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Effect of Gamma Irradiation on the Physio-Mechanical Properties of Nitrile Rubber (NBR-6250) Composites Reinforced by Carbon Black N330

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Abstract

This study investigated the effect of the addition of carbon black N330, as a filler and supportive in the properties of Acrylonitrile Butadiene Rubber NBR-6250, by preparing composites of nitrile rubber containing different percentages of carbon black N330, and then conducting the Vulcanization radiation exposure to different doses of gamma radiation up to (KGy 250), The mechanical properties and the resistance characteristics of some petroleum oils were studied at different radiation doses. The study results showed an increase in tensile strength with the increase of the radiation dose and the increase in the proportion of carbon black loading, achieving the highest tensile strength (24 MPa) at a radiation dose of 150 KGy and a carbon black loading of 50wt%. Then, the tensile strength either stabilizes or begins to decrease with the continuous increase in the radiation dose and carbon black loading. While it was observed that the behavior of elongation at break is contrary to tensile strength behavior. An improvement was also shown in the hardness of the prepared compositions and their resistance to friction with the increase in the radiation dose due, which is attributed to the increase in the number of radiation-induced crosslinking reactions among the macromolecules of the rubber. The results of the study also showed a decrease in the percentage of swelling of the composites prepared in the oils with the increase in radiation dose and also with the increase in loading with carbon black, for instance, the swelling ratio of composites loaded with 50wt% carbon black decreased from 9% to about 5% with an increase in the radiation dose up to 250 KGy. This is because the increased of the crosslinking density of macromolecular chains of rubber with higher radiation doses hinders the penetration and diffusion of oil within the formed three-dimensional rubber network.

Keywords

Nitrile Rubber, Gamma Radiation, Radiation Vulcanization, Carbon Black, Swelling, Crosslinking

1. Introduction

Sealants are most often manufactured using traditional processing systems for rubber materials, such as vulcanization with sulfur or peroxide, which are carried out at high temperatures, causing damage to the properties of the final products through the occurrence of undesirable side reactions, which cannot be avoided, or controlled as they are These treatments are complex, have high-cost procedures, and lead to the generation of environmentally harmful gases, During vulcanization, sulfur reacts with rubber polymers, but this reaction also produces SO_2 as a byproduct. This chemical is a known air pollutant and poses severe risks to both ecosystems and human health. Sulfur dioxide is a highly reactive gas that contributes to acid rain, smog, and particulate matter formation.

The environmental impact of this energy consumption extends beyond CO_2. Vulcanization processes often rely on natural gas or coal-fired boilers, which release methane (CH_4) and nitrous oxide (N_2O)—greenhouse gases with 28 and 265 times the global warming potential of CO_2, respectively [1]-[3].

Radiation treatment using high-energy ionizing rays is an effective way to modify polymeric materials in clear and specific ways, with the aim of obtaining new and distinct properties that are difficult to obtain by other methods. The reactions that take place in polymeric materials as a result of their impact take place at room temperature so that the product can be used immediately after the end of the treatment process, which is of high accuracy and short time, and requires much less energy than traditional treatments when the same effect occurs in the material Polymeric [4] [5].

Unlike natural rubber, nitrile rubber shows a certain percentage of crystallization, and therefore there is a self-reinforcing effect on it. Relatively low, easy to manufacture and handle, and resistant to blistering by aliphatic hydrocarbons. As a result of its high resistance to oils, it is widely used in sealing applications, such as pipes, seals and O-rings [6]-[8].

NBR is a common polymer of butadiene and acrylonitrile, widely used in the manufacture of sealants and O-rings used in various transport methods, where the main part of NBR production is used in machinery and automotive industry due to its high resistance for oil derivatives [9] (Oil, hydrocarbons, grease) and its high resistance to swelling in aliphatic hydrocarbons in a wide range of temperatures [10]. The content of the acrylonitrile (AN) (18% - 45%) of the composition of (NBR), and the higher the content of the (AN) this reduces the elasticity of the product at low temperatures and increases its hardness [11] [12].

Carbon black is generally added to improve the final elasticity properties of rubber, to achieve the specifications imposed by the investment conditions for which it is manufactured, to reduce the cost of the product and to achieve the required reinforcement, significantly increasing the mechanical and thermal properties of the product. Factors that govern carbon particles include surface chemistry, size, shape and specific surface area. The most important method used to increase the properties of rubber and reduce its cost is to strengthen it by the addition of rigid materials [13], which improves the mechanical properties such as tensile strength, abrasion and tear resistance, as well as physical properties and chemical resistance, and depends on the amount of reinforcement and improvement in the characteristics of the type of additive used, Such as: carbon black, silica and clay [14]-[16].

Rubber nitrile is polymeric materials crosslinking by irradiation therefore, when irradiated, its molecular structure cross-linking. which improves its physical and mechanical properties. However, exposure of this rubber to high doses of radiation leads to deterioration of its mechanical and physical properties due to the interactions of dissociation of molecular chains dominate other interactions [17].

The aim of this work is to study the effect of gamma rays at different doses in the properties of nitride 6250 rubber, reinforced by different percentages of carbon black, and determining the optimal radiation dose for obtaining good mechanical and physical properties and determining the mechanical behavior of the material when adding different amounts of carbon black.

2. Materials and Techniques

2.1. Materials

Nitrile-butadiene rubber, under the commercial name of NBR-6250, was supplied by LG, Korea, with an average acrylonitrile content of (40wt%), Mooney viscosity ML1+4 (100 C) 50 + 5 and density 0.99 (g/cm^3). The filler was high abrasion furnace carbon black (HAF) N330, was supplied by Jscstakhanov Carbon Black Chemical Plant Co., Ltd particle size 28 nm, and surface area about 65 - 70 m^2/g, Iodine adsorption number 82[g/kg]. Oxide Zinc (ZnO) as activator supplied by Intermediate Chemicals Co., Ltd density (1.5 - 1.8 g/cm^3). Stearic Acid supplied by KLK OLEO (Shanghai) Co., Ltd Iodine Value (1 g/100g) (0.8 max). Rubber antioxidant IPPD (4010NA) of British origin made by Castle Chemicals Co., Ltd., with dark brown granular appearance, Molecular Weight: 226.31, ash content Max (0.2%), purity (GC) ≥ 0.97%.

Dioctyl phthalate (DOP) ($C_{24}H_{38}O_4$) plasticizer was poured by WSD chemical British origin, molecular weight (426.68), density at 20˚C (0.985g/cm^3) purity ≥ 99.5%.

Petroleum derivative as Gasoline from local source density 0.744 [g/cm^3], Octane Number (90 min), sulfur content (max 10 ppm). Engine oil SHDDE No (40)

Viscosity grade 40, Density at 20˚C (68˚F) = 0.887, Viscosity at 40˚C (104˚F) = 141.0 mm^2/s, Pour point = −24.0˚C/−11.2 ˚F, Flash point = 224.0˚C/435.2 ˚F. Mobil Hydraulic Oil M 46, Density at 15˚C = 0.875 Kg/L, Viscosity at 40 ˚C = 46 cSt, Pour Point = −25˚C, Flash Point C.O.C = 220˚C.

2.2. Sample Preparation

The composites formulations (in parts per hundred parts of rubber (phr)) used in this work are listed in **Table 1**. The composites of nitrile rubber with different proportions of carbon black (0, 10, 20, 30, 40, 50, 60, 70 phr) and other additives was carried out in an internal mixer at 40˚C and a rotation speed of 60 rpm. An overall mixing time of 21 min ensures uniform dispersion of the carbon black filler particles in the elastomer matrix. This process was carried out according to the international standard ASTM D 3182. Homogeneous composites were pressed using German laboratory press type COLLIN under a pressure of 10 MPa and at a temperature of 100˚C for 10 minutes, taking into consideration that the temperature should not exceed 100˚C to avoid premature vulcanization of the rubber. Then the sample with dimensions (20 × 20 × 0.2 cm) was cooled with water under constant pressure to a temperature (25˚C).

Table 1. Proportions of ingredients of the rubber composites (wt%).

Function	Ingredients	Concentration (phr*)							
Rubber matrix	NBR	100	100	100	100	100	100	100	100
Processing aid	Stearic Acid	1	1	1	1	1	1	1	1
Activator	Zinc Oxide	3	3	3	3	3	3	3	3
Antioxidant	IPPD (4010 NA)	1	1	1	1	1	1	1	1
Plasticizers	DOP Oil	3	3	3	3	3	3	3	3
Reinforced filler	CB-N330	0	10	20	30	40	50	60	70

*parts per hundred resin/rubber.

2.3. Irradiation of Samples

The sheets were irradiated in air at room temperature, with different doses of gamma rays (0 - 50 - 100 - 150 - 200 - 250 - 300 KGy), at a dose rate (20.5 [KGy/h]), from a source using cobalt (Co60). The irradiation was carried out in the Department of Radiation Technology, Atomic Energy Authority, Syria.

2.4. Mechanical Measurements

2.4.1. Tensile Test

The tensile properties (tensile strength, elongation at break) were measured for the prepared overlaps according to the Dumbbell model, where five samples were cut for each tensile test, and they were tested using a Machine manufactured by (INSTRON) Model 1011 with a load (300 KN) and a speed (500 mm/min) and at room temperature, according to the standard specification (ISO-37 - Type 2).

2.4.2. Hardness Test

This test was carried out in accordance with the specification (DIN-53505) at room temperature, using a device (Digital Shore Hardness Tester) made by Zwick Company (Germany) using samples of dimensions (40 × 40 × 6 mm).

2.4.3. Abrasive Wear Test

A DIN abrasion testing was performed to determine the resistance of specimen to abrasion caused by friction, which is what seals in contact with moving parts are subjected to. The mass loss was determined by weighing the sample before and after the test. This test was conducted with strict compliance to testing standards (ISO 4649) by using a cylindrical sample with a diameter of 16 mm, as shown in **Figure 1**.

Figure 1. DIN abrasion test apparatus.

Volume loss was calculated according to the following formula:

$$\Delta V = \frac{\Delta m \times 200}{P \times Q}$$

(ΔV): The loss in volume (mm^3).

(ΔM): The weight loss is calculated from the difference in the weight of the sample before and after the test (g).

(P): The abrasive force resulting from the glass paper used (147 N).

(Q): Density of the studied sample.

200: constant related to the device.

The unit of abrasion resistance is typically expressed in mm^3. To convert the volume loss to mass loss, the density of the rubber sample is used. The mass loss is then converted to grams per cubic centimeter using the sample's density. This process allows for a more precise measurement of the material's abrasion resistance.

2.5. Swelling Test

This test was carried out according to ASTM-D471 by immersing the prepared samples with dimensions (2 × 25 × 50 mm) after being accurately weighed and taking their dry weight (W_d) in tightly closed glass containers in different solvents (diesel, gasoline and hydraulic oil) for a period of (100) days at room temperature, then raised and the excess oil was removed from the surface using a filter paper, then the samples were weighed and the weight was recorded (W_w), and the degree of swelling was calculated according to the following relationship:

Degree of swelling = [(Wet weight − Dry weight)/Dry weight] × 100%

$$\text{degree of swelling}(\%) = \frac{W_w - W_d}{W_d} \times 100$$

where: W_d: weight of the sample before immersion (g) - W_w: weight of the sample after immersion (g).

3. Results and Discussion

3.1. Mechanical Properties

3.1.1. Tensile Strength

The tensile strength is related to crosslinking density (CD), the ability of the chains to dissipate energy (DE), molecular imperfection (MI) and polymer chain length [18] [19].

Figure 2 shows the values of tensile strength (TS) as a function of the radiation dose, where the tensile strength of the composites increases with the increase of the radiation dose, due The increase in TS values may be due to crosslinking induced by irradiation, until it reaches a maximum value and then decreases y due to degradation process as well as oxidation processes of the rubber molecular chains (NBR) [20].

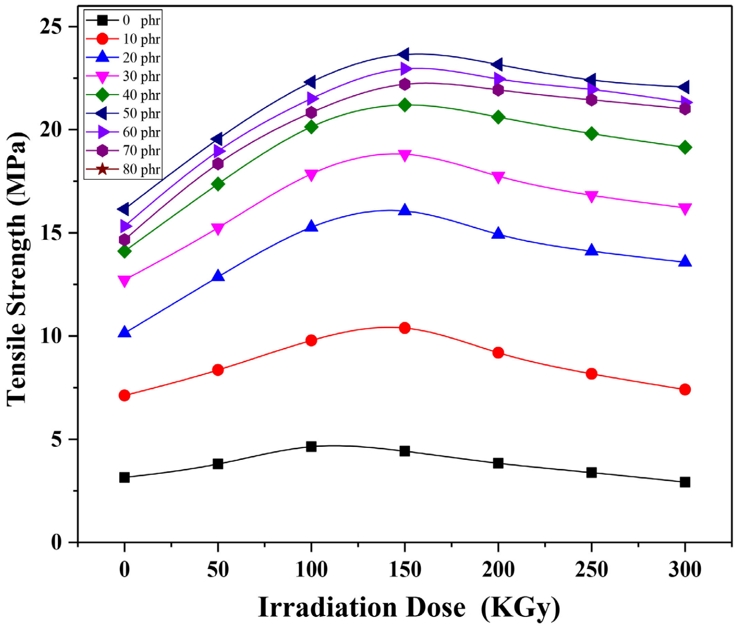

Figure 2. Tensile strength as the function of irradiation dose and carbon black content.

The tensile strength of the carbon black-free rubber sample reaches its maximum tensile strength at a dose of 50 kGy, and then decreases, and when loaded with (10 phr) of black carbon TS reaches its greatest value at the dose (100 KGy), then it decreases, and when loaded at rates higher than (10 phr), the values of TS increase with the increase of the radiation dose, reaching its maximum value at the dose (150 KGy) and then decreasing. the gradual decrease of tensile strength after reaching the maximum value and the reason for that the scission reactions of macromolecular chains dominate at high doses irradiation.

Figure 5 also shows that the tensile strength of the samples with the increase in carbon black loading at all radiation doses, and this indicates that the degree of strength (DR) due to the increased interaction between carbon particles and macromolecules of NBR. which indicates to the ability of carbon black particles to achieve a higher hardening of the structure [21]. In addition, carbon black particles fill the spaces between the rubber macromolecules, which leads to a reduction in the free volume giving more rigidity to the rubber structure [22]. The number of scission reactions affecting polymeric chains at high doses above 150 [KGy].

At higher carbon black loadings, the carbon particles tend to aggregate, in the rubber matrix, which weakens the bonding between the chains and leads to a decrease in tensile strength. The presence of different groups on the surface of the carbon particles such as carboxylic, phenolic, hydroxyl, aldehyde and ketone groups will contribute to the formation of physical and chemical bonds on the contact surfaces between rubber and carbon black particles [23].

3.1.2. Elongation at Break

It can be observed from Figure 3 that the elongation at break increases with the increase of radiation dose up to 50 kGy. At low doses of radiation, the side branches of the rubber chains, in addition to non-crosslinked macromolecules that are highly entangled with each other, can partially stretch under such circumstances [24].

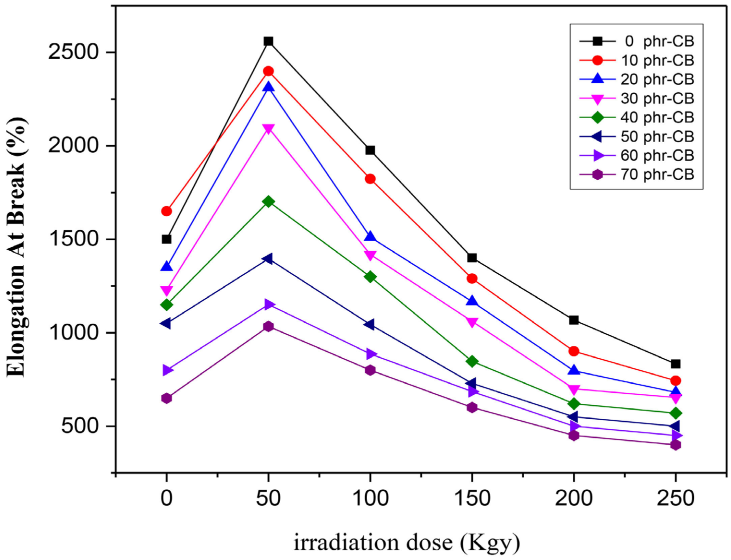

Figure 3. Elongation at break as the function of irradiation dose and carbon black content.

The elongation at break then decreases gradually with the subsequent increase in irradiation dose. This is due to the increase in the density of crosslinking between the rubber chains, it becomes high enough to restrict reorientation process that leading to elongation.

These crosslinks also lead to a decrease in the mobility of the molecular chains, an increase in the number of chain scission reactions under high-dose irradiation also leads to a higher rate reduction in elongation at break, as the structure of the composites becomes stiffer, more brittleness and less flexible [25].

The increased loading with black carbon reduces the mobility of rubber macromolecules due to the formation of physical bonding between the filler particles and the polymer chains, which in turn leads to the stiffening of the matrix [26]. It is noted that the elongation at break decreases at with the increase in the loading rate of the filler due to the hardening of the structure.

In fact, the molecular mobility decreases with increase in reinforcing filler loading, leads to formation of physical bonds between incorporated fillers and the rubber.

3.1.3. Hardness Test

Figure 4 shows the hardness values as a function of the radiation dose, it is noticeable that the hardness value gradually increases with the increase in the radiation dose. As the composites absorb the γ ray energy a large number of free radicals are generated on the rubber chains, which in turn are linked to form (C-C) bonds, which leads to an increase in the average molecular weight [27]. Thus, it leads to an increase in the hardness.

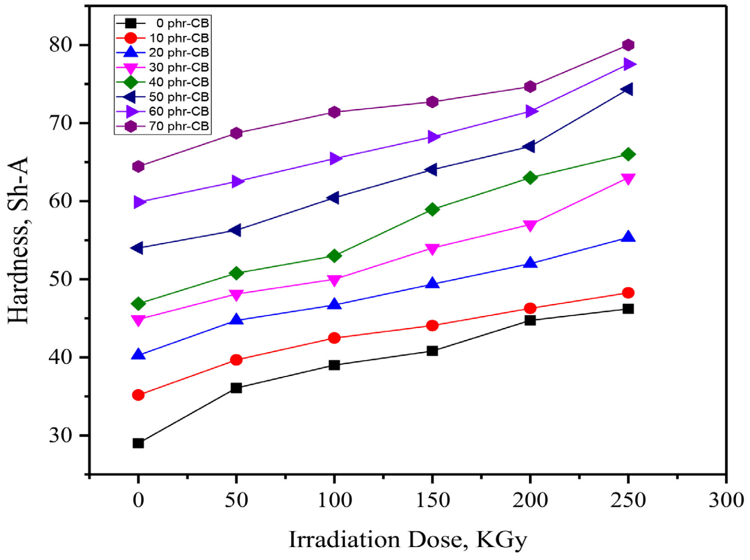

Figure 4. Hardness as the function of irradiation dose and carbon black content.

Increasing the loading of composites with carbon black also leads to a significant increase in hardness. This is due to physical bonding and cohesion strength of black carbon with the rubber macromolecules which lead to increases strengthening effect of rubber composites [28].

3.1.4. Abrasive Wear

Friction and abrasive wear are phenomena of surface–interface interactions that are closely related to the environmental conditions in which materials and components are used. The design of materials and components should take into consideration the specific operating environment, so as to reduce the energy dissipation and component damage caused by surface corrosion and interfacial friction between interacting materials [29].

Movable rubber components are subjected to harsh friction conditions during their service life. This leads to massive economic losses. Figure 5 shows the results of the mass loss caused by friction for the rubber samples loaded with (50 phr) carbon black at different radiation doses.

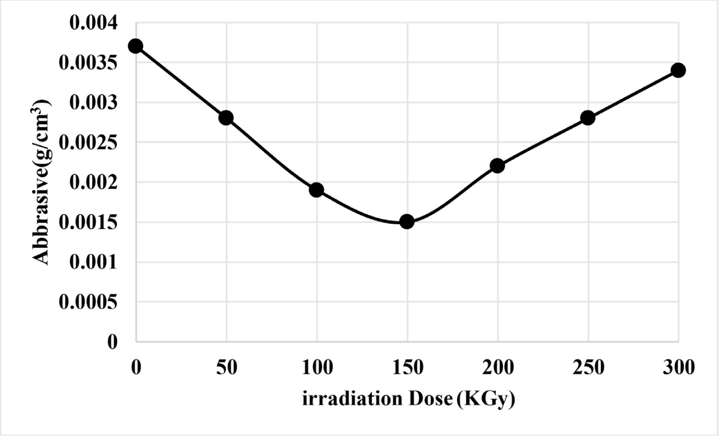

Figure 5. Effect of radiation dose on the abrasive wear caused by friction expressed as weight loss of (NBR) loaded with 50Wt% carbon black.

It can be observed from the figure that the abrasive mass loss decreases with increasing radiation dose. This is due to radiation-induced vulcanization, which leads to for three dimensional network and improve homogeneous distribution of the reinforcing material within the rubber matrix. The increase in the mass loss caused by friction is due to the increased brittleness of rubber composites as the dose irradiation increases above (150 KGy). This leads to the formation of defects and cracks within the rubber structure caused by the unreacted carbon aggregates with the matrix of rubber composites. High-dose irradiation also leads to the dominance of chain scission reactions of macromolecules of nitrile rubber and induces irreversible oxidation reactions of the rubber macromolecules that result in the deterioration of the mechanical properties of the rubber composites.

3.2. Swelling Test

Rubber components have been widely used in automotive industry, especially in applications that are in direct contact with oils and petroleum derivatives. It expresses the resistance of rubber composites to petroleum derivatives or solvents in terms of their swelling ratios. The results are presented in Figures 6-8, which show a decrease in swelling values with increasing radiation dose. This decrease is mainly due to radiation-induced crosslinking of the molecular chains of NBR rubber, [30] as an increase in crosslink density leads to the formation of a three-dimensional polymer network that reduces the permeability and diffusion of oil within the rubber matrix [31].

It is noted that the lowest turbulence rate was recorded in hydraulic oil at all radiation doses and black carbon loading rates, due to the fact that hydraulic oil is less polar than gasoline and diesel [32].

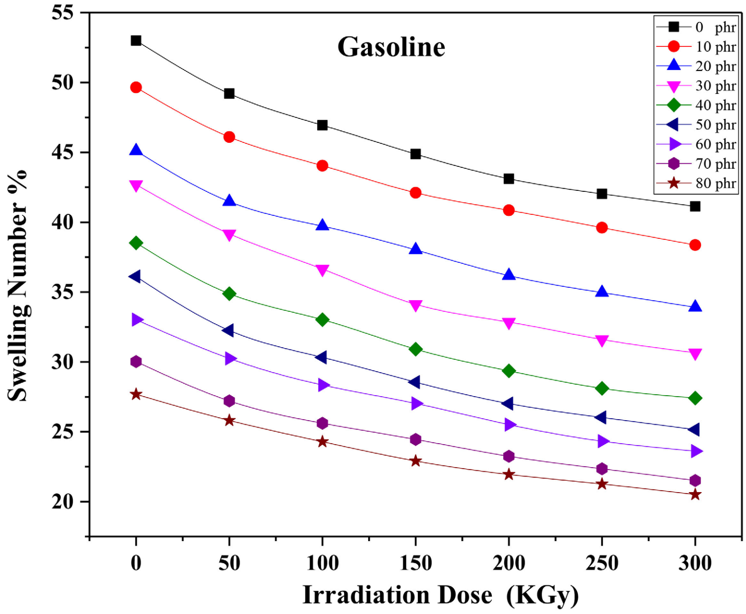

Figure 6. Swelling of samples irradiated different radiation doses in Gasoline at room temperature.

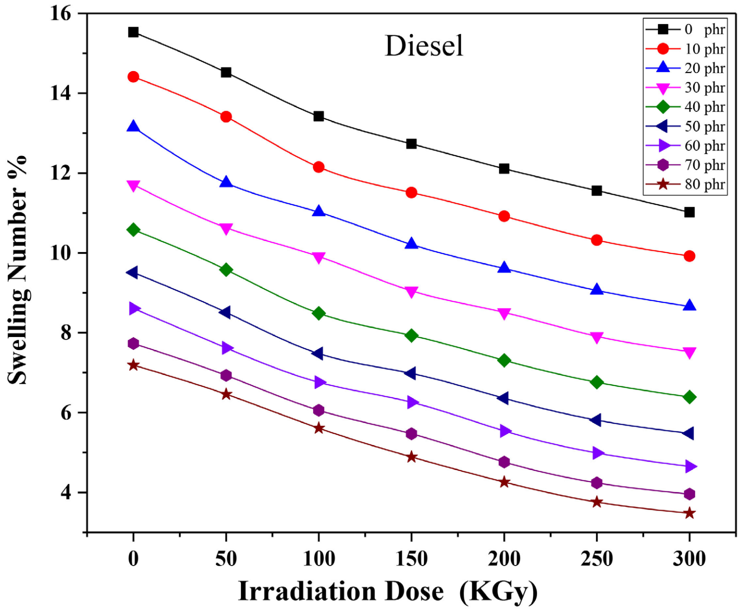

Figure 7. Swelling of samples irradiated different radiation doses in diesel at room temperature.

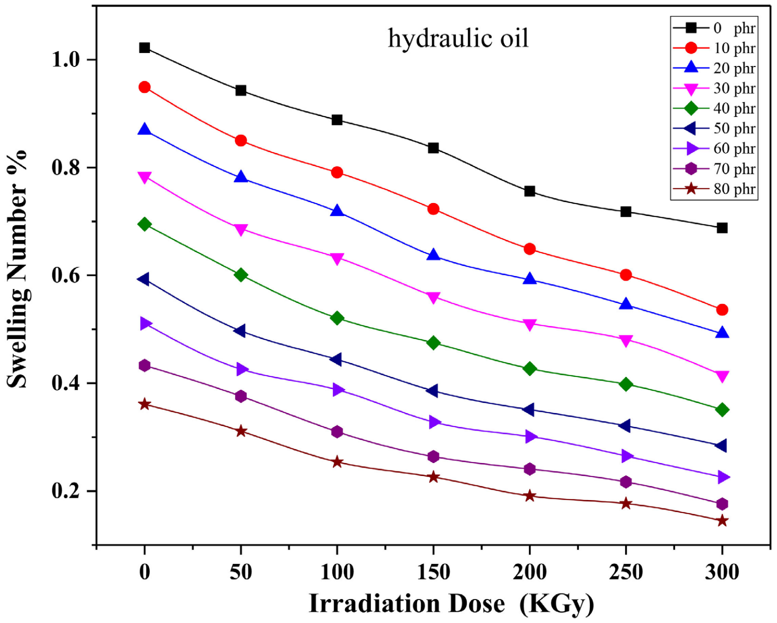

Figure 8. Swelling of samples irradiated different radiation doses in hydraulic oil at room temperature.

The addition of black carbon particles in the spaces between the rubber chains also causes a reduction in the free volume between the rubber molecular chains, which limits the possibility of penetration and diffusion of oil molecules within the rubber structure [33]. Carbon black particles also tend to form aggregates, that prevent the solvent from penetrating into the RM, and the increase in the number of physical and chemical bonds emerging between black carbon and macromolecules significantly reduces the degree of agglomeration [34].

4. Conclusions

In this research, the effect of radiation dose and carbon black (N330) loading on the properties of Nitrile Butadiene Rubber (NBR) was studied, by preparing composites of NBR containing different proportions of carbon black, and then conducting radiation vulcanization by exposing them to different doses of gamma rays up to 300 KGy.

The mechanical properties and resistance composites to some Petroleum derivatives were studied.

Results revealed that the tensile strength increased at all load percentages of carbon black with an increase in the radiation dose, then decreased with irradiation at high doses, while the elongation at break for the composites exhibited the opposite behavior. It was also found that the resistance of rubber composites to certain types of petroleum oils increases with an increase in the radiation dose, and that the composites are most resistant to engine oil, followed by gasoline and then hydraulic oil.

The high mechanical properties of the composites and their high resistance to petroleum oils make them the preferred choice for use in applications where they are in direct contact with these oils, such as seals, engine gaskets, and O-rings.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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