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# Influence of dispersed inorganic fillers on the properties of vulcanized rubber based on ethylene propylene diene monomer rubber SKEPT-40

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**ABSTRACT**

The influence of various powder inorganic fillers (carbon black P 803, P 324 and N 220, silica ZC-120) on the rheometric properties of the rubber compound, physico-mechanical and performance properties of vulcanized rubber based on ethylene propylene diene monomer rubber SKEPT-40, used for rubberizing metal surfaces, was studied. The rubber compound under study based on the above rubber included a vulcanizing agent (sulfur), vulcanization accelerators (2-mercaptophenylthiazole and tetramethylthiuram disulfide), vulcanization activators (zinc white and stearic acid) softener (industrial oil I-8A). It was found that vulcanized rubber with a content of 60.0 parts per hundred parts of rubber (phr) of carbon black N 220 has high physico-mechanical properties, the smallest changes in tensile strength, elongation at break and weight after exposure to aggressive acid-base environments and good frost resistance.

**KEYWORDS**

carbon black P 803, P 324 and N 220 • silica ZC-120 • ethylene propylene diene monomer rubber SKEPT-40 vulcanized rubber • rheometric • physico-mechanical and performance properties

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## Introduction

It is known [1–8] that dispersed inorganic fillers make it possible to regulate the technological properties of rubber compounds and the physico-mechanical properties of vulcanizates. In [1], the effect of Zeosil 1165 PM silicon dioxide modified with the organosilane coupling agent bis-(3-triethoxysilylpropyl) tetrasulfane (TESPT) on the rheometric properties of a rubber compound and the physico-mechanical properties of vulcanized rubber based on natural rubber (NR) was investigated. It was shown that the introduction of modified silicon dioxide into the rubber compound leads to an improvement in the rheometric, rheological of the rubber compound, and physico-mechanical properties of the vulcanized rubber. In [2], it was shown that binary fillers with a silica to graphite powder ratio of 1:1 in an amount of only 20.0 phr provide excellent mechanical properties and improved resistance to thermooxidative aging of composites based on NR compared to single fillers. In [4], the influence of hollow polymer microspheres Expancel 909 DU 80, Expancel 043 DUT 80, Expancel 920 DET 40d25, Expancel 920 DUT 40, Expancel 930 MB 120 and Lega Foam 120 MB in an amount of 5.0 phr on the rheometric properties of the rubber compound, the physico-mechanical and performance properties of oil-resistant rubber was studied. It was found that



vulcanized rubber containing Expance 043 DUT 80 microspheres has the best physico-mechanical properties, high wear resistance and resistance to the effects of aggressive hydrocarbon environments. In [8], the influence of the amount of 5.0, 10.0, 15.0 and 20.0 phr was considered. The effect of montmorillonite on the physico-mechanical properties of vulcanizates based on a mixture of natural rubber/styrene-butadiene rubber in a ratio of 50/50 phr was studied. It was shown that the tensile strength of the filled vulcanized rubber reached its maximum value (11.2 MPa) when the montmorillonite content was 15.0 phr.

As a rule, fillers are used to improve the elastic-strength and performance parameters and the chemical resistance of vulcanized rubbers to the action of aggressive acidic and alkaline environments [9–22]. In the rubber industry, rubber compounds based on ethylene propylene diene monomer rubbers are of practical interest, which are characterized by a balanced resistance to the effects of temperatures [23] and oxidation by ozone [24–27]. Despite the achieved results, in connection with increasing requirements, it remains important to develop rubbers that are resistant to temperature effects and highly resistant to alkaline and acidic environments. To eliminate these shortcomings, dispersed powder fillers are introduced into rubber compounds, of which the effective are carbon black [28–34] and silica [35–38]. Therefore, research on the creation of high-quality rubbers based on ethylene propylene diene monomer rubbers, resistant to the effects of temperatures and aggressive environments, using dispersed inorganic fillers is relevant. In this article, the influence of carbon black P 803, P 324, N 220, and silica ZC-120 as fillers on the rheometric properties of the rubber compound, physico-mechanical, and performance properties of vulcanized rubber based on ethylene propylene diene monomer rubber SKEPT-40, often used in the rubber industry, was studied [39,40].

## Material and Methods

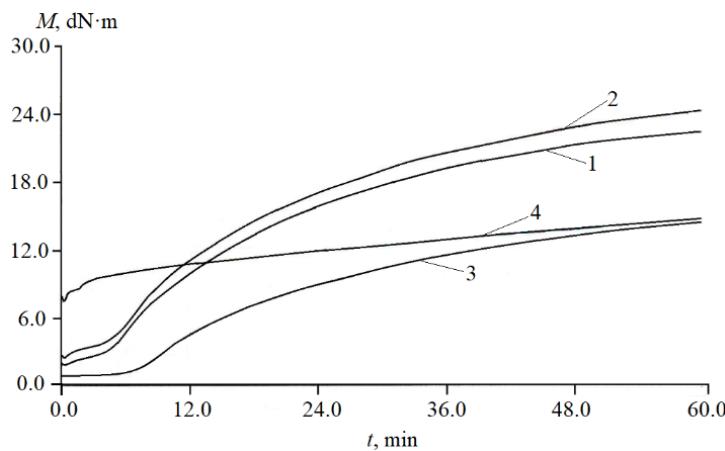
The rubber compound included ethylene propylene diene monomer rubber SKEPT-40 with a Mooney viscosity  $ML_{1+4}$  (100 °C) = 38, ethylene and dicyclopentadiene content of 56 and 6 wt. %, as well as the following ingredients: vulcanizing agent (sulfur), vulcanization accelerators (2-mercaptopbenzothiazole and tetramethylthiuram disulfide), vulcanization activators (zinc white and stearic acid), softener (industrial oil I-8A). Carbon black P 324, N 220 and P 803, as well as silica ZC-120 were used as fillers. Carbon black P 324 and N 220 are powders of a deep black color with a particle size of 0.028–0.036 and 0.024–0.033  $\mu\text{m}$ , bulk density of 340 and 355  $\text{kg/m}^3$ , a specific surface area of 75–82 and 106–114  $\text{m}^2/\text{g}$ , and a dibutyl phthalate absorption of  $100 \pm 5$  and  $113 \pm 7 \text{ cm}^3/100 \text{ g}$ , respectively. Carbon black P 803 is a dark gray powder with a particle size of 9–320  $\mu\text{m}$ , bulk density of 320  $\text{kg/m}^3$ , specific surface area of 14–18  $\text{m}^2/\text{g}$ , dibutyl phthalate absorption of  $83 \pm 7 \text{ cm}^3/100 \text{ g}$ . Silica ZC-120 is a white powder with a particle size of 0.005–0.015  $\mu\text{m}$ , silicon dioxide content of 97 %, specific surface area of 105–135  $\text{m}^2/\text{g}$ , a pH (5 % aqueous suspension) of 6–8.

The rubber compound was prepared on LB 320 160/160 laboratory mills at a roll surface temperature of 60–70 °C for 25 min. The rheometric properties of the rubber compound were studied on an MDR 3000 Basic rheometer at 150 °C for 60 min in

accordance with ASTM D2084-79. To determine the physico-mechanical properties, the rubber compound was vulcanized at a temperature of 150 °C for 60 min in a PV-100-3RT-2-PCD vulcanization press. The elastic strength properties were determined according to [42] Shore A hardness was measured in accordance with [43]. Rebound elasticity was determined according to [44]. Relative residual compression deformation (RCD) was determined according to [45]. Change in tensile strength, relative elongation at break and hardness after thermal-oxidative aging in air was determined according to [46]. The change in conditional tensile strength, relative elongation at break and hardness after exposure to liquid aggressive environments was determined according to [47]. The change in mass after exposure to liquid aggressive environments was found according to [47]. The frost resistance coefficient for elastic recovery after compression was determined according to [48].

## Results and Discussion

Four variants of rubber compounds were studied, containing carbon black P 324, N 220, P 803 and silica ZC-120 in the amount of 60.00 phr (traditional formulation). For each variant of rubber mixture, vulcanization curves were recorded at 150 °C for 60 min. The research results are shown in Fig. 1.



**Fig. 1.** Vulcanization curves of rubber compound (curve numbers correspond to variant numbers)

The rheometric parameters determined on the basis of the obtained vulcanization curves are given in Table 1. It follows from Table 1 that for variant 3 of the rubber compound containing P 803 carbon black as a filler, a significant increase in the scorch time is observed, which may negatively affect the productivity and quality of the final product. At the same time, variant 4 is characterized by the lowest scorch time. The difference between the torques is directly proportional to the degree of chemical crosslinking of the vulcanizate. Thus, the vulcanizate of variant 2 containing N 220 carbon black as a filler should be characterized by a high degree of crosslinking and have better strength properties.

After vulcanization of the rubber compound, the main physico-mechanical properties of the vulcanizates were determined and are given in the Table 1. The Table shows that variant 2 demonstrates the better physico-mechanical characteristics compared to other variants of vulcanizates, which is explained by the smaller particle

**Table 1.** Results of the study of the properties of various variants of rubber compounds and vulcanizates

Fillers, indicators	Variants			
	1	2	3	4
Carbon black P 324, phr	60.0	–	–	–
Carbon black N 220, phr	–	60.0	–	–
Carbon black P 803, phr	–	–	60.0	–
Silica ZC-120, phr	–	–	–	60.0
<i>Rheometric properties of rubber compound</i>				
$M_H$ , dN·m	22.51	24.33	14.37	17.51
$M_L$ , dN·m	2.24	3.11	1.02	8.65
$\Delta M$ , dN·m	20.27	21.22	13.35	8.86
$t_s$ , min	3.31	3.27	8.57	0.50
$t_{90}$ , min	43.17	43.79	46.50	50.36
<i>Physico-mechanical properties of vulcanizates</i>				
$f_p$ , MPa	14.6 ± 0.7	14.6 ± 0.6	6.0 ± 0.3	5.4 ± 0.3
$\varepsilon_p$ , %	356 ± 17	362 ± 19	294 ± 14	682 ± 32
$H$ , units Shore A	72 ± 1	77 ± 1	62 ± 1	68 ± 1
$S$ , %	36 ± 1	37 ± 1	54 ± 1	40 ± 1
RCD at 25 % compression (125 °C × 24 h), %	32.0 ± 1.1	20.0 ± 0.7	30.0 ± 0.9	41.0 ± 1.4
<i>Changes in the physico-mechanical properties of vulcanizates after aging in air (125 °C × 24 h)</i>				
$\Delta f_p$ , %	+(1.0 ± 0.1)	+(1.0 ± 0.1)	+(1.0 ± 0.1)	+(7.0 ± 0.3)
$\Delta \varepsilon_p$ , %	-(15.0 ± 0.6)	-(9.0 ± 0.4)	-(30.0 ± 1.3)	-(52.0 ± 2.1)
$\Delta H$ , units Shore A	+(9 ± 1)	+(7 ± 1)	+(8 ± 1)	+(10 ± 1)
<i>Changes in physico-mechanical properties and mass of vulcanizates in a 20 % NaOH solution (23 °C × 24 h)</i>				
$\Delta f_p$ , %	+(6.0 ± 0.2)	+(3.0 ± 0.1)	+(5.0 ± 0.2)	+(10.0 ± 0.4)
$\Delta \varepsilon_p$ , %	+(24.0 ± 0.8)	+(10.0 ± 0.3)	+(18.0 ± 0.6)	+(14.0 ± 0.5)
$\Delta H$ , units Shore A	+(1 ± 1)	+(3 ± 1)	-(1 ± 1)	+(3 ± 1)
$\Delta m$ , %	0.17 ± 0.01	0.13 ± 0.01	0.18 ± 0.01	0.25 ± 0.01
<i>Changes in physico-mechanical properties and mass of vulcanizates in a 20 % HCl solution (23 °C × 24 h)</i>				
$\Delta f_p$ , %	+(10.0 ± 0.4)	+(2.0 ± 0.1)	-(5.0 ± 0.2)	+(3.0 ± 0.1)
$\Delta \varepsilon_p$ , %	+(12 ± 0.4)	+(6.0 ± 0.2)	-(54.0 ± 2.1)	+(56.0 ± 2.3)
$\Delta H$ , units Shore A	+(1 ± 1)	-(2 ± 1)	-(1 ± 1)	-(7 ± 1)
$\Delta m$ , %	0.43 ± 0.01	0.42 ± 0.01	0.46 ± 0.01	0.45 ± 0.01
<i>Frost resistance of vulcanizates</i>				
$K_B$	0.32 ± 0.01	0.34 ± 0.01	0.40 ± 0.01	0.31 ± 0.01

Note:  $M_H$  is maximum torque;  $M_L$  is minimum torque;  $\Delta M$  is difference between the maximum and minimum torques;  $t_s$  is scorch time;  $t_{90}$  is optimum cure time;  $f_p$  is tensile strength;  $\varepsilon_p$  is elongation at break;  $H$  is hardness;  $S$  is rebound elasticity; RCD is relative residual compression deformation;  $\Delta f_p$ ,  $\Delta \varepsilon_p$  and  $\Delta m$  are relative changes in the tensile strength, elongation at break and mass;  $\Delta H$  is change in hardness;  $K_B$  is the coefficient of frost resistance by elastic recovery after compression.

size and larger specific surface area of the N 220 carbon black used in this variant compared to P 324 and P 803 carbon black, as well as by the poor dispersion of ZC-120 silica in the rubber matrix [41]. At the same time, vulcanizates of variants 3 and 4 have low values of conventional tensile strength and hardness, which limits their use in conditions where high strength and reliability of materials are required. However, variant 4 is characterized by a good indicator of elongation at break. Vulcanizate of variant 2, containing carbon black N 220 as a filler, is characterized by the lowest value of relative residual compression deformation, which indicates its high elastic properties.

The table also shows the results of the study of changes in the physico-mechanical properties and hardness of vulcanizates after thermal aging in air. As can be seen, the stable physico-mechanical properties are found in the vulcanizate of variant 2 of the rubber compound containing carbon black grade N 220. It shows a slight change in the tensile strength, as well as in the elongation at break after thermal aging in air.

The changes in the physico-mechanical properties and weight of the vulcanizates after exposure to aggressive environments were further investigated: 20 % aqueous solutions of NaOH and HCl at 23 °C for 24 h (see Table 1). The data obtained show that the best resistance in a 20 % HCl solution is demonstrated by the vulcanizate of variant 2, which is characterized by the smallest changes in strength and elongation.

Table 1 also shows the results of the study of the frost resistance of the vulcanizates, determined by their elastic recovery after a single 20 % compression for 5 min at -50 °C. As can be seen, all vulcanizates have high frost resistance coefficient values, which indicates the ability to maintain elasticity and physico-mechanical properties at extremely low temperatures, allowing them to be used in cold climates.

## Conclusions

The objects of the study were a rubber compound and vulcanized rubber on its basis, obtained by sulfur vulcanization of ethylene propylene diene monomer rubber SKEPT-40 in the presence of vulcanization accelerators (2-mercaptophenothiazole and tetramethylthiuram disulfide), vulcanization activators (zinc white and stearic acid), a softener (industrial oil I-8A) and various inorganic fillers (carbon black P 803, P 324, N 220 and silica ZC-120) in an amount of 60.0 phr. The rubber compound was prepared on laboratory rollers LB 320 160/160, and then its rheometric properties were determined on a rheometer MDR 3000 Basic. The vulcanized rubber was obtained by vulcanizing the rubber compound in a P-V-100-3RT-2-PCD vulcanization press. The resulting vulcanizates were tested for their physical and mechanical properties (elastic strength properties, Shore A hardness, rebound elasticity, and relative compressive strain), as well as their performance properties (changes in tensile strength, elongation at break, and hardness after thermal-oxidative aging in air, changes in mass after exposure to liquid aggressive environments, and frost resistance).

These results allow us to formulate the following conclusions:

1. The rubber compound containing N 220 carbon black exhibits a large difference between the maximum and minimum torques, which is directly proportional to the degree of chemical crosslinking and the strength properties of the vulcanizate.
2. The vulcanizate containing carbon black N 220 has better and more stable physico-mechanical properties compared to vulcanizates containing carbon blacks P 324, P 803 and silica ZC-120, which explained by the smaller particle size and higher specific surface area of carbon black N 220 compared to carbon black P 324 and P 803, as well as poor dispersion of silica ZC-120 in the rubber matrix. For the vulcanizate containing carbon black N 220, an insignificant change in the conventional tensile strength is observed, as well as the relative elongation at break after thermal aging in air, exposure to aggressive acid-base environments, and high frost resistance.

3. Recommendations for the practical application of the results: a rubber mixture consisting of ethylene propylene diene monomer rubber SKEPT-40 and containing 60.0 phr carbon black N 220, can be recommended for rubberizing the surfaces of tanks used for transporting and storing acid-base solutions.
4. A possible direction for further research is to study the effect of carbon black N 220 on rubber based on other ethylene propylene diene monomer rubbers, such as S 501A and S 505A.

## CRediT authorship contribution statement

**Evgeniy N. Egorov**  : writing – review & editing, writing – original draft, conceptualization, investigation; **Nikolay I. Kol'tsov**  : writing – review & editing, writing – original draft, conceptualization, supervision, data curation.

## Conflict of interest

The authors declare that they have no conflict of interest.

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