

PACS: 81.05.Lg, 83.80.Ab, 82.35.Np, 61.82.Pv

## EFFECT OF GAMMA IRRADIATION ON MECHANICAL AND THERMAL PROPERTIES OF BUTADIENE-NITRILE RUBBER/ZnO NANOCOMPOSITE

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**Abstract:** This paper presents an experimental analysis of the effect of dose and dose rate parameters during gamma irradiation of nanocomposites based on NBR, influence of nano-sized zinc oxide and crosslinking agent disulfo- chloride benzene (DSCB). Nanocomposites were irradiated in vacuated ampoules by gamma radiation from a Co60 source to various doses (D) from 100 to 250kGy and the results of irradiated samples, compared to non-irradiated samples (D = 0). The thermal behavior of samples and changes of results due to gamma irradiated dose NBR/DSCB/n-ZnO nanocomposites has been studied by thermogravimetric analysis. The mechanical properties like tensile strength, elongation at break and modulus at different elongations were studied and compared with those of non-irradiated ones. The results indicated that gamma radiation improves the mechanical properties while the thermal stability is decreased. Characterization, properties, and structure of prepared nanocomposite were investigated and reported using FTIR, TGA, SEM analysis.

**Keywords:** Nitrile butadiene rubber, nano zinc oxide, gamma irradiation, mechanical properties, thermal properties

### 1. Introduction

Acrylonitrile–butadiene rubber (NBR) seals are commercially available for more than 50 years because of its low cost, excellent resistance to oil, fuels, and greases, and easy processability [1]. NBR belongs to the crosslinking type rubber when exposed to high energy radiation. It is well known that the exposure of crosslinking type polymers to radiation provides improved stability and mechanical properties. Radiation can produce crosslink densities like those obtained by sulfur curing. The type of carbon–carbon crosslink formed in this method improves the mechanical properties at the higher temperature.

When ionizing radiations are used, ionization and excitation processes may take place as the first effect of irradiation leading eventually to the formation of trapped free radicals, electrons or ions. Two adjacent free radicals could be formed which will couple together immediately leading to the formation of crosslink or mobile free radical will move around until it finds a partner to give again crosslink. Therefore, crosslinking in polymers upon irradiation may take place between carbon atoms far from each other.[2]

However, to crosslink NBR, high radiation doses are required to reach the desired crosslink density. But at high radiation doses, the mechanical properties are adversely affected due to the degradation induced by radiation.[3]

The effect of radiation dose on the mechanical properties of NR/BR blending system was studied [4]. A comparison was made between sulfur vulcanization and radiation vulcanization for

an optimal nature rubber (NR)/ butyl rubber (BR) blending ratio (60/40) at the dose range from 10 to 150kGy. The result shows that the mechanical properties, especially, tensile strength, elongation at break, and tear strength have been improved significantly by radiation–vulcanization.

An effective technique to improve the technical parameters and the spatial grid of polymer systems is the introduction of these nanoscale inorganic modifiers. [5, 6] The action of these modifiers is often due to their influence on the process crosslinking of polymers, including the ability to form a chemical bond.

It can be assumed that when cross-linking the butadiene-nitrile rubber (NBR) with disulfochloride-benzene (DSCB), the type of nano-oxide used will also affect the crosslinking speed of polymer mixtures and the properties of the elastomer.

In all probability, the role of nanopowders in this system is even more diverse, firstly, it can cause activation of the double bond of both rubber and the cross-linking agent [7] in the second, as in the systems described above, it is possible that it participates in the formation of adsorption bonds, in addition, Nano-oxides of metals can interact with hydrogen chloride, which is released when cross-linking unsaturated rubbers [8]. The resulting metal chlorides are catalysts for the crosslinking process. This is particularly true for crosslinking of the general purpose with chlorine-containing aromatic compounds.

Generally, nanocomposite materials can be regarded as a special group of materials that some unique properties which are potentially applicable in the wide range of areas. The addition of inorganic nanosized particles into the matrix of a polymer material leads to the formation of a new composite material which tends to exhibit different characteristic properties that actually differs greatly from the actual starting material. Attaining of homogenous nanosized dispersion during the synthetic procedure for the formation of the nanocomposite material can be very difficult, due to the agglomeration tendency of the nanoparticles. [9-11].

One of the attractive type nano modifiers are oxides of metals (OM) [12] which are several levels were equally dispersed in the polymer type matrix. [13]

It is believed that the reason for the unique effect of OM polymer systems is their large chemical activity associated with nano particle size (2-100nm). Thus, OM can have a double effect: to act as structures for the adjustable matrix or the ability of the formation of additional nodes, mesh links in elastomeric systems. Initiating crosslinking with OM and the possibility of reproducing the particles in the polymer matrix may also determine the improvement in mechanical properties of nanocomposites. Thus, the amplification effect: increase in dynamic endurance and tensile strength was observed with the introduction of the OM in the ethylene-propylene copolymer [14].

Recent advances in the field of processing of nitrile rubber (NBR) have led to the expansion of their modifications and allowed to receive new crosslinked elastomeric materials. Completed earlier studies of the structure and mechanical properties of various compositions NBR [15], showed that the NBR has a complicated structure, including C-C and  $-C\equiv N$  bond.

Zinc oxide is a widely used compound in butadiene nitrile rubber industry due to the excellent properties that show as an activator for radiation-chemical vulcanisation. ZnO is an important inorganic pigment for the rubber industry, and over the past 100 years, the rubber industry has used an increasing number of the physical and chemical properties of ZnO. Different approaches have been considered for reducing zinc levels. Between all the alternatives proposed, the use of nano–sized ZnO particles with high surface area seems to be promising.

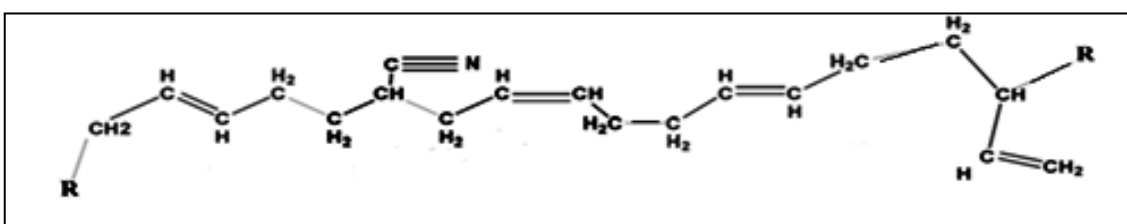
But the detailed effect of zinc oxide nanoparticles and effect of irradiation in the rubber compounds has not been studied. Hence, the aim of the present article is being studied properties of nanocomposites dependence of which are on the dose of the gamma rays developed by the

based on NBR in the presence of ZnO nanoparticles.

## 2. Material and methods

### 2.1. Materials

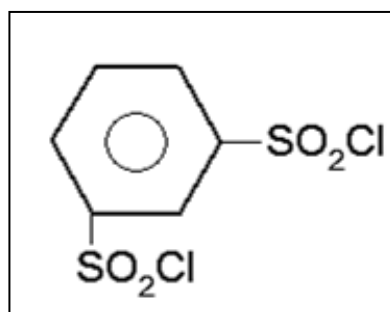
In this work, for receive nanocomposites was used butadiene-acrylonitrile rubber was copolymers of butadiene (BD) and acrylonitrile (AN) produced by means of an emulsion polymerisation reaction initiated by redox catalyst systems. The content of acrylic nitrile (AN) was 40% in the molecule. The Mooney viscosity was (ML1+4 (100°C):47). Chemical structure of NBR is shown in Scheme 1.



Scheme 1. Chemical structure of NBR

As activation crosslinking polymer used nano zinc oxide (ZnO). With the introduction of ZnO, nanopowders pay attention to the dimension (20-25nm) dispersion and purity (99.8). Specific surface (250 g/m<sup>2</sup>), true density 5.606 g/cm<sup>3</sup>. Powder dosage was varied in the range 0.8-4.0 phr. The nanomaterials had been obtained from the Inc. Houston, TX, USA.

In the process used crosslinking agent disulphochloride benzene (DSCB) aromatic compounds, which react readily with macromolecules NBR. DSCB is an organosulfur compound with the formula C<sub>6</sub>H<sub>4</sub>(SO<sub>2</sub>)<sub>2</sub>Cl<sub>2</sub>. It is a colorless viscous oil that dissolves in organic solvents, but reacts with compounds containing reactive N-H and O-H bonds. Chemical structure of DSCB is shown in Scheme 2.



Scheme 2. Chemical structure of DSCB

### 2.2. Methods

Nanocomposites based on NBR were prepared on two roll-mixing mills (outside diameter 470 mm, working distance 300 mm, a speed of slow roll 24 rpm and friction ratio of (1:1.4) in accordance with ASTM D3182-07. First, NBR (100 phr) was masticated for 2 min followed by

the addition of ZnO nanopowders and DSCB. (Table 1)

Table 1. Composition of the NBR-based rubber compounds [phr]

Ingredients	Contents, phr
NBR	100
DSCB	4.0
ZnO	5.0

Compounds were finally sheeted again in the rolling direction into slabs of about 0,2 mm thickness, and the sheets were pressed in clean molds of a hydraulic press. The molds were brought to 423K and held at this temperature for 5 min at a pressure of 160 kg/cm<sup>2</sup>.

For the irradiation of the samples were placed in a 1 g vial of glass evacuated for hours 1,3 h 10<sup>-1</sup> Pa residual pressure. The exposure to radiochemical processing was performed in air at room temperature inside of the source Co<sup>60</sup>  $\gamma$ -radiation was used for irradiation giving a dose rate of about 4.9 kGy/h. Irradiation of samples was carried out at the Institute of Radiation Problems, Azerbaijan. The absorption currents of samples calculated by comparing the electron densities of the investigated dosimetry systems.

The molecular structure and isomeric composition of nanocomposites NBR were recorded on Fourier-transform IR Spectrophotometer (FTIR) in the 3000 to 700 cm<sup>-1</sup>. Rubber films were prepared by applying the solution (toluene) to a substrate and evaporation of the solvent constant.

For the morphological observation of NBR/DSCB/n-ZnO composites was used JEOL-JSM-5400 (Japan) scanning electron microscope (SEM).

The crosslink density ( $\nu_r$ ) of the vulcanisates was determined by their equilibrium swelling in toluene based on the Flory-Rehner equation [16]. The Huggins parameter of the elastomer-solvent interaction ( $\chi$ ) was calculated from the Equation (1):

$$\chi = 0.3809 + 0.6707V_r \quad (1)$$

where  $V_r$  is the volume fraction of the elastomer in the swollen gel.

The thermal stability of the vulcanisates was studied using a TGA/DSC analyzer. Samples were heated from 25 to 700°C under the nitrogen atmosphere with a heating rate of 10°C/min. The weight of the sample was about 5 mg.

The nanocomposite samples for mechanical tests carried out on dumbbell shaped specimens of 4 mm width and 50 mm length. Tensile strength (TS), elongation at break % ( $E_b$ ), and modulus at 100% elongation were determined measured in accordance with ASTM D-412 using a tensile testing machine P-5.

### 3. Results and discussion

#### Infrared spectroscopy (FTIR).

The fact of the formation bonds in the NBR system in the presence of nanopowders of zinc oxide, the crosslinking agent of DSCB and effect of gamma radiation is confirmed by the data of FTIR spectroscopy.

In order to identify the characteristic groups present on the surface of the nanocomposites samples that confirm the effectiveness of the modification process, were subjected to spectroscopic analysis Figure 1, depicts the FTIR spectrum of the nanocomposite based on NBR/DSCB/n-ZnO, at various radiation doses. (D= 0, 100, 250 kGy).

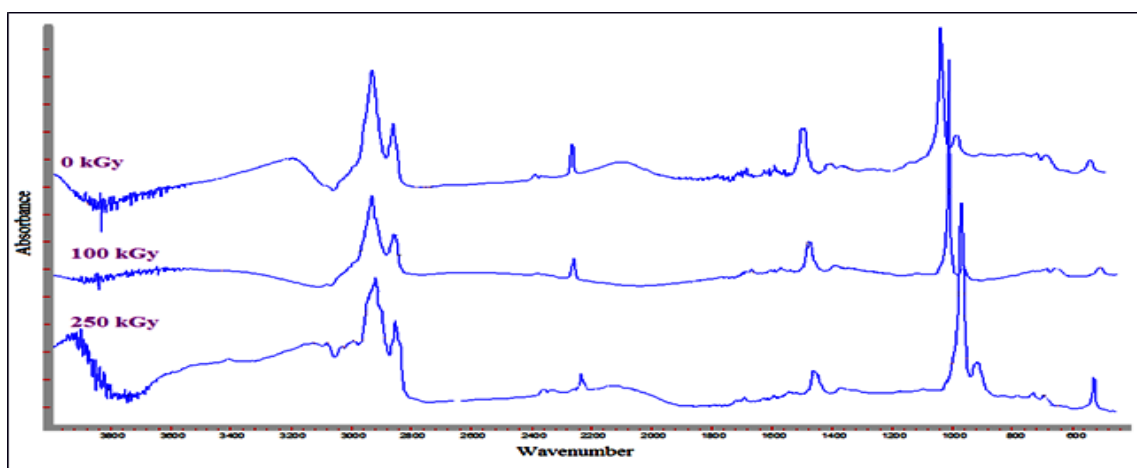


Fig. 1. FTIR spectrum of NBR/DSCB/n-ZnO nanocomposite at various radiation doses.

The main attention was paid to the intensity of the band stretching vibration of the nitrile group  $-C\equiv N$ , located in the area of  $2230\text{ cm}^{-1}$  and  $960\text{ cm}^{-1}$  is due to the  $-C-H$  wagging motion vibration of butadiene. The absorption band noticeable near  $1713\text{ cm}^{-1}$ , is attributed to the carbonyl group suggesting the changes induced by degradation reactions and ascertaining the type of degradation taking place via the initial irradiation. [17] FT-IR spectra of NBR/DSCB/n-ZnO nanocomposite exhibit absorption peaks distinctly at  $852\text{ cm}^{-1}$ ,  $745\text{ cm}^{-1}$ , and  $456\text{ cm}^{-1}$  was observed which is attributed to the presence of ZnO nanoparticles.

The emergence of new bands in the region adjacent to  $2240\text{ cm}^{-1}$  is probably due to the formation of complex compounds of groups  $-C\equiv N$  and zinc chloride. Visibly reduces the absorption intensity in the region of  $750\text{ cm}^{-1}$  due to the vibrations of  $C-Cl$  [18]. This gives reason to believe that during heating and irradiation (250 kGy) holds intensive elimination of chlorine from DSCB molecules. On cleavage of said chlorine, apparently, decrease in band intensity and  $1230\text{ cm}^{-1}$ , which are in the field of analogical IR absorption study substituted aromatic structures [19, 20]

#### Rheological and structure parameters

For the evaluate crosslinking processes of swelling samples in toluene solutions, the sol-gel analysis method [21] determined the structural parameters, the number of crosslinked molecules extracted within 48 h. cold acetone in an argon atmosphere of thermal and irradiated samples. In the presence of a ZnO nanopowder, the crosslinking reaction proceeds intensively Table 2.

Table 2. Rheological and structure parameters of system NBR/DSCB/n-ZnO

Crosslinking mode	Rheological and structure parameters of system NBR/DSCB/n-ZnO					
	Mooney viscosity, MO	Intrinsic viscosity $[\eta]$ , dL/g	The number of cross-linked molecules, $1/M_{irr} \cdot 10^4$ mol/cm <sup>3</sup>	The degree of crosslinking, $\gamma$	The content of sol fraction, S, %	The equal degree of swelling, $M_n$ , %
423Kx40'	58,0	9.0	1.56	21.6	0.59	67
100 kGy	28,0	0.54	0.78	12.0	0.19	102
150 kGy	30,0	0.41	0.66	11.0	0.24	96
250 kGy	34,0	0.36	0.58	9.0	0.34	98

It is known that the radiation-chemical yield of crosslinking ( $G_c$ ) and the formation of effective crosslinks in NBR depends both on the absorbed dose and on metal oxides [22].

The analysis shows that irradiation in the air at 293 K crosslinking of polymer systems increases insignificantly. In the irradiated system (NBR-DSCB-n-ZnO), the rate of crosslinking is higher than in nonirradiated systems. This is explained by the fact that the selected absorbed dose (100-250 kGy) renders a significant influence on both the process speed and the radiation-chemical yield ( $G$ ), the number of grid chains ( $1/M_c$ ) and the number of crosslinked molecules ( $1/M_{nc}$ ). At the same time, it should be noted that the activating ability of metal oxides is influenced by the particle size and the specific surface area. So nano-ZnO provides a greater speed of cross-linking of the mixtures compared to micro powders of ZnO. [23]

The amount of soluble sol fraction of (S) polymer systems after heat treatment (423K 40') is 0.59 and in irradiated samples at 250 kGy is 0.34 so that the polymer is completely insoluble, which indicates the cross-linking of polymer chain molecules (see Table 2).

As can be seen from the results obtained, the introduction of nanopowders metal oxides and also the cross-linking agent DSCB has a significant effect on the intrinsic viscosity ( $\eta_{ins}$ ), the absence of nanopowders in the polymer system proceeds slowly both in thermal and irradiated samples. (Fig 2)

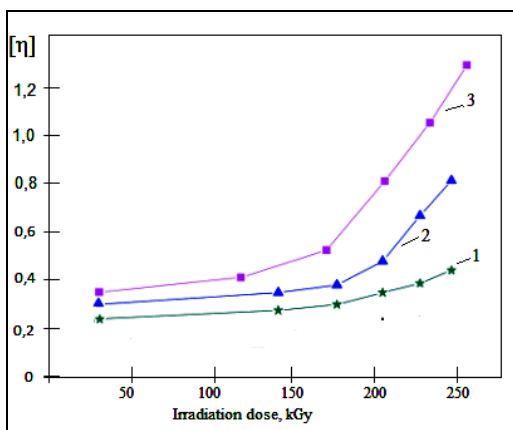


Fig. 2. The kinetics of change of the intrinsic viscosity polymer composite systems of irradiation dose: 1. NBR; 2. NBR-n-ZnO; 3. BNK-n-ZnO-DSCB

### TGA/DSC thermograms analyzer

The effect of nano-sized powders on the thermal properties of the NBR as a whole has not yet been studied sufficiently.

The degradation temperature of NBR/DSCB/n-ZnO blends has been analyzed by the thermogravimetric method that is considered one of the widely used techniques to illustrate the thermal stability of polymers over a wide range of temperature. The initial TGA thermograms for unirradiated and irradiated NBR blends are shown in Table 3.

Table 3. The effect of irradiation dose on the TGA thermogram of NBR/DSCB/n-ZnO composite

Mechanical properties	D <sub>100</sub>	D <sub>150</sub>	D <sub>250</sub>
Modulus at 100% elongation (MPa)	4	5,2	8
Tensile strength (TS), MPa	14	12	11
Elongation at break (E <sub>b</sub> ), %	450	260	225
Hardness, Shore A	65	70	78

For elastomeric mixtures based on NBR used in aggressive media, the main parameters are the stability of the temperature and hydrocarbon media (fuels and oils) in the temperature range - 40.+ 423K. Relations with this, of particular interest, are the study of the thermal characteristics of nanocomposites.

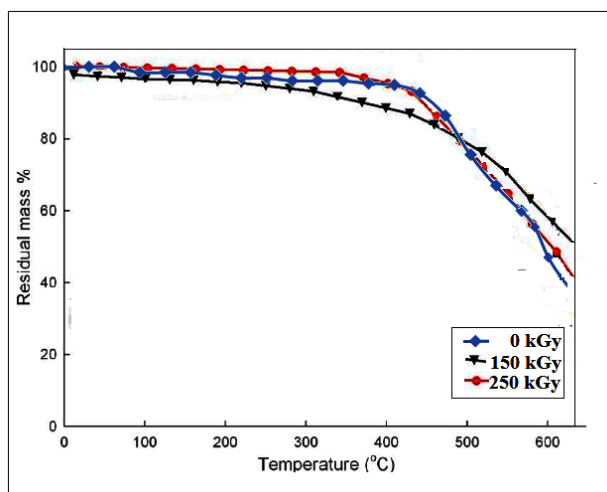


Fig. 4. The effect of irradiation dose on the TGA thermogram of nanocomposite

Analysis on the TGA thermogram of nanocomposites (NBR/DSCB/n-ZnO) shows that (Figure 4), the onset of degradation was at 561 K, whereas 10% at 623 K and 75% was at 683K. However, the reaction rate of the decomposition of the nano composite was small which the sample from the mixture of rubbers loses 10% of the initial mass. Thus, no appreciable mass loss in the thermogram above this temperature could be detected. The effect of irradiation dose on the TGA thermogram of the blend is presented in figure 4. The release of additional heat when the sample is heated allows one to assume the possibility of the reaction of the main chain of NBR on the residual double bonds.[25].

## Mechanical properties

Effect of radiation on nanocomposite based on NBR resulting in the formation of carbon-carbon bonds in the presence of n-ZnO and DSCB, during the cross-linking, zinc chloride is formed, which is capable of activating the crosslinking process in rubber. [26]

The change in the mechanical properties of nano composites in any physical field is ultimately due to the occurrence of competing processes of crosslinking, as well as to changes in the potential of intermolecular interaction. Mechanical properties, namely, tensile strength (TS), tensile modulus at 100% elongation, and hardness have been followed up as a function of irradiation dose. Physical and mechanical properties of filled vulcanizates, based on NBR are presented in Table 4.

*Table 4. Physical and mechanical properties of nanocomposites*

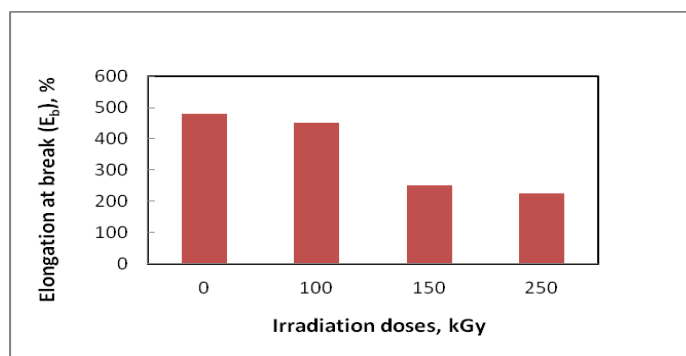
Mechanical properties	D <sub>100</sub>	D <sub>150</sub>	D <sub>250</sub>
Modulus at 100% elongation (MPa)	4	5,2	8
Tensile strength (TS), MPa	14	12	11
Elongation at break (E <sub>b</sub> ), %	450	260	225
Hardness, Shore A	65	70	78

A gamma radiation beam accelerator was used to generate free radicals on the carbon atoms of the NBR and coagent. The radicals may either be reacting with each other or graft on the coagent forming crosslinks. [27]

Table 4 shows the results for radiation curing of NBR by different radiation doses up to 250 kGy in the presence of nano-ZnO and DSCB. The D=100 kGy was found to increase linearly with radiation dose indicating the linear relationship of cross-link density with radiation dose, whereas the latter inversely lowers the values of M<sub>c</sub>.

The increase in values of V<sub>r</sub>, decrease in E<sub>b</sub>, Q and in solubility were considerable by increasing radiation dose indicating the increase of cross-link density of vulcanizate. Also, there is an increase in TS up to 100 kGy, beyond which there is a very slight decrease in TS values dose. The leveling off or decrease in TS by radiation may be explained as follows: TS of a polymer is a function of cross-link density and energy dissipation, at higher crosslinking density, i.e. at 250 kGy, the segments of the macromolecule become immobile, the system becomes stiffer and the elasticity decreases. [28, 29]

Also, it can be assumed that the rate of two processes, namely crosslinking and degradation may then occur with the same rate, hindered mobility of macromolecules due to increased rate of crosslinking may contribute also to decreasing the values of TS. [ 30] strength, elongation at break and hardness for irradiated and non-irradiated nanocomposites are shown in Figure 5 (1, 2, 3).





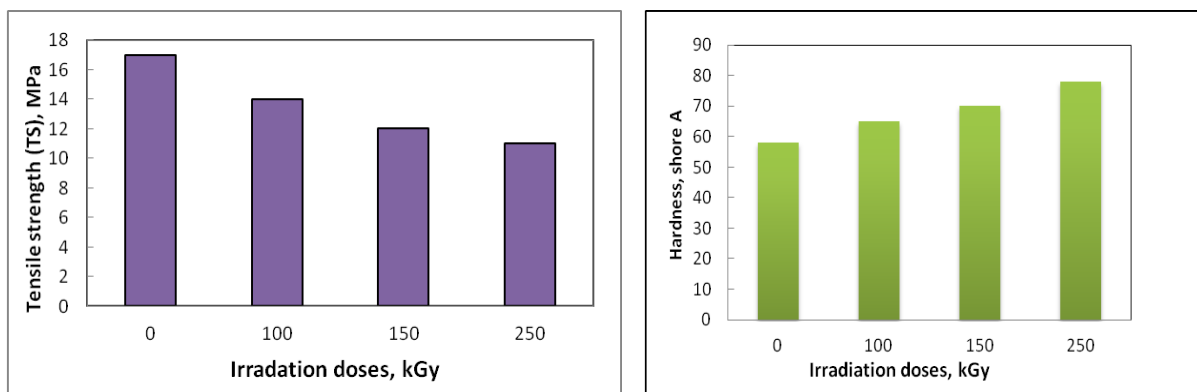


Fig. 5: Tensile strength, elongation at break and hardness for irradiated and non-irradiated nanocomposite

#### 4. Conclusions

The first time studied the chemical interaction disulfide chloride benzene (DSCB) compounds with nitrile-butadiene rubber (NBR) in the presence of nanoscale metal oxide powders. Considered in general terms the impact of nano zinc oxide during thermal and radiation crosslinking to influence the rheological properties of the vulcanizates. It is shown that using disulfide chloride benzene (DSCB) allows you to get the maximum growth of the elastomer molecular weight, with increased density in the quasi-system grid vulcanizates. Staple NBR, DSCB activated largely nano-sized zinc oxide powder, which acts as a scavenger of hydrogen chloride released during the reaction of the elastomer and cross-linking agent to form an effective cross-linking of the resulting zinc chloride is able to activate the crosslinking in the elastomer and to participate in creating focal cross ties. Studied polymeric systems are reticulated macromolecular structures in which a low molecular weight benzene disulfide chloride benzene (DSCB) enters into a chemical crosslinking initiator active nano powders together with sulfur and a filler. Additives, metal oxide nanopowders cause a change in the molecular chain of the elastomer and curing kinetics contributes to the formation of the chemical structure (C-S-C; C-C) and the properties of crosslinked elastomers.

Thus, the obtained results show that DSCB with nano-oxide metals can be used in vulcanisates based on NBR to increase the strength, conditional stresses, hardness of vulcanizates without the addition of special substances (organic peroxides, altaks) initiating its vulcanization.

#### Acknowledgments

This work supported by the Science Development Foundation under the President of the Republic of Azerbaijan Grant № EIF-KETPL-2-2015-1(25)-56/10/1

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## ВЛИЯНИЕ ГАММА-ОБЛУЧЕНИЯ НА МЕХАНИЧЕСКИЕ И ТЕРМИЧЕСКИЕ СВОЙСТВА НАНОКОМПОЗИТА НА ОСНОВЕ БУТАДИНО-НИТРИЛЬНОГО КАУЧУКА/ZnO

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**Резюме:** В данной работе представлен экспериментальный анализ влияния мощности дозы гамма-облучения на механические и термические свойства нанокompозитов на основе бутадиен-нитрильного каучука (БНК), а так же влияние порошка наноразмерного цинк оксида и сшивающего агента дисульфохлоридбензола (ДСХБ) на процесс сшивания. Нанокompозиты облучали в гамма-камере, вакуумированных ампулах при комнатной температуре с использованием источника  $Co^{60}$  в разных дозах: 0, 100, 150 и 250 кГр соответственно.

Сравнивали результаты изменения структурных параметров, тепловые и механические свойства облученных и необлученных нанокompозитов БНК/ДСХБ/n-ZnO ( $D = 0$ ) с использованием данных SEM, FTIR, DTA и универсальной испытательной машины (УИМ). Результаты показали, что гамма-облучение изменяет исходную структуру и улучшает механические свойства нанокompозита.

**Ключевые слова:** бутадиен-нитрильный каучук, нанокompозит, сшивание, гамма-излучение

## BUTADIEN-NİTRİL KAUCUKU/ZnO ƏSASLI NANOKOMPÖZİTLƏRİN MEXANİKİ VƏ TERMİKİ XASSƏLƏRİNƏ QAMMA-ŞÜALANMANIN TƏSİRİ

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**Xülasə:** Verilmiş işdə butadien-nitril kaucuku əsasında alınmış nanokompözlərin qamma-şüalanmanın dozasından asılı olaraq mexaniki və termiki xassələrin dəyişməsi, həmçinin, nanoölçülü sink oksid tozunun və tikici agent disulfokloridbenzolun (DSXB) tikilmə prosesinə təsiri, təcrübi analizlərin nəticələri təqdim olunmuşdur. Nanokompözlər vakuum ampulalarda, otaq temperaturunda müxtəlif dozalarda ( $D = 0, 100, 150, 250$  kGy) şüalandırılmışdır. Qamma şüalanmanın mənbəyi kimi  $Co^{60}$  izotopu seçilmişdir. BNK/DSXB/n-ZnO əsaslı müxtəlif dozalarla şüalanmış və şüalanmamış nanokompözlərin struktur, fiziki-mexaniki və termiki xassələri SEM, FTIR və DTA analiz metodları ilə tədqiq olunmuşdur. Alınmış nəticələr göstərmişdir ki, qamma-şüalanmanın təsirindən udulan dozadan asılı olaraq nanokompözitin ilkin struktur quruluşunu dəyişir və mexaniki xassələrini yüksəldir.

**Açar sözlər:** butadien-nitril kaucuku, nanokompözit, tikilmə, qamma-şüalanma