

## THE EFFECT OF COMPOSITION AND STRUCTURE OF NBR-BASED ELASTOMER BLENDS IN THE VULCANIZATION PROCESS AND STUDY OF THEIR AGING BY EXPOSURE TO HEAT AND RADIATION

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**Abstract.** The vulcanization process of elastomeric blends based on chlorinated aromatic nitrile-butadiene rubber (SKN-40M) has been studied in the present work. The structural parameters (molecular weight, plasticity, rigidity, number of network chains, cross-linked molecules) in thermovulcanizates were investigated on an example of nitrile-butadiene rubber blend with polyvinyl chloride using viscometry and the sol-gel process.

**Keywords:** rheology, vulcanization, hexachloroparaxy-lene, nitrile butadiene rubber, cross-linking.

### 1. Introduction

Due to the expansion of the application areas for elastomeric materials (EM), one of the requirements for them is resistance to aggressive environments (oils, heat, and radiation). Therefore, data on the regularities of EM aging in aggressive environments are of direct practical importance.

It is known from the literature<sup>1-4</sup> that to increase the resistance of EM made of nitrile butadiene rubbers to aggressive environments, rubbers are subjected to crosslinking with low molecular weight compounds. However, as shown by Onishchenko,<sup>5</sup> the resulting EM with semi-sulfide cross-links undergo significant changes in the physical and mechanical characteristics of the seal (hardness, strength, permanent deformation, dynamic endurance) under prolonged exposure to the sealed liquid and temperature. The low aggressive resistance of EM is

explained by their structural features, in particular, chemical bonds between high and low molecular weight compounds, as well as the difference in binding energies.<sup>6-10</sup>

The works<sup>11-19</sup> present data on the influence of various factors on the mechanical-chemical processes, which occur in elastomers based on saturated and unsaturated elastomers. The authors<sup>3,16,20,21</sup> studied the behavior of some stressed NBR-based rubbers under the  $\gamma$  radiation. However, no clear indications exist in the literature about heat and radiation-resistant elastomers based on SKN-40M rubber. The modern oil industry and nuclear engineering require the creation of reliable sealing devices for fixed and movable links.

In this regard, information on the effect of various factors on the aging of elastic polymers is of great importance. Such information is valuable for both practical purposes and scientific provision. Despite the large number of studies on the destruction of elastomers under typical operational conditions, they are still relevant today. With the development of the oil industry, engineering industry, and nuclear technology, the implementation of new equipment and units requires the creation of new synthetic elastic materials operated under difficult dynamic and climatic conditions. It is known that agidol-2 (2,2-methylene-bis-4-methyl-6-tert-butylphenol) is a heat stabilizer and effectively protects EM based on unsaturated diene rubbers from oxidative aging, provides high stability of materials when used in liquid aggressive environments.<sup>22</sup> It was interesting to use these products as antioxidants, which have a high reactivity and react only at their decomposition temperatures (over 373 K).

The aim of this study is the formation of the chemical structure in NBR (SKN-40M), and the selection of new low-molecular crosslinking agents, modifiers, and sensitizers for NBR vulcanization, to obtain heat- and radiation-resistant elastomer materials.

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## 2. Experimental

### 2.1. Materials

SKN-40M nitrile butadiene rubber (NBR) was used as the object of research. The NBR average molecular weight (Mw) was 222,000. Plasticization of the elastomer (SKN-40M) was carried out at 313 K for 8 min on a laboratory mill.<sup>13</sup>

Previous studies reported the effect of hexachloro-p-xylene (HCPX) as a stabilizer 2,4-dichloro-diethylamino-

sim-triazine (DCDEAST) and a sensitizer 2,4-dimethyl-phenylmaleimide (DMPM) to evaluate the effectiveness of chlorinated aromatic compounds as crosslinking agents.<sup>14,15</sup> When choosing carbon black (P324, P514), the following indicators were taken into account: structure, elemental composition, particle size, oil number, and specific surface area. Petroleum products – fuel oil and oil PN-6, which has a high boiling point (480 K) and low viscosity were used for plasticization of blends. Besides these compounds, ED-5 epoxy-diane resin was used as a modifier. The composition of the studied blends is given in Table 1.

**Table 1.** The composition of elastomeric blends based on SKN-40M

Components	Content (parts per 100 parts of rubber by weight)
SKN-40M	100
HCPX	4.0
DCDEAST	2.0
DMPM	3.0
Zinc oxide	5.0
Epoxy resin (ED-5)	6.0
Agidol-2	3.0
Fuel oil	4.0
PN-6	1.5
P324	40
P514	20

### 2.2. Methods

Elastomeric blends were prepared according to the recipe for SKN-40M with a roll fraction of 1:2. The samples were vulcanized at a temperature of 423 K in an electric press for 60 min. Irradiation of heat vulcanizates was carried out using a Co<sup>60</sup> source. The radiation source power was 1.42 Gy/s during the study period. The particle size in the finished blend was 1-3 μm.

The rheological properties of elastomeric blends were determined by the known method.<sup>8</sup> Structural parameters of filled and unfilled vulcanizates were determined by sol-gel processes.<sup>10</sup> The change in the compound of polar groups ( $-C\equiv N$ ,  $>C=O$ ,  $C-Cl$ ) during the aging of unfilled samples was determined using IR spectroscopy by changing relative optical density (ROD).

The deformation and relaxation properties of the elastomer were evaluated after aging at 373–473 K in air and in benzene.<sup>1,13</sup>

Similar experiments were carried out for heat vulcanizates during their aging under a radiation-absorbed dose (1000-2000 kGy). The work consisted of three stages: 1) development of crosslinking systems for SKN-40M rubber; 2) study of the structural parameters of the obtained vulcanizates; 3) study of the influence of temperature and radiation on the aging of elastomers in air and in benzene.

## 3. Results and Discussion

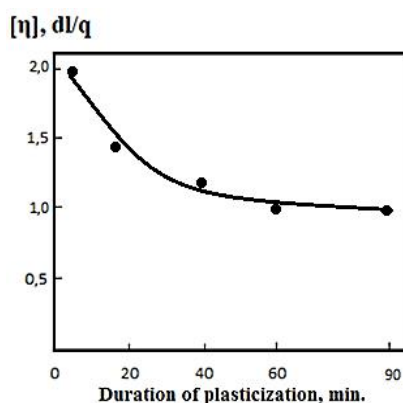
As a rule, crosslinking of NBRs with low molecular weight compounds involves the interaction of components of different structures. In principle, any technological characteristic of a mixture of nitrile-butadiene rubber and polyvinyl chloride will obviously be a function of the relative content of structural features of each component. Therefore, the determination of the viscoelastic behavior of SKN-40M rubber is the most important test that crosslinking polymer systems should be subjected to.

Viscoelastic behavior during crosslinking with low molecular weight compounds is related to the structure and molecular weight. The results show that mechanical plasticization of blends (without carbon black) is characterized by a change in plasticity, rigidity, and Mooney viscosity with an increase in plasticization time (Table 2). It can be seen that slight changes in plasticity are observed after 20 min of model blend mechanical plasticization. An increase in the plasticization time to 30 min leads to minor changes in the plasticity parameters, and an increase in the plasticization time to 90 min reduces it sharply. Apparently, this phenomenon is associated with the onset of degradation in the samples. The subsequent increase in time of mechanical plasticization of elastomer blends does not lead to significant changes in the stiffness coefficient and remains within 1000-1200 gs.

**Table 2.** Dependence of the plastic-elastic properties of elastomeric blends (SKN-40M) on plasticization time in mills (343 K x 90')

Plasticization time, min.	Mooney viscosity, c.u.	Rigidity, gs	Plasticity, stand. units
10	80	2500	0.60
30	70	2000	0.40
60	60	1500	0.30
90	40	1000	0.25

The decrease in rigidity is obviously associated with mechanical plasticization and the influence of low molecular weight compounds under the action of applied forces, in which free radicals appear. The kinetics of the change in the inherent viscosity of the elastomer blends is described by monotonically decreasing curves (Fig. 1).



**Fig. 1.** Dependence of the inherent viscosity [η] of solutions on the plasticization time of filled blends based on SKN-40M elastomer

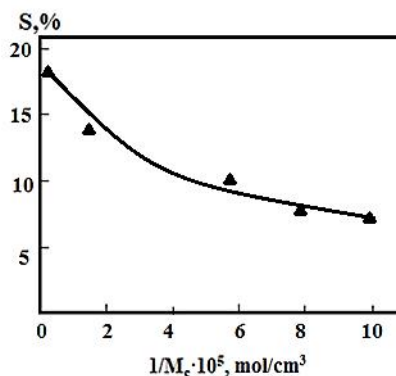
At the same time, a high rate of change in the properties of the mixtures during mechanical plasticization within 90 min is observed. According to the data on the intrinsic viscosity of the polymer mixture during mechanical plasticization, the molecular weight was determined to be in the range of 220,000-135,000.

When using carbon black, the crosslinking process of elastomeric mixtures after heating in an electric press is characterized by completely different patterns (P324+P514). The data obtained (Table 3) indicate that the plasticity of thermosetting vulcanizates decreases after 40 min from the start of the reaction. The destructive chan-

ges in the thermo-vulcanizates are completed and then the plasticity increases monotonically until it reaches a constant value within 60 min (1100 gs).

The most critical parameter affecting the network structure of vulcanizates is the yield change in sol-gel characteristics.

The result of the sol-gel process of the studied vulcanizates is shown in Fig. 2, which shows that the amount of sol fraction depends on the number of network chains (1/M<sub>s</sub>). The change in the amount of sol at a constant 1/M<sub>s</sub> is observed with a change in the molecular weight distribution and is obviously related to the peculiarities of the adsorption of the elastomer of the dispersed medium (MMR).



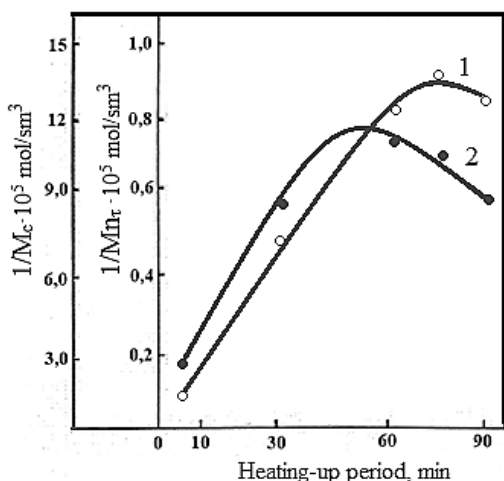
**Fig.2.** Dependence of the amount of the sol fraction (S) on the concentration of the number of network chains of the (1/M<sub>c</sub>) of filled vulcanizates based on SKN-40M

The results of determining the structural parameters of the crosslinked network formed under the action of a crosslinking agent by the sol-gel process allow us to more fully characterize the properties of blends based on SKN-40M rubber obtained with a carbon black.

**Table 3.** Dependence of the plastic-elastic properties of elastomer blends (SKN-40M) on heating time (423K x 40')

Heating-up period, minutes	Mooney viscosity, c.u.	Rigidity, gs	Plasticity, stand. units
10	90	1800	0.40
20	80	1500	0.35
30	65	1200	0.27
40	55	800	0.20

The data represented in Fig. 3 show the change in the number of chains in the crosslinked molecules vs. the crosslinking time. The results indicate that carbon black crosslinking occurs more efficiently.



**Fig. 3.** Dependence of the structural parameters of the network of vulcanizates based on SKN-40M on the heating-up period. 1-number of crosslinked molecules ( $1/M_{n_r}$ ); 2-number of network chains ( $1/M_c$ )

The experimental data, obtained by us earlier,<sup>23</sup> and present observations allow us to present the effect of low molecular weight compounds on the process of structurization of the SKN-40M elastomer as follows. The rate of crosslinking in the main period can be considered as a consequence of the HCPX formation during thermal dissociation, which is capable of interacting with the elastomer.

The density of the vulcanization networks obtained with HCPX vulcanizates is a consequence of the fact that the HCPX used has a crosslinking effect, which leads to the formation of C-C bonds between the elastomer molecules.

It should be mentioned that when the number of crosslinked molecules in SKN-40M rubber becomes significant, the strength of the vulcanizate decreases. Indeed, as can be seen from Table 4, a decrease in rupture resistance occurs when the C-C chemical bond becomes unchanged, while its relative elongation decreases, as a re-

sult, a monotonous increase in the concentration of active chains ( $1/M_c$ ) is observed.

So, the obtained data allow us to consider that model systems based on SKN-40M rubber dissociate completely in the volume of the elastomer during mechanical plasticization and low-molecular-weight HCPX becomes an effective structuring agent for SKN-40M rubber during heating-up.

Studies have shown that the crosslinking rate of SKN-40M elastomer increases with the increase in temperature during aging in air (Table 5). The most intense crosslinking is observed at temperatures above 398 K. The crosslinking rate increases with increasing temperature during aging in benzene at 398 K, as well as in air, and changes insignificantly at temperatures above 398 K. At the same time, the aging of the elastomer in fuel occurs to a lesser extent than in air. Compared to aging in air, the lower degree of elastomer crosslinking in benzene (3:1) is obviously associated with a decrease in the rates of elastomer oxidation and thermal crosslinking involving nitrile groups, as well as the effect of swelling on the degradation and crosslinking processes. On the other hand, a decrease in the content of  $-C\equiv N$ ,  $>C=O$ ,  $C-Cl$  groups is observed to a greater extent than at 398 K during aging at 473 K.

As can be seen from Table 5, during aging in benzene in a stressed state, a different pattern of changes in the properties of the elastomer is observed, in particular, the accumulation of residual deformation with increasing temperature continuously increases, as in air. The obtained patterns confirm the above conclusions about the increase in the contribution of destruction during aging in benzene and in air. The increase in the contribution of destructive processes in the presence of static stress is one of the signs of mechanical activation of the chemical process.

Stress measurements of deformed elastomers exposed to radiation are one of the most common methods used in the study of radiation aging. It simultaneously solves two problems: quantification of structural changes in vulcanizates during thermal irradiation and assessment of radiation resistance by changes in contact stress, which is responsible for the sealing ability of the material.

**Table 4.** Dependence of vulcanizates properties on the HCPX content in the system

HCPX content (parts per 100 parts of rubber by weight)	Number of network chains, $1/M_c$ , mol/cm <sup>3</sup>	Tensile strength, $\sigma$ , MPa	Relativity elongation, $\epsilon$ , %
0.5	1.7	11.0	720
1.0	2.4	13.0	640
1.5	3.6	15.0	600
2.0	5.1	16.0	580
2.5	7.6	18.0	540
3.0	9.1	20.0	510

**Table 5.** The effect of temperature on the aging of the elastomer based on SKN-40M in air and in benzene

Aging. T, K	Degree of cross-linking $\gamma$ (24 h)	Change in the content of polar groups in elastomers during their aging in air				Deformation set under compression (E. resid. 20 %. 24h)		Equilibrium modulus $E_{\infty}$ % from baseline		Stress relaxation $\sigma_1/\sigma_0$ MPa (24h)	
	In air	In gaso-line benzene (3:1)	-C $\equiv$ N	C-Cl	$\square$ C=O	In air	In gaso-line benzene (3:1)	In air	In gaso-line benzene (3:1)	In air	In gaso-line benzene (3:1)
373	9.7	3.6	0.44	0.31	0.57	60	40	20	40	0.9	0.8
398	12.8	5.9	0.38	0.27	0.50	70	50	40	60	0.8	0.7
423	16.0	8.3	0.30	0.22	0.41	80	65	60	80	0.7	0.6
473	21.0	12.4	0.22	0.17	0.36	90	75	80	100	0.7	0.5

**Table 6.** Effect of radiation on the aging of SKN-40M elastomer in air and in benzene

Absorbed dose, kGy	Cross-link yield, $N_c \cdot 10^{-19}$ , bond/cm <sup>3</sup>	Change in the content of polar groups in elastomers during their aging in fuel				Deformation set under compression (E. resid. 20 %. 24h)		Equilibrium modulus, $E_{\infty}$ % from baseline		Stress relaxation $\sigma_1/\sigma_0$ MPa (24h)	
	In air	In gaso-line benzene (3:1)	-C $\equiv$ N	C-Cl	$\square$ C=O	In air	In gaso-line benzene (3:1)	In air	In gaso-line benzene (3:1)	In air	In gaso-line benzene (3:1)
1000	14.5	2.2	0.31	0.39	0.66	30	20	100	80	0.7	0.8
1250	16.8	3.6	0.27	0.34	0.60	60	30	120	90	0.6	0.5
1500	20.1	4.7	0.21	0.30	0.56	80	60	118	100	0.5	0.6
2000	22.4	5.5	0.19	0.28	0.49	100	85	118	90	0.4	0.6

The study of changes in the concentration of crosslinks in unfilled heat vulcanizates under the influence of radiation after aging showed the presence of co-vulcanization, i.e., the formation of a three-dimensional structure due to the interaction of polymer radicals formed during elastomer irradiation. Table 6 shows that, when heat vulcanizates are irradiated with a dose of 2000 kGy, the yield of crosslinks ( $N_c$ ) in the elastomer is 22.4.109 bonds per 100 eV of absorbed energy. The radiation-chemical processes are intensified at dose rates above 2000 kGy.

The study of the change in the composition of polar groups in irradiated heat vulcanizates in benzene showed that oxidation processes are practically not manifested up to a dose of 1500 kGy. To perform a quantitative comparison of the change in unsaturation, heat vulcanizates were irradiated with a dose of up to 2000 kGy in order to obtain a high-density network. The use of such high doses for comparison is legitimate, since the rate of change in unsaturation remains constant at a dose of 2000 kGy. The observed absorption bands at specific wavelengths (970cm<sup>-1</sup> cm indicating alterations in 1,4 cis double bond configurations, 2240 cm<sup>-1</sup> suggesting changes in -C $\equiv$ N groups, cm<sup>-1</sup> indicating the presence of C-Cl groups, and

1720 cm<sup>-1</sup> signifying modifications in >C=O groups) in the context of vulcanizates imply structural and chemical alterations resulting from irradiation.<sup>10</sup> The change in these bands after exposure to benzene can be caused by cis-trans isomerization after irradiation, as well as by the consumption of double bonds. Increasing the irradiation dose over 2000 kGy leads to a 9 % decrease in cis-unsaturation.

Thus, the observed changes in the properties of irradiated heat vulcanizates during aging in fuel at a dose of more than 1500 kGy are explained by oxidation processes, and at a dose of more than 2000 kGy, destructive processes are observed in the polymer, which contributes to a significant drop in unsaturation.

It is known that NBR-based elastomers are widely used in nuclear engineering for the manufacture of elastomeric sealing materials. Therefore, the study of the regularities of radiation aging of these elastomers in a wide range of absorbed doses depending on the chemical nature of the elastomers is of considerable interest. The aging of elastomers in a wide range of absorbed doses can be characterized by changes in deformation during compression (E, %), the equilibrium modulus (E, %), and the stress relaxation rate under static tests.

The radiation resistance of elastomeric materials in a compressed state is assessed by the absorbed dose, at which it is 80 %, and  $\sigma_1/\sigma_0 = 0.2$ .<sup>23</sup>

The data obtained (Table 6) indicate that the crosslinking of residual deformations prevails in E, and relaxation is insignificant in cases where there is a sharp increase in the equilibrium modulus in air and in fuel. On the contrary, the accumulation of residual deformations changes insignificantly with a small increase in the equilibrium modulus but the stresses relax at a high rate. Unlike the thermal aging of an elastomer, there is no correlation between the rate of residual deformation accumulation and the rate of stress relaxation during radiation aging. The residual deformation of the elastomer at doses of 1000-2000 kGy monotonically increases and amounts to 40-45 %, *i.e.*, the elastomer in this case should practically not work as a sealant. At doses of 2000 kGy, the stress drops by no more than 20-30 %.

So, the evaluation of the radiation resistance of elastomeric materials can be carried out both by the change in stress relaxation and by the accumulation of residual deformation. When choosing elastomeric materials for operation under dynamic conditions and other types of aging, radiation resistance should be characterized by changes in dynamic characteristics.

## 4. Conclusions

The abovementioned experimental data allow us to present the behavior of high- and low-molecular-weight compounds as crosslinking agents and explain their distinctive features. The rate of NBR crosslinking with HCPX in the main period can be considered as a consequence of the formation of low molecular weight products capable of interacting with NBR during thermal dissociation. The density of the vulcanization networks obtained under the influence of HCPX, DCDEAST, DMPM, and ZnO is a consequence of the fact that the used components have a crosslinking effect, which leads to the formation of C-C crosslinks between the elastomer molecules. Crosslinked NBR with a stabilizer (DCDEAST) has a series of positive qualities: it retains its average molecular weight, which has an excellent effect on the elasticity of composites, and the stiffness of vulcanizers is reduced by 2 times. The use of maleimide compounds as a sensitizer reduces the absorbed dose and radiation resistance. The assessment of thermal and radiation resistance of elastomeric materials can be characterized by changes in stress relaxation and the accumulation of residual deformation.

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## ВПЛИВ СКЛАДУ І СТРУКТУРИ СУМІШЕЙ ЕЛАСТОМЕРІВ НА ОСНОВІ БНК В ПРОЦЕСІ ВУЛКАНІЗАЦІЇ ТА ДОСЛІДЖЕННЯ ЇХНЬОГО СТАРІННЯ ПІД ДІЄЮ ТЕПЛА І РАДІАЦІЇ

**Анотація.** Досліджено процес вулканізації еластомерних сумішей на основі хлорованого ароматичного нітрил-бутадієнового каучуку (СКН-40М). На прикладі суміші бутадієн-нітрильного каучуку з полівінілхлоридом досліджено структурні параметри (молекулярна маса, пластичність, жорсткість, кількість ланцюгів сітки, зшиті молекули) в термовулканізатах за допомогою віскозиметрії та золь-гель процесу.

**Ключові слова:** реологія, вулканізація, гексахлоропара-ксилен, нітрилбутадієновий каучук, зшивання.