical characteristics of vulcanizates of various options for the rubber mixture are given in Table. 2.

From the data in Table 2 it follows that the equal-mass replacement of kaolin with dispersed fillers contributes to an increase in the conditional stress at 100% stretching, conditional tensile strength, relative elongation at break, and tear resistance of vulcanizates. The vulcanizate of the third version of the rubber compound containing NDP-230 diatomite is characterized by the highest strength and tear resistance.

Further, the operational properties were studied - changes in the physical and mechanical parameters and hardness of vulcanizates after aging in air and exposure to SZhR-1 at a temperature of 100 ºC for 24 hours (see Table 3).

Table 3. Changes in physical and mechanical properties and hardness of vulcanizates after aging in air and in SZhR-1

Parameter	Variants of the rubber mixture				
	1	2	3	4	5
After air aging					
Δf_p, %	-18.6	-18.4	-15.2	-17.5	-15.8
$\Delta \varepsilon_p$, %	-34.3	-32.5	-30.2	-32.4	-33.5
ΔH, Shore A units	+4	+3	+2	+1	+2
After exposure to SZhR-1					
Δf_p, %	-48.4	-42.4	-39.6	-43.1	-43.5
$\Delta \varepsilon_p$, %	-32.1	-29.5	-26.3	-30.1	-29.4
ΔH, Shore A units	-23	-20	-17	-16	-19
Note: Δf_p, $\Delta \varepsilon_p$, – relative change in tensile strength and elongation at break, respectively; ΔH – change in hardness.					

From the data in Table 3 shows that after daily thermal-oxidative aging in air, a decrease in physical and mechanical parameters and an increase in the hardness of vulcanizates are observed. Moreover, the replacement of kaolin with dispersed fillers leads to smaller changes in the physical and mechanical properties and hardness of the vulcanizates. The vulcanizate containing NDP-230 diatomite has the least changes in physical and mechanical properties. Replacing kaolin with dispersed fillers also leads to smaller changes in the physical and mechanical parameters and hardness of vulcanizates after their daily holding in SZhR-1 heated to 100 °C. Moreover, in SZhR-1, the hardness of the vulcanizates is significantly reduced due to the loosening of the network structure of the vulcanizates with a standard mixture of hydrocarbons.

Figures 2-4 show the results of a study of the change in the mass of vulcanizates after their holding at a temperature of 23 °C for 24 hours in various media: industrial oil I-20A, distilled water and SZhR-1.

As can be seen from Figs. 2-4, the replacement of kaolin with dispersed fillers leads to a decrease in the change in the mass of vulcanizates after their daily exposure in all three media. In hydrocarbon media (SZhR-1 and industrial oil I-20A), changes in the mass of vulcanizates are higher than in distilled water. The smallest change in mass after exposure in all environments has a vulcanizate rubber mixture containing NDP-230 diatomite. In Table 4 shows the results of studies of the dynamic properties of vulcanizates.

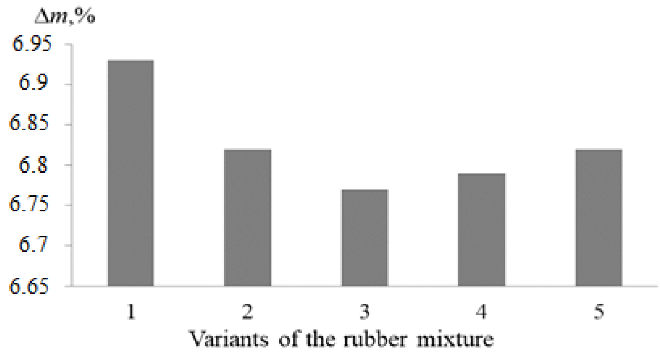

Fig. 2. Change in mass of vulcanizates after exposure in industrial oil I-20A

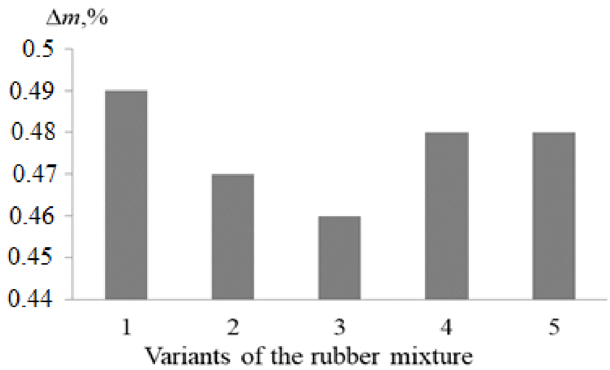

Fig. 3. Change in mass of vulcanizates after exposure in distilled water

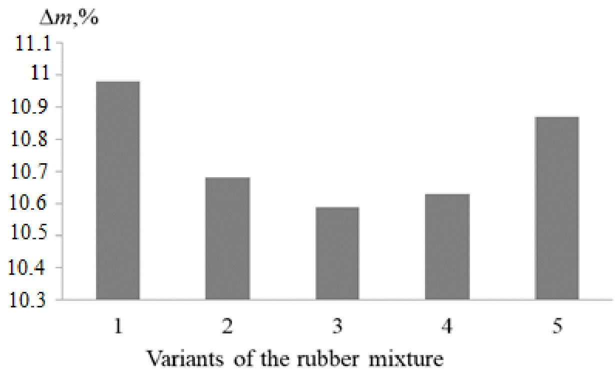

Fig.4. Change in mass of vulcanizates after exposure in SZhR-1

The data in Table 4 show that the equal-mass replacement of kaolin with dispersed fillers in the composition of the rubber mixture leads to an increase in the tan delta and the storage modulus of the vulcanizates. An increase in the storage modulus of vulcanizates indicates an increase in their physical and mechanical properties, which is consistent with the results given in Table 2 for vulcanizates containing particulate fillers. It is known [34,35] that polymer composite materials with increased values of the tan delta (loss factor) have improved dynamic properties. According to [36–38], such polymers should contain fillers with large particles and a low specific surface area. These conditions are met by the vulcanizate of the second variant of the rubber mixture containing NDP-D-400 diatomite, which, compared to other dispersed fillers used, consists of larger particles and has a smaller specific surface area. For the vulcanizate of the third variant of the rubber compound containing NDP-230 diatomite, the value of the tan delta is less than for the vulcanizate of the second variant of the rubber mixture, however, this value is sufficient for rail fasteners. The second and third rubber options are similar in rheometric, physical, mechanical and operational properties. Therefore, diatomites NDP-D-400 and NDP-230 can equally be used in the rubber composition for rail fasteners.

Table 4. Dynamic properties of rubber compound vulcanizates

Parameter	Variants of the rubber mixture				
	1	2	3	4	5
tgδ	0.230	0.246	0.241	0.235	0.238
$E'\cdot10^{-7}$, Pa	5.19	5.20	5.23	5.21	5.22
Note: *tgδ* – tan delta (loss factor); *E′* – storage modulus.					

Conclusions

The effect of dispersed fillers (NDP-D-400 and NDP-230 diatomites, microquartz and magnesium hydrosilicate) on the rheometric properties of the rubber mixture, physical and mechanical, operational and dynamic performance of rubber based on general and special purpose caoutchoucs (isoprene, butadiene-methylstyrene and nitrile butadiene). It has been established that rubber containing diatomites NDP-230 and NDP-D-400 in the amount of 5.0 phr, can be recommended for the manufacture of rail fastening gaskets with improved physical, mechanical, operational and dynamic properties.

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