

Investigations of the Influence of Chlorine Contained Aromatic and Maleimide Compounds on the Structure of the Vulcanizates Net on the Base of Polyblend and the Creation Technology of Heat and Radiation Durable Materials

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Abstract The structural parameters (molecular mass, plasticity, number of chains' net and cross-linked molecules) of thermal vulcanizers on the basis of butadiene-nitrile rubber and polyvinyl chloride blends were studied by viscosity and sol-gel analysis methods. An alteration of polar groups content (-C≡N, C=O, C-Cl) and unsaturation of thermal and thermal irradiated vulcanizers were investigated. According to alterations of residual deformation's accumulation, relaxation of tension and equilibrium modulus in air and in fuel, the properties of elastomers were established at temperature (373-473K) and radiation (1000-2000 kGy) aging.

Keywords Blend, Cross-linking, Polyvinyl chloride, Irradiation, Rheology

1. Introduction

One of the usage requirements of elastomer materials (EM) is the stability of them against aggressive environment (oil, high temperature and radiation) due to their exploitation area. Therefore, data which concerns the regularities of EM aging in aggressive environment have directly application importance.

It is obvious from the references [1-7] that for the enhancement of resistance of EM, based on butadiene-nitrile rubbers, they are exposed to the structuring by low-molecular compounds (thiazole, thiurame and captax). However, as shown in references [4, 5], the obtained EM which contains polysulfide cross-linked bonds for a long time of action of sealed liquid and temperature is exposed the main alterations in physical and mechanical parameters of compacting (hardness, strength, residual deformation, dynamic endurance). Low aggressive-stability of EM is explained by their structural peculiarities, in particular, by chemical bonds between high molecular and low molecular

compounds and also by difference in energies of bond [8-10].

As well as known, sulfur is the primary vulcanizing agent in the manufacture of elastomers. With the participation of these compounds the rate of sulfur gases release increases and these gaseous forms are harmful to environment.

In the references [11-19], data of various factors influence on mechanical and chemical processes which occurs in saturated and unsaturated elastomers are represented.

The authors [1, 3, 6, 7, 20] have studied the behavior of some strained rubbers, based on NBR by the influence of γ - irradiation. However, in references, clear instructions regarding to radiation-stable elastomers on the basis of SKN-40-PVC-30 (polyblend-mixture of acrylonitrile butadiene rubber and polyvinyl chloride) are absent.

Modern oil and atomic industry require the creation reliable hermetic equipment for mobile and immobile connections. According to this issue, data about the influence of various factors on the aging process of elastic polymers acquires an enormous importance. Such information is important for some practical purposes as well as the scientific aims.

The elastomers are relevant now in spite of their destruction investigations in exploitation conditions. With the development of oil, mechanical engineering industry and

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the atomic technique, an implementation of the new equipment and aggregates require the creation of the new synthetic elastic materials which are exploited in complex dynamical and climatic conditions.

It is known that maleimide compounds (4,4-dithio bis N-phenyl maleimide) are the thermal stabilizer and it efficiently protects EM, based on unsaturated diene rubbers from oxidative aging and provides high stability of materials during exploitation in liquid aggressive media [21-26].

It was interesting to use these products as anti-aging agents which possess high reactivity and undergo the reaction only at their decomposition temperatures (over 373 K).

In selection of low-molecular compounds for structuring of rubber SKN-40-PVC-30, the chemical interaction at the border of two phases was basically considered. It should be noted that elastomer SKN-40-PVC-30 has a high Defo hardness (1700-2500 gs) and worst mixing with low-molecular products. For facilitation of plastification process on rolls and giving elasticity, epoxide resin ED-5 was used as a modifier. Zinc oxide, oil PN-6 and heavy oil which are introduced into initial composition have also been used as an activator. For estimation of efficiency of action of chlorine containing aromatic compounds hexachlorine p-xylene (HCPX), which easily soluble in polyblend during mechanical plastification was used as cross-linking agents. C-Cl bond, contained in polar group activates the output of cross-linking in macromolecule. Pre-vulcanization during addition of HCPX to mixture instead of sulfur has not been detected. Main regularities of the protective effect of sensitizer triazine compounds (2-Amino 4,6-bis trichlorine methyl symm triazine ABTST) under the influence of heat and gamma radiation stored in their filled vulcanizers. In selection of carbon black (P324) [27-29] the following parameters were taken into consideration: structure, elemental composition, size of particles, oil number and

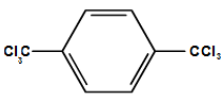
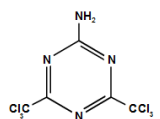
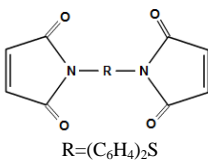
specific surface (used carbon black particle sizes is 29-32nm, specific geometric surface 85-90 m²/g, and oil number 115-200ml/100g).

For the providing high elasticity of the vulcanizers, and the decrease of strength of PB, diene epoxy resin (ED-5) and naphthenic oil PN-6 were introduced to the content of modifier. In this work the structure and properties of elastomer on the basis of polyblend (mixture of butadiene-nitrile rubber with polyvinyl chloride-SKN-40-PVC-30) have been studied during aging processes in air, in fuel and under action of radiation.

2. Experimental

The investigated object is the mixture of acrylonitrile butadiene rubber with polyvinyl chloride-polyblend SKN-40-PVC-30, which is obtained by the joint coagulation NBR (content associated acrylonitrile 37.8%) with PVC (70:30). According to the study of IR spectroscopy in butadiene part of the polymer, during the polymerization at 300K, isomeric composition of the double bonds are 1,2-isomer 14.3%, 1,4-isomer 14.8% and trans-1,4-isomer 70.9%. Macromolecules of SKN-40-PVC-30 polyblend consist of statically distributed butadiene, acrylonitrile and polyvinyl chloride. During vulcanization of polyblend, isomers influence to the microstructure, molecular weight and the yield of cross-links. Therefore, the average molecular weight and (Mn = 69 thousand, Mw = 226 thousand, Mw/Mn=3.27) [1] polydispersity were determined, by gel permeation chromatography prior to curing processes in polyblend. For this step the HG-1302 liquid chromatography was used. Three columns filled with Waters[®] sterogel with different pores. The solvent was chloroform and flow rate and temperature conditions were 1 ml/min and 298 K ± 0.1, respectively.

Table 1. Compound tested as cross-linking (1), sensitizers (2) and stabilizing (3) vulcanization agent for polyblend (SKN-40-PVC-30)

	Name of low molecular compounds	Structure	Mol. Weight	Melting point, K	Activation Energy of PB, kcal/mol	physical state	toxicity
1	Hexachlorine p-xylene (HCPX)		289	355	24.6	powder	—
2	2-amino-4,6-bis trichlorine methyl symm triasine (ABTST)		330	400	18.0	powder	—
3	4,4-di tio bis -N phenyl maleimide (DTBPM)		191	391	17.5	powder	—

The following composition of filled polyblend mixture contains these compounds by weight: SKN-40-30-PVC-100; HCPX-4.0; ABTST-2,0; DTBPM-3,0; ZnO-5,0, ED-5-6,0; naphthenic oil (PN-6)-1,5; carbon black (P324)-50,0. The tested physical properties of low molecular compounds are shown in the table 1.

Plastication of polyblend with components was carried out on laboratory mill at 300K. After cooling, the samples were cured at about 423 K \pm 2 during 40 minutes in vulcanizing mold. Temperature durability of the elastomers was assessed in thermostat between 373 and 473 K.

Irradiation of thermal vulcanizers was performed on Co⁶⁰ source. Duration of exposure was 2000 kGy. Power of the radiation source during the study period was 1,42 Gy/s. The particle size in the final mixture was 1-3 mkm. The absorbed dose in the samples was calculated by comparing the electron densities and dosimetry systems [30-32]. The intrinsic viscosity of thermal vulcanizers determined on the basis of SDS in toluene at 293 K by a well-known method with the use of Ubellohde type viscometer. The calculation was performed by Mark–Houwink equation: $[\eta] = kM^\alpha$, constant values were $K = 4,9 \cdot 10^4$ and $\alpha = 0,64$ (for toluene).

Yield of structure parameters (number of chain grid $1/M_c$ and the number of cross-linked molecules $1/M_{n_r}$) in unsaturated polyblend was determined with Flory-Rehner equation [33] and has also been used the equation of the high elasticity theory, which gives the opportunity to qualitative evaluation of vulcanization grid density of unfilled and filled elastomers.

Rheological composition of elastomers and blends were determined by known methods [34]. Structural parameters of the filled and unfilled vulcanizers were determined by sol-gel analysis. Alterations in the content of polar groups (C≡N, C=O, C-Cl) during aging process of unfilled samples were determined by IR spectroscopy to change the relative optical density (ROD) [35-37]. Deformation, tensile and relaxation properties of the elastomer were evaluated by aging at 373- 423 K [38] in air and in the fuel TS-1 (white spirit + cyclodecane + ethyl mercaptane, 60:20:20).

The analogous experiments were carried out for thermal vulcanizers at their aging under the action of absorbed dose of irradiation (1000-2000 kGy).

The work consisted of three stages: 1) development of cross-linked systems for rubbers SKN-40-PVC-30; 2) investigation of structural parameters of the prepared vulcanizers; 3) study of influence of temperature and radiation to aging process of elastomers in air and in fuel.

3. Results and Discussion

Cross-linking of polyblend with low-molecular compounds, as a rule, consists of interaction of components of various structure. Basically, any technological characteristics of mixture of butadiene-nitrile rubber with polyvinyl chloride, obviously, will be function of relative content of each component. Therefore, determination of elastic-viscous properties of SKN-40-PVC-30 rubber is an important test which cross-linking of polymer systems should be subjected.

The elastic-viscous properties are related with structure and molecular mass during plastification with low-molecular compounds. As the results of investigations, mechanical plastification of mixtures (without technical carbon) is characterized by the change of plasticity, rigidity and Mooney viscosity with the increase of plasticizing time (Figure 1 and 2).

It is seen that the insignificant changes of plasticity are observed in mechanical plastification of model mixtures for 20 min. An increase on plasticity time up to 30 min leads to insignificant changes of plasticity parameters and an increase of plastification time up to 90 min deteriorates dramatically. Obviously, this phenomenon is connected with the beginning of destruction time in the samples by the increase of time on mechanical plastification of elastomer mixtures does not lead to essential changes of rigidity coefficient and remains steadily in the ranges between 1000-1200gs (figure 1, curve 2).

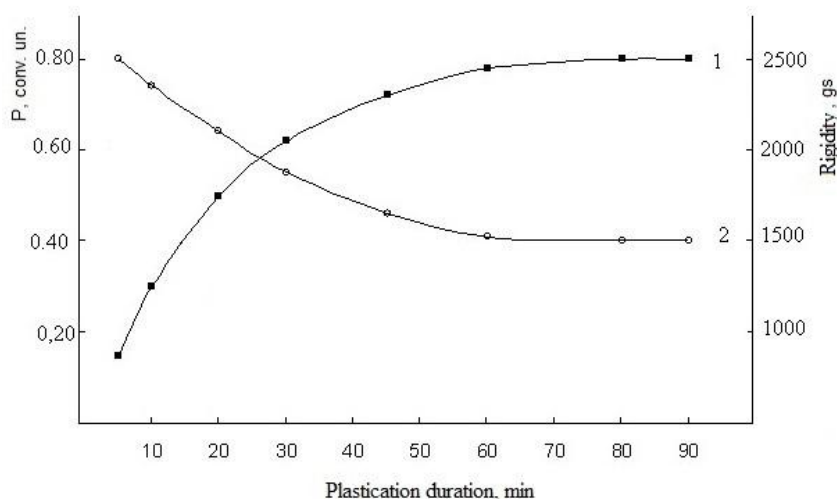


Figure 1. Dependence of plasticity (1) and rigidity (2) of elastomer mixtures on the basis of SKN-40-PVC-30 on plastification duration

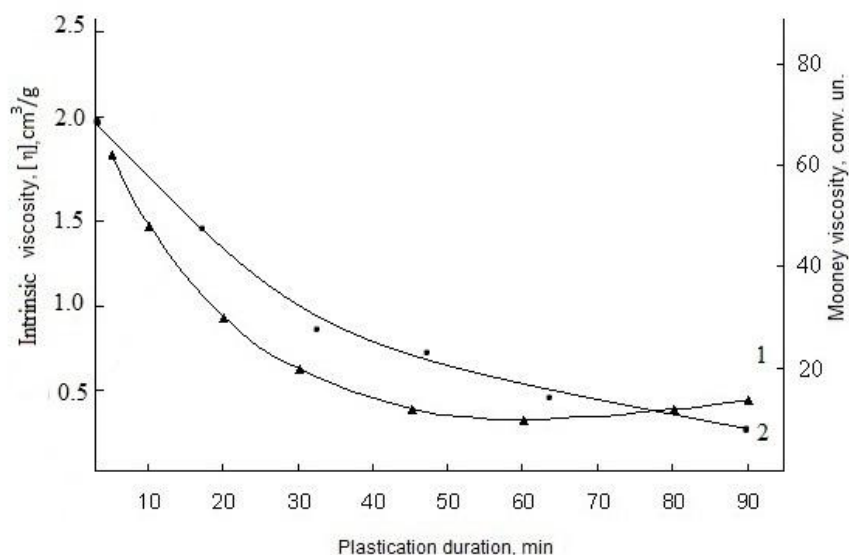


Figure 2. Dependence of Mooney viscosity(1) and intrinsic viscosity (2) of elastomer mixtures on the basis of SKN-40-PVC-30 on plastification duration of unfilled mixtures on the base of elastomer SKN-40-PVC-30

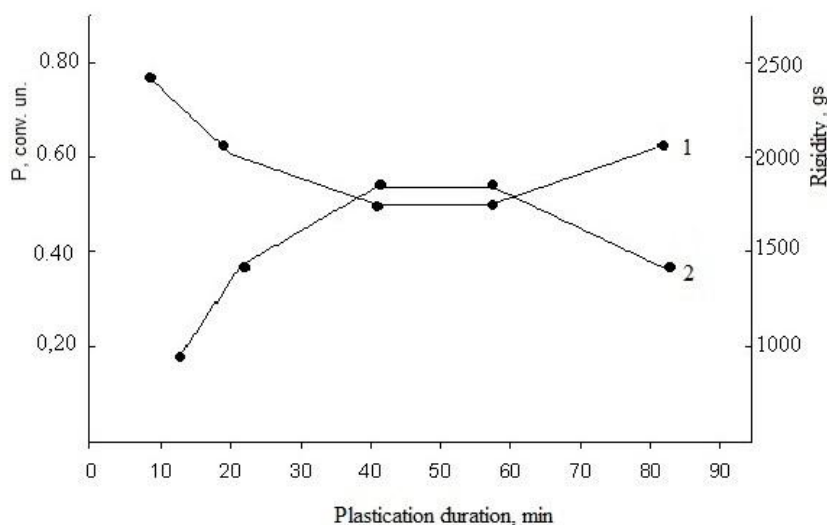


Figure 3. Dependence of plasticity (1) and rigidity (2) of filled mixtures on the basis of SKN-40- PVC-30 on heating duration (423 K x 90 min)

A decrease of rigidity has been apparently connected with mechanical plastification and influence of low-molecular compounds under the action of applied forces where within the interaction appear free radicals.

Kinetics of characteristic viscosity's alterations of elastomer mixture is described by monotonously diminishing curves (Figure 2, curve 2).

In this case for mixtures a large rate of change on properties in mechanical plastification for 90 min is observed. On the basis of characteristic viscosity data in mechanical plastification the value of molecular mass has been determined from 220000 to 135000 in the polymer mixture.

The process of cross-linking of elastomer mixtures after heating in electric press under the combined action of carbon black (P324) is characterized by absolutely distinctive regularities.

The obtained data (Figure 3) is the evidence about that plasticity of thermal vulcanizers has been decreased after 40

min since starting the reaction. The destructive changes of thermal vulcanizers are completed and, further, at 60 min plasticity is increased up to achievement of constant value (1100 gs).

The quantity and composition of sol fraction are the most sensitive parameters of net's structure. The results of sol-gel analysis of studied vulcanizers are presented in figure 4. It seems that a quantity of sol fraction depends on number of network chains. A quantity alteration of sol at constant $1/M_c$, molecular-mass distribution (MMD) is observed and it obviously, has been connected with peculiarities of adsorption of elastomer of disperse medium.

The results of determination (by means of sol-gel analysis) of structural parameters of vulcanization network formed under action of cross-linking agent allow to characterize entirely the properties of mixtures on the basis of rubber SKN-40-PVC-30, prepared with the participation of technical carbon.

Data presented in Figure 4 which depicts the chains number alteration in cross-linked molecules within cross-linking time indicates that cross-linking in presence of technical carbon takes place most efficiently.

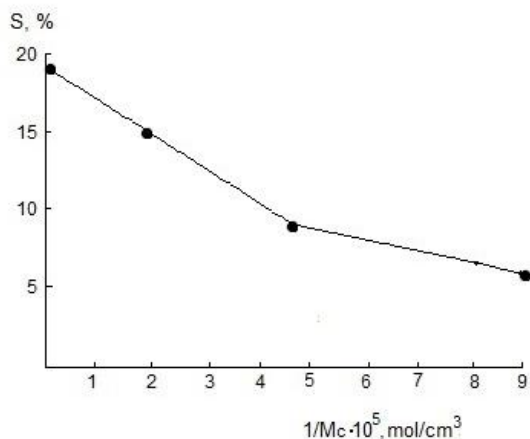


Figure 4. Dependence of quantity of sol-fraction on concentration of number of chains of networks of filled vulcanizers on the base on SKN-40-PVC-30

Aforementioned experimental data and observations which have been made previously [39] allow describing the action of low-molecular compounds in process of cross-linking of the SKN-40-PVC-30 elastomer. Cross-linking rate in the main period can be considered as a consequence of formation in thermal dissociation HCPX which is capable for interaction with elastomer.

Density of vulcanization networks of vulcanizers prepared with the participation of HCPX is a consequence of the fact, that the applied HCPX possesses structuring action leading to formation of C-C bonds between molecules of elastomer.

It should be noted that when a number of cross-linked molecules in SKN-40-PVC-30 rubber becomes considerable, durability of vulcanizers decreases. In essence, as it seen from figure 5, a decrease of break resistance occurs at the formed chemical bond C-C becomes stable and its relative elongation decreases, in a result observes a monotonous increase of concentration of active chains ($1/M_c$).

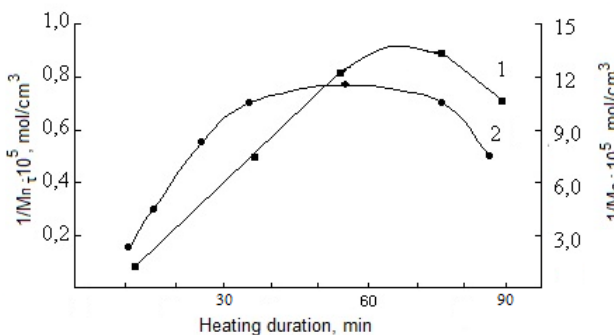


Figure 5. Dependence of concentration of structure parameters of network of cures on the basis on SKN-40-PVC-30 on heating duration. 1-number of cross-linked molecules, 2-number of chain network

Thus, the obtained data allows considering that in mechanical plastification of model systems on the basis of

the SKN-40-PVC-30 rubber completely dissociates in volume of elastomer and during heating of low-molecular HCPX becomes effective structuring agents for the SKN-40-PVC-30 rubber.

For the interaction assessment of dosage on outputs of the number of chains grids in the systems SKN-40-PVC-30+HCPX, SKN-40-PVC-30+ABTST and SKN-40-PVC-30+DTBPM the following results were determined: in the SKN-40-PVC-30+HCPX system the output increased and was equal to $1/M_c=8.2 \cdot 10^5 \text{ mol/cm}^3$ (figure 6, curve 1). Practically close density of nets of vulcanizers achieved in the SKN-40-PVC-30+ABTST mixture and was equal to $1/M_c=6.9 \cdot 10^5 \text{ mol/cm}^3$ (figure 6, curve 2). In the presence of DTBPM maximal value is observed and it is equal to $1/M_c=4.7 \cdot 10^5 \text{ mol/cm}^3$ (figure 6, curve 3).

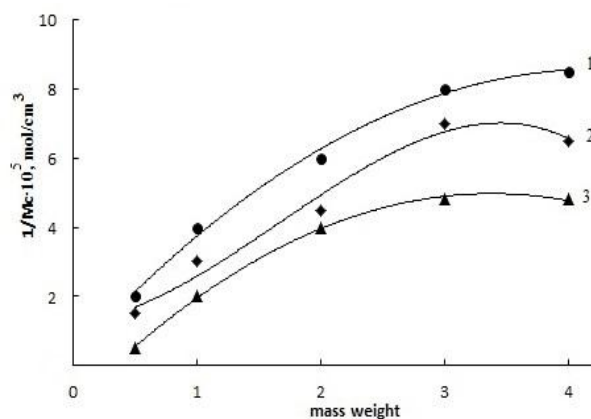


Figure 6. Influence concentration of HCPX, ABTST and DMPM to the yield of number chain networks on base SKN-40-PVC-30

For the determining the influence of heat to the aging process static tension rubber which is made from SKN-40-PVC-30 was aged in ambient condition and in a statistically deformed state tensile ϵ 10 and 20% (figure 7).

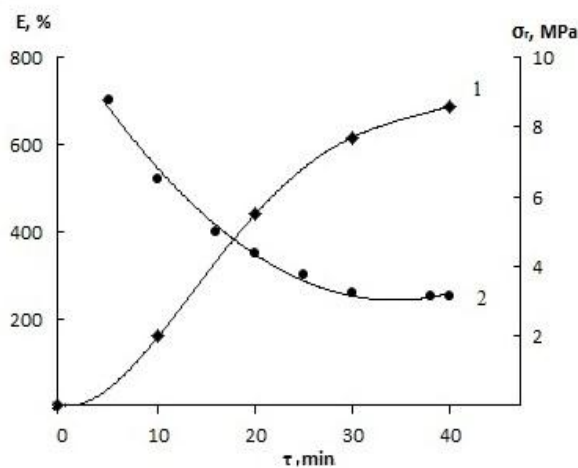


Figure 7. Influence of time duration to the properties of the cures (SKN-40-PVC-30 -100, HCPX -4,0 + ABTST-2,0 + DTBPM-3, 0) 1-tensile strength; 2- Elongation (mode 423 K \times 40)

For clarifying the role of heat on aging, studied rubbers were incubated at 373-473 K for 24hours.

Properties of rubbers in aging processes in air and in

thermostat were characterized with the deformation durability properties. Persistence of statically strained rubbers was evaluated by time up to appearance of cracks.

The investigations showed that cross-linking rate of SKN-40-PVC-30 elastomer during aging process in air increases with the rise of temperature (table 2).

The most intensive cross-linking is observed at temperature over 398 K. During the aging process in fuel at 398 K, as well as in air, cross-linking pace increases with the temperature rise and over 398 K changes insignificantly. In this case, aging process of elastomer in fuel appears in low degrees than in air. The low degree of cross-linking of elastomer in fuel in comparison with aging process in air has apparently been stipulated by decrease of oxidation rates of elastomer and thermo-cross-linking with the participation of nitrile groups and also by influence of swelling on processes of destruction and cross-linking. On the other hand, aging in fuel at 473 K is higher than at 398 K and accompanies decrease of content of C≡N, C=O, C-Cl groups. The obtained results indicate that decrease of cross-linking and polar groups between 398 K and 473K depends on increase of contribution of destructive processes during the aging of elastomers. It can be concluded that the observing change of polar group decrease during aging process in fuel between 398 K and 473 K explains basically by proceeding of oxidation processes. It is known that [1] for deformation characteristics of elastomers for a long time action of loading, increased temperature and active medium are necessary. One of these conditions is sufficient for change of deformation properties of elastomers. The work stability of elastomer

materials can be characterized with sufficient accuracy by means of study of accumulation of residual deformation, equilibrium modulus and relaxation stress.

As it seen from table 2, during the aging process in fuel TS-1 in stressed state, the alteration of elastomer's properties is observed, specially, an accumulation of residual deformation with temperature rise continuously grows as it occurs in air.

The obtained regularities confirm aforesaid conclusions about increase of contribution of aging destruction in fuel and in air. An increase of contribution of destructive processes in presence of static pressure is one of the attributes of mechanical activation of chemical process. The changes of conditionally-equilibrium modulus of elastomer samples during aging process in air and in fuel, presented in table 2, indicate an increase of fraction of destructive processes in both aging conditions. The obtained results are the evidence that the arising swelling in fuel and mechanical pressures activates the destruction process of macromolecules, i.e. aging proceeds as a mechanically activated chemical reaction.

It is seen from table 2 that the changes of relaxation stress after aging process of elastomers are different. Difference in change of relaxation stress can be explained with high adsorption capacity. Difference of relaxation processes in air and in fuel is explained by two elementary processes of relaxation in elastomers: 1- regrouping of supramolecular structure; 2- regrouping of chemical bonds [33].

Table 2. Influence of temperature on aging of elastomer on the base of SKN-40-PVC-30 in air and in fuel

T, aging K	Degree of cross-linking, γ (24h)	Change of content of polar groups (ROD)				Residual deformation at compression (E, res., 20%, 24h)		Equilibrium modulus, E_{∞} % from initial		Relaxation stress σ_t/σ_0 , MPa	
	In air	In fuel TS-1	C≡N	C-Cl	C=O	In air	In fuel TS-1	In air	In fuel TS-1	In air	In fuel TS-1
373	9.7	3.6	0.44	0.57	0.57	60	40	20	40	0.9	0.8
398	12.8	5,9	0.38	0.50	0.50	70	50	40	60	0.8	0.7
423	16.8	8,3	0.30	0.41	0.41	80	65	60	80	0.7	0.6
473	21.0	12,4	0.20	0.36	0.36	90	75	80	100	0.7	0.5

Table 3. Influence of temperature on aging of elastomer of SKN-40-PVC-30 on air and in fuel

Absorbed dose, kGy	Yield of cross-link bonds, $N_c \cdot 10^{-19}$, cm^3		Change of content of polar groups (ROD) in elastomers at their aging in fuel			Residual deformation at compression (E, res, 20%, 24h)		Equilibrium modulus, E_{∞} % at initial		Relaxation stress σ_t/σ_0 , MPa (24h)	
	In air	In fuel	-C≡N	C-Cl	C=O	In air	In fuel TS-1	In air	In fuel	In air	In fuel TS-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

With the comparing of the values during aging of rubbers under heat in air and in the fuel (as it seen from table 2 and table 3) it can be concluded that the presence of 3.0 wt. of DTBPM in the rubber composition causes to the inhibition of thermal oxidative processes.

A measurement of stress of deformed elastomers exposed to irradiation is one of the most popular methods which are used in study of radiation aging process. In this case two problems are simultaneously solved: quantitative determination of structural alterations in irradiated thermal vulcanizers and estimation of radiation stability according to change of contact stress which is responsible for compacting capacity of material. A study of concentration change of cross-link bonds in unfilled thermal vulcanizers after aging process by radiation showed occurrence of co-vulcanization, i.e. formation of three-dimensional structure due to interaction of polymer radicals formed during irradiation of elastomers. Table 3 shows that during irradiation of thermal vulcanizers at 2000 kGy an output of cross-link bonds (N_c) in elastomer is $22,4 \cdot 10^{-19}$, cm^3 . More than 2000 kGy dose the radiation-chemical processes are intensified and destructive phenomena of double bonds are observed.

The investigation of change of polar groups content in irradiated thermal vulcanizers in fuel TS-1 showed that up to 1500 kGy the oxidative processes are not observed practically. For carrying out of quantitative comparison of changes of unsaturation, thermal vulcanizers have been irradiated up to 2000 kGy with the aiming of preparation of average-density networks. By use of such large doses for comparison is correct as change rate of saturation remains fairly unchanged at 2000 kGy. The change of unsaturation in irradiated unfilled thermal vulcanizers (table 2), and the absorption bands 970 cm^{-1} , corresponding to double bonds in 1.4 cis-configuration, 2240 cm^{-1} ($-\text{C}\equiv\text{N}$), 730 cm^{-1} ($\text{C}-\text{Cl}$) and 1720 cm^{-1} ($\text{C}=\text{O}$) [40-41] has been noted. The change of these bands after aging in fuel TS-1 can be caused by cis-trans isomerization after irradiation and also by consumption of double bonds. An increase of dose of irradiation (higher than 2000 kGy) leads to decrease of cis-unsaturation by 9%.

Thus, the observed changes of properties of irradiated thermal vulcanizers during aging process in fuel higher than 1500 kGy are explained by proceeding of oxidation processes and higher than 2000 kGy the destructive processes in polymer, which favor considerable fall of unsaturation. It is known that elastomers on the basis of NBR are widely used in atomic technique for preparation of sealing elastic materials. Therefore, a study of regularities of radiation aging of these elastomers in the wide intervals of adsorbed doses depending on chemical nature of elastomers is in considerable interest. The aging process of elastomers in wide range of adsorbed doses can be characterized by the change of residual deformation (E) at compression, equilibrium modulus (E_0) and relaxation stress rates (σ_t/σ_0) in static conditions of test. According to the GOST-9.701-79 radiation stability of elastomer materials in compressed state is estimated by adsorbed dose at which

$E_{\text{res}}=80\%$, and $\sigma_t/\sigma_0 =0.2$.

4. Conclusions

Analysis of the experimental results shows that cross-linking of polyblend which is prepared by emulsion polymerization of low-molecular cross-linking agents, sensitizers and antioxidants (HCPX, ABTST, DTBPM) usually consists of many components of various structures.

In principle, any technological characteristics of polyblend obviously will affect the function of the relative content and structural features of each component. This is largely due to the fact that the hyper branched macromolecule characterized by a high density. The most important fact is that the mechanical plastification occurs in a few changes in elastic properties of elastomers. During the processing of polyblend components by mechanical plastification, takes place its failure initial molecular uniformity and the destruction of an ordered structure. Reduction in rigidity during the mechanical plastification can be explained by an increase of elasticity and defects of net.

Correction equations theory of high elasticity gave the opportunity to use them mainly for qualitative assessment of the spatial grid density of vulcanizers. The results show that the low molecular weight compounds have structural parameters, thermal vulcanizes accelerate cross-linking yield, and by the cross-linked molecules increases monotonically with increasing cure time.

Comparison of the structural features of grids cured products (stapling) of reservoir with their elastic and physical and chemical properties suggests that in the presence of 4.0 wt. of HCPX, 2.0 wt. of ABTST and 3.0 wt. of DTBPM causes inhibition processes on aging temperature of elastomeric materials in air and in fuel.

The presence of polar groups ($-\text{C}\equiv\text{N}$, $\text{C}=\text{O}$, $\text{C}-\text{Cl}$) provides effective protection elastomer materials in air and in fuel.

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