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EFFECT OF γ-IRRADIATION ON THE STRUCTURE AND STRUCTURAL PARAMETERS OF THE NETWORK OF A MIXTURE OF BUTADIENE NITRILE RUBBER AND POLYVINYL CHLORIDE

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The effect of γ-irradiation on the structure and structural parameters of the network of mixture of butadiene nitrile rubber and polyvinyl chloride (SKN-40-PVC-30) in the absorbed dose range of Φ_γ = 100...800 kGy in air and in vacuum was investigated by FTIR spectroscopy method and sol-gel analysis. The results indicate that irradiation of the SKN-40-PVC-30 mixture in air promotes a higher crosslinking rate compared to irradiation in vacuum within the irradiation dose range of Φ_γ = 100…500 kGy, however at $\Phi_\gamma \geq$ 800 kGy carried out scission prosess. The investigations confirm that SKN-40-PVC-30 mixture could be used effectivly at Φ_γ = 100...500 kGy.

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INTRODUCTION

Most elastomers are crosslinked polymers, however their tendency to form crosslinks in elastomers of different natures is not the same and is determined by both the chemical structure of the polymer chain and the morphology of the polymer and the specific irradiation conditions. Furthermore, the efficiency of the radiation-induced crosslinking process depends to a large extent on the nature of the monomer ratio in the copolymers [1–5].

Limited attention has been devoted to studying the effect of irradiation on the nature of the structural parameters of the spatial network in irradiated semi-blends (blends of nitrile butadiene rubber with polyvinyl chloride). There is little information in the literature for other grades of nitrile butadiene rubbers [6–8]. However, the process of radiation exposure to a mixture of nitrile butadiene rubber (BNR) with polyvinyl chloride (SKN-40-PVC-30) has been insufficiently explored. There are only some data on the effect of irradiation on the crosslinking rate for isoprene, polybutadiene and natural rubbers [9–14].

However, comprehensive investigations into the effect of radiation on the number of network chains ($1/M_c$), the concentration of crosslinked molecules ($1/Mn_\tau$), rheological properties and the distribution of double bonds in SKN-40-PVC-30 have not been carried out.

Depending on the irradiation conditions, polymer structure, and the composition of polymer groups, not only crosslinking reactions but also destructive processes and chemical modifications of the polymer-elastomer chains can occur during irradiation. As a result of the reactions occurring, along with crosslinks in the irradiated sample of the group not included in the composition of the nodes, there are segments of chains with a modified microstructure. At any level of crosslinking, terminal segments of elastomer molecules remain [13–21], that do not bear stresses and, therefore, do not contribute to the effective spatial network. The proportion of end segments increases due to degradation processes after irradiation. In cases of extensive crosslinking, fragments of molecular chain capable of diffusing into solvent are also formed, along with unreacted molecules they make up the soluble sol fraction in irradiated elastomer mixtures.

Systematic studies on the effects of γ-irradiation on the radiation-chemical yield, the average molecular weight of a chain segment (the number of network chains – $1/M_c$) and the number of crosslinked molecules ($1/Mn_\tau$), rheological behavior and the double bonds content in the SKN-40-PVC-30 mixtures have not been carried out.

This study presents the results of a study of the effect of γ-irradiation on the structure and the structural parameters of the network of mixture of nitrile butadiene rubber with polyvinyl chloride (SKN-40-PVC-30) using sol-gel analysis and FTIR spectroscopy method.

1. EXPERIMENTAL PART

1.1. MATERIALS USED

The object of this study was a mixture of butadiene-nitrile rubber (BNR) and polyvinyl chloride (SKN-40-PVC-30) in a ratio of 70:30. The mixtures based on SKN-40 with polyvinyl chloride were prepared using a laboratory two-roll mill with a diameter of 200 mm and a length of 450 mm. The rollers were maintained at a temperature of 293...300 K, the front roller rotated at a speed of 20 rpm. Initially, the BNR was plasticized on the rollers for 2 min. Subsequently, 30 wt.% of PVC was added to the softened BNR composition. The mixture at a ratio of 70:30 was stirred for 7 min. Based on the Fourier-transform infrared spectroscopy study in the butadiene part of the polymer, at 300 K, the isomeric composition of double bonds was determined, which in the studied copolymer were: 1, 2 isomers – 14.3%; 1, 4 isomers – 14.8%, and trans 1, 4 isomers – 70, 9%.

Macromolecules of SKN-40-PVC-30 rubber consist of statically distributed units of butadiene, acrylonitrile and polyvinyl chloride. Features of the action of radiation effect on microstructure of the BNR mixture, molecular weight, molecular weight distribution (MWD) and gel content. Therefore, the average number values (M_c), molecular weights (M_w) and polydispersity (M_n = 69 thousand, M_w = 226 thousand, M_W/M_n = 3.28) were determined using gel permeation chromatography (GPC).

1.2. RESEARCH METHODS

The prepared 1 g samples were sealed in *Luch* glass ampoules and evacuated for 3 h to a residual pressure of $1.3 \cdot 10^{-1}$ Pa. Radiolysis of the sealed ampoules containing the samples was carried out with γ-rays of ^{60}Co at a dose rate of 4.9 Gy/s at room temperature. The absorbed dose in the studied samples was determined by comparing the electron densities of the experimental and dosimetric systems [1, 3]. The molecular weights (intrinsic viscosity) of the irradiated elastomer mixtures (SKN-40-PVC-30) were measured in toluene at 293 K using a standard technique involving an Ubbelohde viscometer. Calculations were performed using the Mark Houwink equation $[\eta] = KM^{\alpha}$, with the constant value: $K = 4.9 \cdot 10^4$ and $\alpha = 0.64$ (for toluene), according to [20, 21].

For the quantitative spatial network of crosslinked binary mixtures of butadiene-nitrile rubber (BNR) with polyvinyl chloride (PVC), the following parameters M_c and Mn_τ are used [18]:

$1/M_c$ – respectively, the number of network chains, mol/cm^3;

$1/Mn_\tau$ – number of crosslinked molecules in the sample, mol/cm^3.

These parameters of the spatial network and molecular characteristics of crosslinked elastomer mixtures are determined using sol-gel analysis data, which are based on sequential extraction of samples first with acetone and then with toluene. Extraction with acetone is performed to extract non-rubber impurities from the mixtures, while during extraction with toluene, the soluble fraction of elastomer mixtures is extracted.

Extraction of elastomer mixtures (SKN-40-PVC-300) irradiated with γ-rays is carried out in Soxhlet apparatuses consisting of an exiccator with a drain tube of a reflux condenser and a flask into which the extracting liquid is poured.

Calculation of the main parameters of the spatial network of irradiated samples is carried out according to the formulas:

1. Content of the sol fraction:

$$S = \frac{P_a - P_T}{P_Э}, \qquad (1)$$

where $P_Э$ is the mass of the sample extracted with acetone, g; P_T is the mass of the sample extracted with toluene.

2. Equilibrium degree of swelling (M_H):

$$M_H = \frac{P_H}{P_T} \cdot 100, \qquad (2)$$

where P_H is the mass of the swollen sample, g; P_T is the mass of the sample after extraction with toluene.

3. The number of active chains of the network ($1/M_C$), mol/cm^3:

$$1/M_C = \frac{\ln(1-V_Г) + \mu \cdot V_Г^2}{\rho_K V_0 \left(V_R^{1/2} - \frac{2V_Г}{f} \right)}, \qquad (3)$$

where V_0 is the molar volume of the solvent; μ is the constant of interaction of the polymer with the solvent; f is the functionality of the crosslinks.

4. The number of crosslinked molecules ($1/M_n\tau$), mol/cm^3:

$$1/M_n\tau = \frac{S + \sqrt{S}}{M_C}. \qquad (4)$$

Fourier-transform infrared (FTIR) transmission spectra of the SKN-40-PVC-30 (70:30) blend were recorded before and after γ-irradiation using a Varian 640 FTIR spectrometer at room temperature in the wavenumber range of 2400...400 cm^{-1}. The positions of the absorption band maxima (ν), the half width ($\nu_{1/2}$), and the optical densities (D) of the characteristic functional group bands were determined and calculated using the appropriate formula.

$$D = \lg I/I_0, \qquad (5)$$

where D, I, and I_0 are the optical density, the transmission and the incident intensities, respectively [22].

2. RESULTS AND DISCUSSION

2.1. EFFECT OF γ-IRRADIATION ON THE STRUCTURE OF SKN-40-PVC-30

In order to determine the effect of γ-radiation on the structure of SKN-40-PVC-30 samples, FTIR transmission spectra were recorded before and after γ-irradiation at doses of Φ_γ = 100…800 kGy in air at room temperature (T = 300 K). A comparative analysis of the transmission spectra revealed that at irradiation doses of Φγ = 100…500 kGy, no significant structural changes were observed. the change in the spectra is insignificant, therefore the spectral measurements were carried out after irradiation up to 800 kGy in air.

Therefore, we obtained, studied in detail and analyzed the FTIR transmission spectra of the initial (Fig. 1, curve 1) and gamma-irradiated at a dose of Φ_γ = 800 kGy (see Fig. 1, curve 2) SKN-40-PVC-30 samples in air.

As can be seen from the spectrum of the initial sample, its molecular structure is characterized by the presence of a number of transmission bands related to different functional groups. The main observed transmission bands and their assignments of initial and gamma irradiated SKN-40-PVC-30 are listed in Table.

These functional groups include CH groups associated with nitrile and chlorine moieties, CH_2 groups, carbonyl (C=O) groups, and C=C double bonds of the polymer chains (see Fig. 1, curve 1).

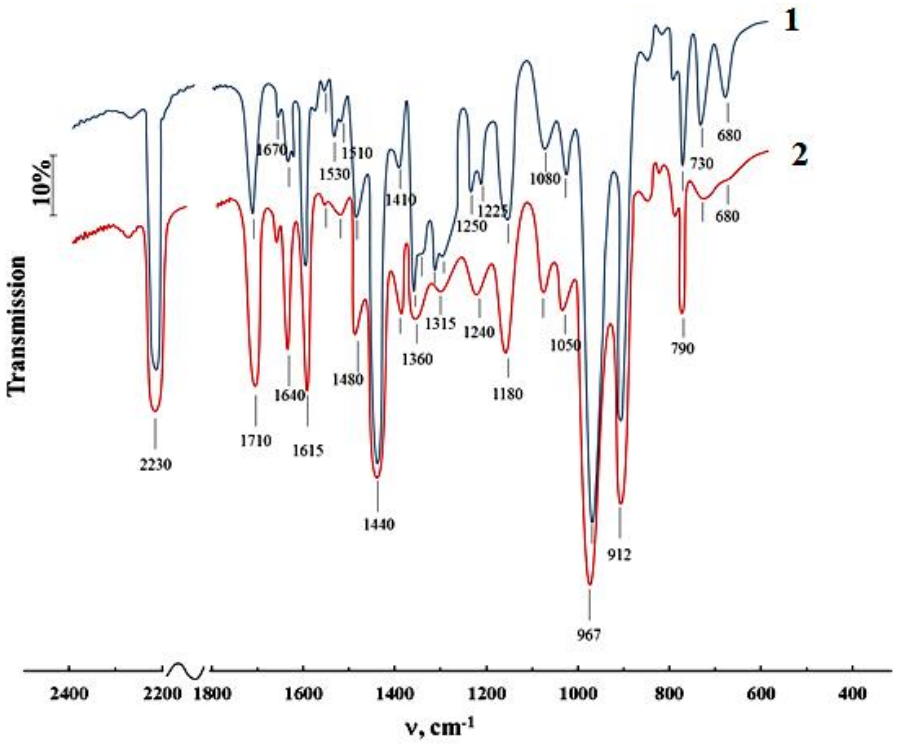

Fig. 1. FTIR-spectra of initial (1) and gamma irradiated at Φ_γ = 800 kGy (2) of SKN-40-PVC-30

Main vibrations of initial and gamma irradiated SKN-40-PVC-30

Wave number ν, cm^{-1}	İnitial	Dose,kGy		Functional group, types of vibration
	0	500	800	
2230	2230	2230	2230	ν_s (-C≡N) stretching nitrile
1710	1710	1710	1710	ν_s (-C=O) stretching
1670-1615	1670, 1640, 1625	1640, 1625	1640	ν_s (-C=C-) stretching 1, 2 trans and 1, 2 cis
1480-1410	1480, 1440, 1410	1480, 1440, 1410	1480, 1440, 1410	ν_δ (C-H) deformation in the CH_2 group
1360-1310	1360, 1340, 1325, 1310 dublet	1360, 1340, 1325,1310 dublet	1360, 1315 wide	ν_δ (C-H) deformation trans (-CH=CH-)
1250-1225	1250-1225	1250-1225	1240 wide	ν_δ (C-H) deformation cis (-CH=CH-)
967	–	–	967	ν_δ (out of plane) 1, 4 trans C=C
912	–	–	912	ν_δ (C=C) group 1, 4 cis C=C
790	–	–	790	ν_δ (C=C) out of plane
730	–	–	730 wide, very weak	ν_s (C-Cl) stretching
680	–	680	680, very weak	ν_δ (C-H) out of plane

The primary analytical bands for identifying SKN-40-PVC-30 are those with maxima at 2230 and 730 cm^{-1}, corresponding to the stretching vibrations of the nitrile (–C≡N) and C–Cl groups, respectively. The transmission bands in the regions around 1710 and 1690…1500 cm^{-1} are attributed to the stretching vibrations of carbonyl (C=O) and vinyl (C=C) groups. Vinyl groups are also observed in the deformation vibration region, indicated by bands at 967 and 912 cm^{-1}, corresponding to 1, 4- and 1, 2-trans configurations. The presence of the 1,4-trans configuration is further confirmed by the transmission band for C=C stretching at 1640 cm^{-1} (see curve 1). The transmission bands in the 1480…1410 and 1360…1310 cm^{-1} regions are attributed to C–H deformation vibrations in –CH fragments of CH_2 and CH_2 groups [23–26].

After γ-irradiation of SKN-40-PVC-30 samples at Φγ = 800 kGy, the following significant changes are observed in the FTIR transmission spectra (see Fig. 1, curve 2):

1. The optical densities of the –C≡N and C–Cl stretching bands decrease by factors of approximately

1.5 and 2.6, respectively, while the half width of the C–Cl band increases by ~3.5 times, reaching $\nu_{1/2} \approx 85$ cm^{-1}.

2. γ-irradiation leads to an increase in the optical densities of the C=O and C=C bands, attributed to the formation of various oxidative degradation products (also evidenced by a ~1.4-fold increase in the half width of the C=O band) and a rise in the concentration of trans-1, 4-vinyl C=C groups.

3. γ-irradiation induces noticeable conformational changes in the SKN-40-PVC-30 structure. In the spectral regions 1600…1500, 1400…1280, and 1280…1200 cm^{-1}, a redistribution of the optical densities of doublet bands corresponding to different (trans and cis isomeric) configurations is observed, accompanied by shifts in band maxima and broadening. This leads to the disappearance of the doublet structure associated with specific configurations, indicating the occurrence of chain scission processes. The observed broadening of the trans and cis isomeric bands is likely attributable to increased amorphization within the SKN-40-PVC-30 structure.

Thus, a comparison of changes in crosslink yield, transverse bonding, double bond distribution, and FTIR spectral features indicates that radiolysis of SKN-40-PVC-30 results in a general increase in unsaturation in the rubber matrix and enhanced radiation sensitivity, manifested in an increased effective yield of crosslinks and transverse bonds in the dose range $\Phi\gamma$ = 100…500 kGy. At doses of $\Phi\gamma$ = 800 kGy, chain scission processes become predominant, leading to significant structural modifications, including radiation-oxidation, conformational redistribution, and structural amorphization.

2.2. EFFECT OF γ-IRRADIATION ON THE STRUCTURAL PARAMETERS OF THE NETWORK OF SKN-40-PVC-30

Figs. 2 and 3 present dose dependences of the characteristic viscosity of SKN-40-PVC-30 in air and in vacuum (curves 1, 2) and the sol fraction content in the SKN-40-PVC-30, respectively. As shown in the Fig. 2, the characteristic viscosity of SKN-40-PVC-30 rubber is 0.4 at a dose of 100 kGy. With an increase in the absorbed dose, the molecular weight of the rubber increases.

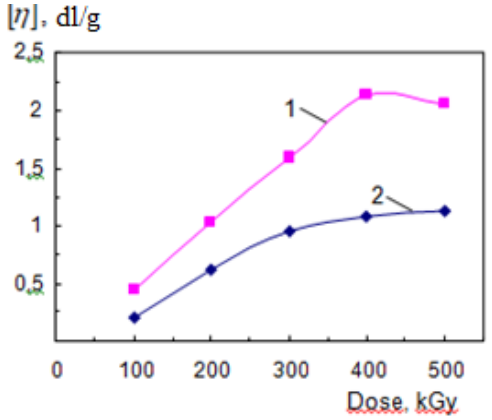

Fig. 2. Dose dependences of the characteristic viscosity of SKN-40-PVC-30: 1 – in air; 2 – in vacuum

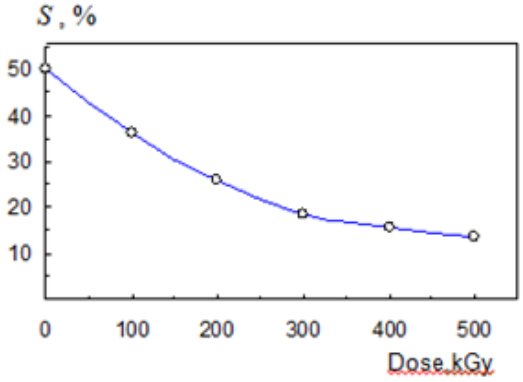

Fig. 3. Dose dependence of the sol fraction content in the SKN-40-PVC-30

When irradiating SKN-40-PVC-30 rubber in air in the dose range of 100...500 kGy, an increase in the molecular weight is observed. In this case, the characteristic viscosity increases from 0.4 to 2.3. However, beyond an absorbed dose of 400 kGy, a decline in characteristic viscosity is observed (see Fig. 2, curve 1). A decrease in the molecular weight of the rubber is a consequence of degradation of the main chain of the polymer. The increase in viscosity in the range of absorbed dose values of 100...500 kGy may be associated with the formation of a spatial structure due to the reaction of intramolecular crosslinking. In contrast, when the samples are irradiated under vacuum, an opposite trend is observed. The decrease in molecular weight at increased doses (500 kGy) is associated with low gel formation, which leads to the breakdown of polymer chains (see Fig. 2, curve 2).

At irradiation doses of 100 kGy, the amount of soluble sol fraction reaches 15% (Fig. 3). However, at doses of 400...500 kGy, the polymer becomes completely insoluble, indicating crosslinking of polymer chain molecules. Consequently, an increase in the molecular weight of SKN-40-PVC-30 rubber, due to irradiation in air, occurs with the participation of vinyl bonds located at the 1, 2 positions [12]. It is well known that one of the key factors influencing the efficiency of the crosslinking processes is the irradiation condition (the presence of oxygen). As shown in Fig. 4, the effect of the absorbed dose on the crosslinking rate in air leads to a change in the crosslinking rate of SKN-40-PVC-30, and the nature of the effect of air on the process rate depends on the radiation intensity. At a dose of 500 kGy, the radiation-chemical yield of the number of crosslinks is 4.8 per 100 eV of absorbed energy (see Fig. 4, curve 1). At high radiation doses (over 500 kGy), a decline in the efficiency of the radiation-chemical yield of crosslinks is observed. From the analysis of studies in a vacuum it is evident that in the process of crosslinking the rubber SKN-40-PVC-30, under the influence of radiation, at the same dose values, the yield of crosslinks in the rubber is lower. From Fig. 4 it is evident that in a vacuum the concentration of crosslinks, at a dose of 500 kGy, is $3.3 \cdot 10^{-5}$ mol/cm^3.

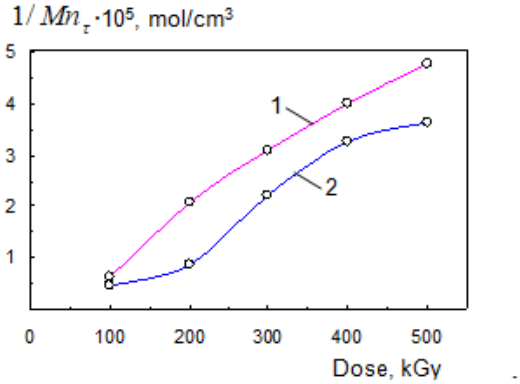

Fig. 4. Dose dependences of the concentration of crosslinks SKN-40-PVC-30: 1 – in air; 2 – in vacuum

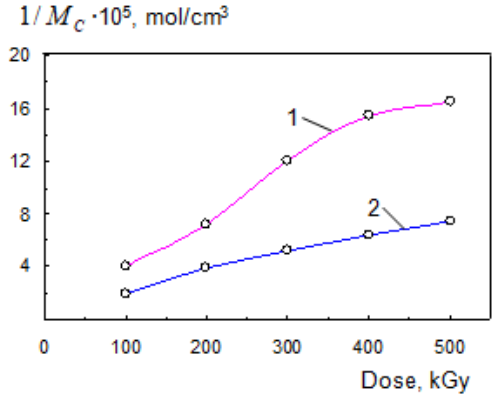

Fig. 5. Dose dependences of the concentration of the number of network chains SKN-40-PVC-30: 1 – in air; 2 – in vacuum

This can be explained as follows: according to prior studies [11, 13], with an increase in the radiation dose, chain oxidation processes developing in elastomers intensify due to the presence of traces or oxygen-containing groups, which can lead to additional crosslinking depending on the nature of the elastomer. It is possible that this circumstance leads to a noticeable increase in the yield of crosslinks in SKN-40-PVC-30 at a dose of 500 kGy.

The results of the experiment show that the accumulation of crosslinks during irradiation in air occurs at a higher rate than in a vacuum.

The process of crosslink formation in SKN-40-PVC-30 penetrates very intensively. At the same time, the effective yield of crosslinks at a dose of 400 kGy is 14 bonds per 100 eV and 19 bonds at doses above 400 kGy, which is higher than the values given in works on SKN-40 [7].

At the same dose (400 kGy), the yield of crosslinks in a vacuum is 10 bonds per 100 eV). It should be noted that the change in the average molecular weight (M_n = 87.0 thousand) is associated with crosslinking processes, which lead to an increase in the number of crosslinks in the rubber molecule. With increasing radiation dose, chain oxidation processes developing in rubber as a result of the presence of traces of oxygen and oxygen-containing groups intensify, which can lead to additional crosslinking or additional destruction depending on the nature of the rubber. Perhaps, it is this circumstance that leads to an increase in the yield of crosslinks in SKN-40-PVC-30 rubber. Indeed, with an increase in the radiation dose, the value of the yield of crosslinks in rubber decreases with an increase in the density of the spatial network (Fig. 5).

As a result of the study of the structural parameters of the irradiated rubber SKN-40-PVC-30, by the sol-gel analysis method it was established that the value of the radiation-chemical yield of crosslinks in butadiene units, in the range of the irradiation dose of 300...500 kGy, is 14–19 bonds per 100 eV, which significantly exceeds the corresponding value for high-molecular butadiene-nitrile rubber SKN-40 (9 bonds per 100 eV) [15].

Therefore, using the sol-gel analysis method, it was established that the radiolysis of SKN-40-PVC-30 rubber results in an increase in the content of unsaturated groups within the elastomer molecule and an enhanced sensitivity to radiation, which is manifested in an increase in the crosslinking yield. It is shown that the yield at a dose of 500 kGy in a vacuum is 3.3 crosslinks per 100 eV, and in air – 4.8.

The nature of the obtained dependences of the crosslink yield on absorbed dose and FTIR spectra indicate that when the rubber mixture is irradiated at a dose of 800 kGy in air, double bonds of various configurations appear. In this case, the distribution in SKN-40-PVC-30 rubber is 16% 1, 2, 64% 1, 4-trans, and 20% 1, 4-cis isomers. At the same dose (500 kGy), the yield of crosslinks in a vacuum is 10 bonds per 100 eV, and in air – 19 bonds.

CONCLUSIONS

The effect of γ-irradiation on the structure and structural parameters of the network in a mixture of nitrile butadiene rubber with polyvinyl chloride (SKN-40-PVC-30) was investigated using sol-gel analysis and FTIR spectroscopy method. A comparison of data on changes changes in the yield of crosslinks, transverse bond formation, double bond distribution, and structural modifications based on FTIR spectra indicates that, during the radiolysis (radiation-induced chemical transformation) of SKN-40-PVC-30, there is a general increase in unsaturation within the rubber matrix. This is accompanied by enhanced radiation sensitivity, evidenced by an increase in the effective yield of crosslinks and transverse bonds in the irradiation dose range of Φγ = 100…500 kGy.

However, at higher doses (Φγ = 800 kGy), the contribution of chain scission processes becomes significant, leading to pronounced structural changes. These include radiation oxidation, conformational redistributions accompanied by the disappearance and broadening of characteristic bands of trans- and cis-isomers, and increased amorphization of the structure. The obtained data indicate that the material exhibits good radiation resistance at doses up to Φγ ≤ 500 kGy.

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ВПЛИВ γ-ОПРОМІНЕННЯ НА СТРУКТУРУ ТА СТРУКТУРНІ ПАРАМЕТРИ СІТКИ СУМІШІ БУТАДІЄН-НІТРИЛЬНОГО КАУЧУКУ ТА ПОЛІВІНІЛХЛОРИДУ

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Вплив γ-опромінення на структуру та структурні параметри сітки суміші бутадієн-нітрильного каучуку та полівінілхлориду (СКН-40-ПВХ-30) у діапазоні поглинених доз Φ_γ = 100...800 кГр на повітрі та у вакуумі досліджували методом ІЧ-спектроскопії з перетворенням Фур'є та золь-гель-аналізу. Результати показують, що опромінення суміші СКН-40-ПВХ-30 на повітрі сприяє вищій швидкості зшивання, як порівняти з опроміненням у вакуумі у діапазоні доз опромінення Φ_γ = 100...500 кГр, проте при $\Phi_\gamma \geq$ 800 кГр проводився процес розщеплення. Дослідження підтверджують, що суміш СКН-40-ПВХ-30 може бути ефективно використана при Φ_γ = 100...500 кГр.