

Thermo-mechanical Properties of Polyvinyl Chloride/vulcanized Nitrile Butadiene Rubber Blends Cured by Gamma Irradiation

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COMPATIBLE blends of polyvinyl chloride/vulcanized nitrile butadiene rubber, PVC/NBR, can be readily prepared based on the common polymer polar characters. However, the enhancement of PVC contributes to the aging resistance of NBR and the fact that NBR can act as a permanent plasticizer for PVC deserve progressive evaluation of their composites. Compounding of polyvinyl chloride (PVC)/nitrile butadiene rubber (NBR) at various ratios were produced by melt mixing. Neat PVC and PVC/NBR formulations were then gamma irradiated with the integral doses of 25, 50, 100 and 150kGy. It has been indicated, that radiation- induced microstructural changes were imparted to the pristine and blend formulations. Developed unirradiated and irradiated blends were characterized by various physical, mechanical and thermal investigations: In toluene swelling and, motor and brake oils resistance, tensile strength, elastic modulus and elongation at break and, the thermogravimetric and differential scanning calorimetry analyses. The examinations described distinctive improvement in the blend respective behaviors correlated with the rubbery feed ratio and radiation dose.

Keywords: Thermo-mechanical properties, Oil resistance, Gamma irradiation.

Introduction

Modern technology thrusts challenging depends on the performance capabilities of polymers. A new approach to the science and technology of polymer blends has emerged recently, i.e., polymer blends by design rather than by availability. Acrylonitrile butadiene rubber (NBR) is a well-known unsaturated copolymer for about five decades, a simply processable, tough, and flexible rubber. Therefore, it has been used in many industrial purposes such as hoses, O-ring seals, insulation base products and other many packaging materials (Ahmed, 2015). However, its use in automotive applications is interesting, but the aging resistance is limited because of the unsaturated backbone of the butadiene (Hassan et al., 2012).

Technically related NBR is composed of 24-30 wt% of acrylonitrile which provides some benefits such as, good processability, resistance to oils and hydrocarbons. NBR has also a wide range of service temperature (from -35°C up to 100°C) (Kalaf et al., 2012 and El-Nemr, 2011). It is well known that NBR is a crosslinking

type polymer; hence, a high-energy radiation improves its stability and mechanical properties via establishing the desired crosslink density. Nevertheless, at higher levels of radiation dose, the mechanical properties are adversely affected due to the radiation- induced degradation.

Among thermoplastics, PVC presents a variety of options; its polar nature allows preparation of compatible blends with complementary dissimilar polymers. The poor stability of PVC against heat and some kinds of radiation is attributed to structural defects formed during polymerization. These defects are mainly due to allylic and tertiary chlorine atoms, known as labile chlorine atoms. These labile atoms are extremely unstable and easily detached by effect of heat or even sunlight to evolve HCl gas, leading to the formation of the polyenes and hence, to a high degree of discoloration (de Silva et al., 2008).

PVC is a polymer widely used for radio-sterilisable food packaging and medical devices. However, when the polymer systems are submitted to sterilization by gamma radiation (25kGy dose), their molecular structure undergoes sharp

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DOI: 10.21608/ejrsa.2018.1815.1023

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modification in the physical properties mainly as a result of main chain scission and crosslinking effects (Vinhas et al., 2004). For PVC both processes coexist and either may be predominant depending not only upon the chemical structure of the polymer, but also upon the surrounding conditions; temperature, environment, dose rate, etc..., under which irradiation is performed.

It is possible to get new materials with improved properties by blending elastomers and plastics. If the elastomer is the major phase and the plastic is the minor one, this blend will have the properties of the reinforced elastomer. However, if the plastic is the major phase, then the obtained blend will be a toughened plastic material. One of the most common blends in modern science is polyvinyl chloride (PVC) with acrylonitrile butadiene rubber (NBR) (Ahmed, 2015). The incorporation generally improves the physico-mechanical and chemical properties. NBR/PVC blends can be conventionally milled, extruded, or compression molded using traditional processing equipment for natural and synthetic rubbers (Mazumdar et al., 1983). Due to the miscible nature of NBR/PVC blend, the soft blends of NBR/PVC can be categorized as a thermoplastic elastomer (TPE) and, more specifically, as a melt processable rubber (Hassan et al., 2012).

The possibility of radiation processing of the polymeric material in the solid state opens up new opportunities to obtain materials with well-tailored properties. Implementation of radiation in the processing of polymers is an alternative to the traditional chemical methods, mostly environmentally non-friendly, to modify the molecular structure of polymers. Several studies on gamma radiation-induced modifications of polymers have been reported (Ibrahim et al., 2015; Hassan et al., 2013 a, b and El-Nemr et al., 2015). It is well known that the main affect of the interactions between gamma rays and polymers is the formation of free radicals, whose further evolution can cause crosslinking, with an increase in the molecular weight, or scission with a decrease in the molecular weight and chain branching. Irradiation induces the formation of free radicals which lead to more C–C bonds within the network where more energy is dissipated in the stretching process (Ibrahim et al., 2015).

The present work aimed to elaborate on the produced polyvinyl chloride/vulcanized nitrile

butadiene rubber blend composition and radiation-induced physical alteration and oil resistance with respect to neat PVC. Some comparative thermo-mechanical properties were also investigated. Explanatory suggestions for the yielded characteristics were delivered.

Experimental

Materials

The employed chemicals are as follows: Polyvinyl chloride (PVC) was obtained from Sidi Kerir Petrochemical Company (SIDPEC), Egypt, whereas Acrylonitrile butadiene rubber (NBR), European N3345, acrylonitrile content 34% of Mooney viscosity (ML [1+4] at 100°C) from Enichem Co., Italy; Zinc oxide from Shijiazhuang Golden Color Chemical Co., China; Stearic acid 99.7%, mp. 56°C from Hebei Liancheng Chemical Co., China; Mercaptobenzothiazyl disulfide from Sulfur Standard Chemical Company, India; Tetra methyl thiuram disulfide from Bayer India, India; 1,2-dihydro-2,2,4-trimethyl quinoline from NOCIL, India.

Preparation of rubber vulcanizate

NBR was mixed on a rubber mill (300mm x 470mm) with a gear ratio of 1.14:1 at room temperature with 1phr (parts by weight per hundred parts of rubber) of 1,2-dihydro-2,2,4-trimethyl quinoline, 5phr of zincoxide, 1phr of mercapto benzothiazyl disulfide, 1phr of tetra methyl thiuram disulfide and 1phr of sulfur as curing agent.

Blending process

PVC/NBR blends of various compositions (70/30, 50/50, 30/70) were melted in Brabender Plastic order PL2100 Mixer with a volume capacity of about 200cm³ at 150°C, 60rpm for 10min, where PVC was first inserted into the mixer for about 2min. There after, NBR vulcanizate was introduced to mix with the molten PVC until uniform dispersion.

Gamma irradiation treatment

Irradiation was carried out at the National Center for Radiation Research and Technology, Atomic Energy Authority, Cairo, Egypt. The prepared blends of PVC/NBR of various ratios were subjected to gamma radiation (gamma cell type 4000 A, India) in air, at ambient humidity and temperature with different radiation doses, namely 25, 50, 100, 150kGy at a dose rate of 2.8kGy/h.

Measurements

Physical parameters: Swelling and oil resistance

For determining the swelling capacity of the plastic/rubber blend, three pieces of each sample of approximately uniform size and weight ($\approx 0.5\text{g}$) were accurately weighed (W_1) and immersed in 50ml toluene at room temperature for 24h. There after, the samples were taken out and put between two pieces of filter paper, then put between two sheets of glass, each weighted 98.4g, kept for 5sec, then transferred to a weighting bottle and reweighted (W_2).

The swelling percentage (Q) was calculated by Eq. (1):

$$Q\% = (W_2 - W_1 / W_1) \times 100 \quad (1)$$

The same sequence was carried out for the oil resistance measurements by immersing the samples in motor and brake oils for three days.

Mechanical properties

The mechanical properties: Tensile strength, tensile modulus and elongation at break were tested at room temperature using Mecmesim, Model 10-I, UK, at a crosshead speed of 500mm/min. The mechanical investigations were examined in triplets.

Thermal characteristics

Thermogravimetric analysis (TGA):

Thermogravimetric analysis (TGA) was performed on a Shimadzu TGA-50 System, Japan, by heating within the temperature range 20–600°C at a heating rate of 10°C/min under a controlled dry nitrogen flow of 20ml/min.

Decomposition activation energy

The kinetic parameters, at a heating rate of 20°C/min, served for calculating the activation energy of the prepared blend and composite using one integral step, i.e. one heating run within the temperature range 20- 600°C. The decomposition conversion X, can be calculated by Kissinger equation:

$$X = W_0 - W_t / W_0 - W_f \quad (2)$$

where, W_0 is the original mass of the test sample; W_t is the mass at time t and W_f is the final mass at the end of decomposition. Thus, decomposition reaction may be simply expressed by the

following formula:

$$dX/dt = A \exp(-E_a/Rq)(1-X) \quad (3)$$

where, A is the pre-exponential factor, E_a is activation energy, q is heat energy and t is time. Rearranging Eq. (3) followed by integration gives:

$$\ln[-\ln(1-X)/T^2] = \ln [AR/H E_a (1-2RT/E_a)] - E_a/RT \quad (4)$$

The expression $\ln [AR/HE (1-2RT/E_a)]$ is essentially constant. If the left side of Eq. (4) is plotted versus $1/T$, a straight line may be obtained if the process is a first order reaction. From the slope, $-E_a/R$ the activation energy E_a can be determined.

Differential scanning calorimetry (DSC)

The thermal properties of all blends were investigated by employing the calorimeter system Shimadzu Type DSC-50 under constant operating conditions of 20ml/min within the temperature range from ambient to 200°C at a heating rate of 10°C/min.

Result and Discussion

Swelling properties

The swelling characteristics of the PVC/NBR blends in toluene are shown in Fig. 1. The percentage of toluene uptake decreased by the addition of plastic phase into the rubbery moiety as PVC particles fill into the intermolecular gaps amongst NBR chains (Abu-Abdeen & Elamer, 2010). Figure 1 also demonstrates the impact of the radiation dose on the PVC/NBR blend. Obviously, the decrease in swelling by irradiation indicates the occurrence of the radiation-induced crosslinking with in the blend. Apparently, in absence of thermoplast, the abrupt decrease in swelling is strongly noticeable up to 25kGy. A similar reduction in swelling was attained by incorporating 30% PVC. This may suggest the progressive reduction in chain mobility as a result of a radiation-induced growing three-dimensional network structure of the rubber chain (Chantara et al., 2011) and the intermolecular intrusion of the plastic particles.

Oil resistance

The nature of the implemented oils and the crosslink density of the polymer are the main parameters controlling the degree of swelling.

Typically, due to the intrinsic higher viscosity of brake oil, the reported swelling results revealed a stronger oil resistance behavior than that of motor oil. It is also well known that NBR demonstrates oil resistance; therefore NBR remains an important material in the formulation of engineering rubber blends. Table 1 illustrates the effect of the two applied types of oil on the swelling percentage of the unirradiated and irradiated blends, at different ratios immersed for three days at $\approx 25^\circ\text{C}$. The blend swells most in both oils as the plastic phase increases while the trend fades out as the NBR ratio increases within the matrix, unlike the previously mentioned behavior in toluene solvent. NBR incremental incorporation limits the

penetration of oils into the radiation-cured blends. The utmost recorded value of oil resistance was reported as NBR ratio reached 70% in both oils.

Meanwhile, a remarkable improvement in oil resistance with dose was explicitly observed. The phenomenon can be explained by the fact that the increase in the degree of radiation-induced network structure leads to a further compactness in rubber moiety. The 50/50 sample cured with 150kGy recorded the highest oil resistance where swelling % comparatively lowered down to nearly two thirds the value reported for the unirradiated sample. The penetration of both oils into blend formulation remarkably decreased to 100kGy.

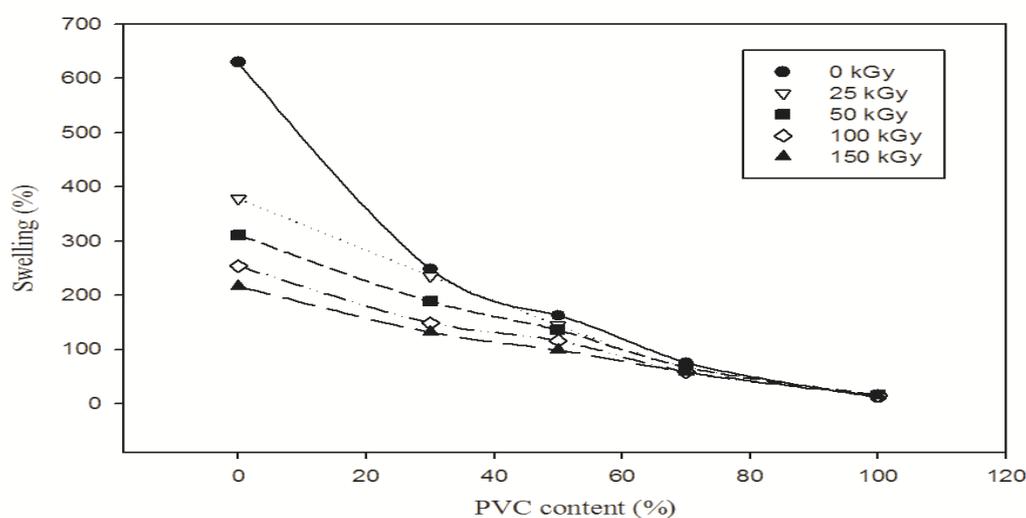


Fig. 1. Swelling (%) in toluene of PVC/NBR blend at various gamma radiation doses.

TABLE 1. Oil swelling percentage of the unirradiated and irradiated PVC/NBR blends in motor and brake oils (3 days at room temperature $\approx 25^\circ\text{C}$).

PVC/ NBR wt%	Unirradiated blend		Radiation dose (kGy)	Irradiated blend		
	Motor oil (%)	Brake oil (%)		Blend composition wt%	Motor oil (%)	Brake oil (%)
PVC	28.6	8.5	0		23.2	7.1
70 PVC/30 NBR	24.6	6.5	25		20.2	5.8
50 PVC/50 NBR	23.2	5.1	50	PVC/NBR, 50/50	18.3	4.6
30 PVC/70 NBR	21.3	5.5	100		16.8	3.8
NBR	19.4	4.3	150		16.1	3.4

Mechanical measurements

In accordance with the swelling investigation outcomes, blend mechanical parameters generally enhance by the inclusion of the thermoplastic matrix into the rubber moiety (Abu-Abdeen & Elamer, 2010) and ionizing irradiation (Chantara et al., 2011). The applied load of PVC can be transferred across the strengthened interface, resulting in a higher tensile strength. Figure 2 displays the influence of blend ratio and radiation dose on the tensile parameter of the PVC/NBR formulation. It can be clearly observed that the tensile strength value of the unirradiated sample gradually increases by the PVC content from 3.5MPa to 9.5MPa at 70phr loading, beyond which it shows a steadiness. Where by, a consistent systemic increase in tensile strength was remarked over the cited radiation dose range up to 100kGy yielding ~14MPa.

It is known that higher contents of PVC assimilating into the NBR matrix lead to improvement in toughness whereas the elastomer phase remains as dispersed particles (Ahmed, 2015). Furthermore, it has been reported that the compounding of PVC in NBR rubber leads to increasing the modulus at 100% elongation, tensile strength, hardness and strain energy (Gheno et al., 2009). The variation in 100% modulus with the PVC feed ratio and radiation dose is shown in Fig. 3. The figure illustrates, that the rigidity of rubber vulcanizates is generally enhanced due to the inclusion of PVC particles, exhibiting distinguished improvement with a plastic level higher than 70%, with relatively limited radiation influence. This can be attributed to the creation of physical crosslinks on one hand and the presence of PVC domains (hard segments) in the rubber

matrix on the other (Hassan et al., 2012).

Figures 2 and 3 exhibit alike impact trend of gamma irradiation on both blend parameters: Tensile strength and 100% modulus. The recorded values for both parameters steadily increased by curing the blend with radiation up to 100kGy, beyond which observed reduction in the values were determined. The former results can be ascribed to the growing crosslink density within the matrix. Whereas the consequent inclining values may be either accounted for the predominance of chain scission mechanism or the embrittlement of the blend due to the establishment of a high extent of crosslinking (Chantara et al., 2011).

Variation in elongation at break % of PVC/NBR blend, as a function of PVC loading, is described in Fig. 4. The trend of excessive increase in elongation % up to 30phr, is significant, allowing the rubber gum to undergo strain-induced crystallization to that level of thermoplast incorporation (Khalid et al., 2010). Nevertheless, a further increase in PVC content led to a considerable reduction in elasticity. Figure 4 also displays the systemic decrease in elongation % with radiation dose. This may be accounted for the increase in the degree of radiation-induced crosslinking with dose and the yielded network structure that eventually leads to stiffness in the polymer matrix (Wang et al., 2009). Furthermore, under the applied dose level and PVC ratio, the progressive hindrance in the macromolecular chain mobility may add to the apparent reduction in elongation. Radiation-induced degradation process that takes place mainly within PVC moiety may also contribute to this phenomenon.

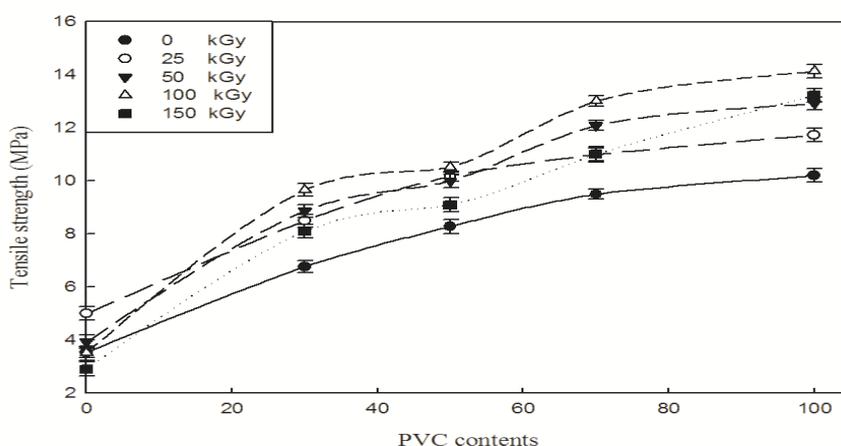


Fig. 2. Tensile strength (MPa) of PVC/NBR blend at various gamma radiation doses.

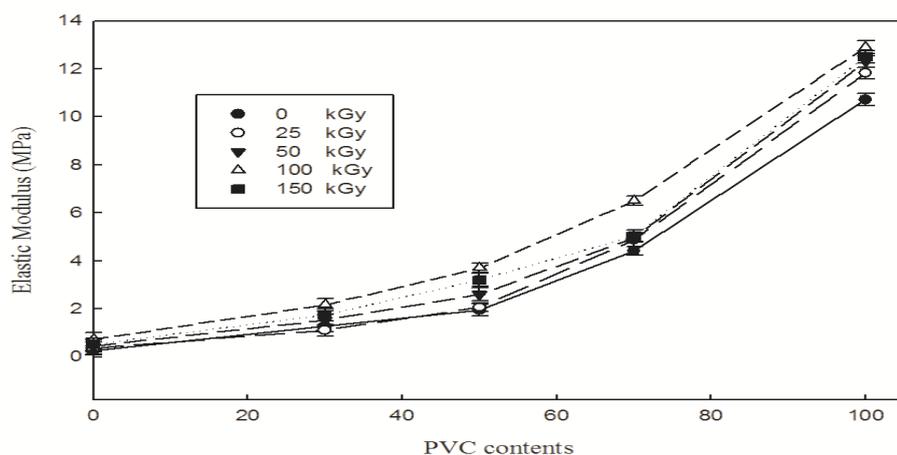


Fig. 3. Elastic modulus (MPa) of PVC/NBR blend at various gamma radiation doses.

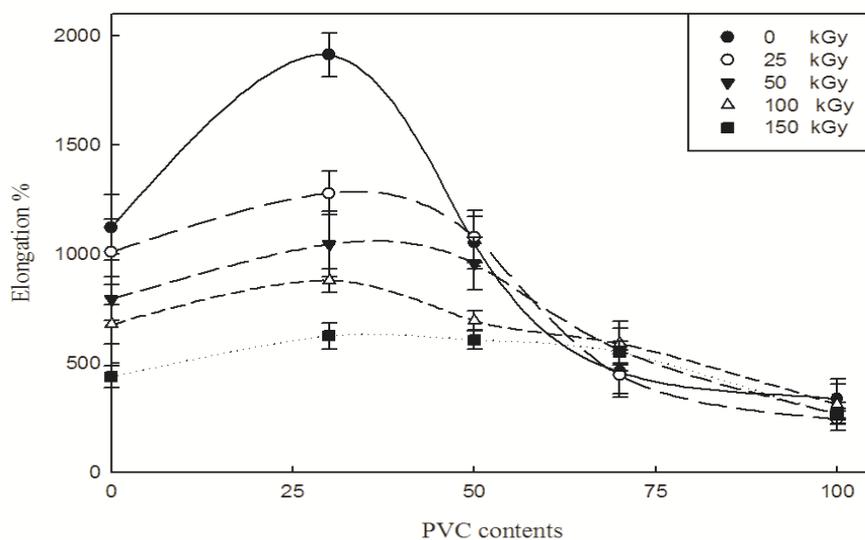


Fig. 4. Elongation (%) for PVC/NBR blend at various gamma radiation doses.

Thermal stability

Thermogravimetric analysis

Thermogravimetric analysis (TGA) is a standard procedure in which changes in the mass of a specimen is monitored as the specimen is progressively heated. The TGA thermograms for neat PVC and PVC/NBR, 50/50 wt%, at 10°C/min heating rate in nitrogen environment, are given in Fig. 5 and summarized in Table 2. A similar to neat PVC, the PVC/NBR blend revealed two-stage degradation: The first stage can be attributed to the volatilization of hydrogen chloride molecules followed by the formation of the conjugated polyene sequences within

the temperature range 260°C to 360°C, whereas the second stage corresponds to the thermal cracking of the carbonaceous conjugated polyene sequences within the temperature range 400°C to 500°C.

The thermal stability is given by the initial decomposition temperature, T_{onset} corresponding to $\approx 10\%$ losses taken as a measure for the onset degradation, and the values are listed in Table 2. Degradation rapidly takes place beyond $T_{0.5}$, the temperature at which 50% loss occurs, whereas the residual weight is reported at 600°C. The 50 wt% NBR load improved the thermal stability

of the neat PVC as indicated by the elevation in the onset degradation temperature from 218°C to 243°C. In addition, the increment in $T_{0.5}$ of the blend emphasizes the role of NBR in constructing the blend skeleton, hence improving thermal stability. This may be argued on the basis that NBR acts as an insulator via mass transport barrier against HCl evolution, leading to slower decomposition in PVC. These observations prove one of the commercially important and miscible polymer blends, PVC/NBR (Gheno et al., 2009).

The TGA thermograms of the initial and irradiated blend PVC/NBR, 50/50 wt%, are shown in Fig. 6. As seen in Table 2, the utmost thermal stability was attained by irradiation at 100kGy as illustrated by $T_{0.5}$. Systemic improvement in thermal stability with dose can be accounted for the radiation-induced intermolecular crosslinking

additional barrier against HCl evolution, supporting the earlier observation of the significant increase in mechanical parameters by irradiation.

Decomposition activation energy

The Kissinger method at a fix conversion was employed for determining the kinetic parameters of neat PVC and its evenly formulated blend with NBR are presented in Table 3. Obviously, the activation energy (E_a) of the unirradiated blend PVC/NBR, 50/50wt%, is fairly higher than that of the plastic component, confirming the blend better thermal stability. Nevertheless, the activation energy of the irradiated composite revealed nearly one degree of order decrease with dose. This may be ascribed to the relative concurrent radiation-induced degradation process, occurs mainly within the thermoplastic moiety.

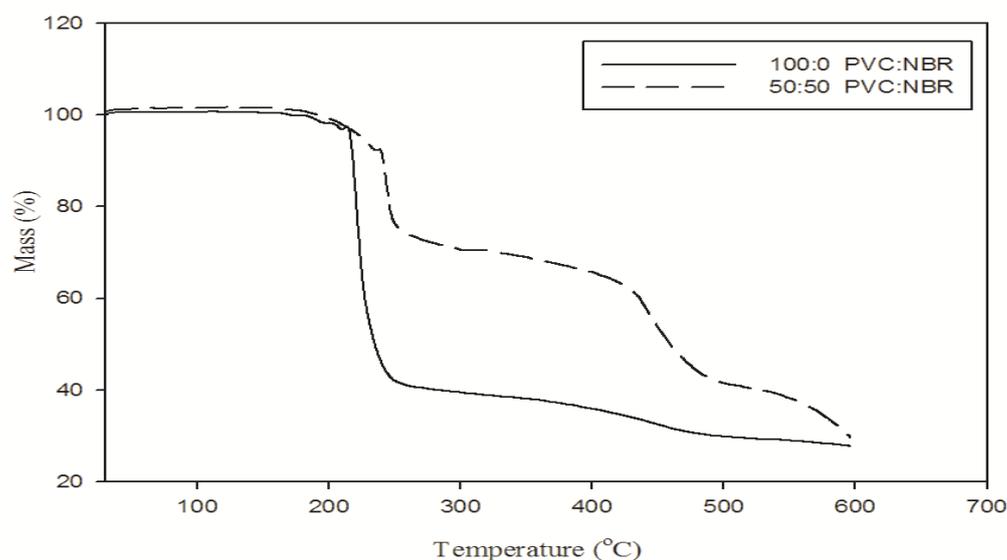


Fig. 5. TGA thermograms of PVC and PVC/NBR blend.

TABLE 2. Decomposition temperatures of neat PVC and its blend with NBR, 50/50wt%, at various radiation doses.

Blend composition	Dose (kGy)	T_{onset} (°C)	$T_{0.25}$ (°C)	$T_{0.5}$ (°C)	Charred residue at 600°C (mass%)
PVC	0	218.0	224.0	236.0	27.8
	0	243.0	254.0	461.0	29.7
PVC/NBR, 50/50wt%	25	242.0	272.0	458.0	33.3
	50	241.0	282.7	460.0	30.0
	100	233.0	309.0	462.0	33.6

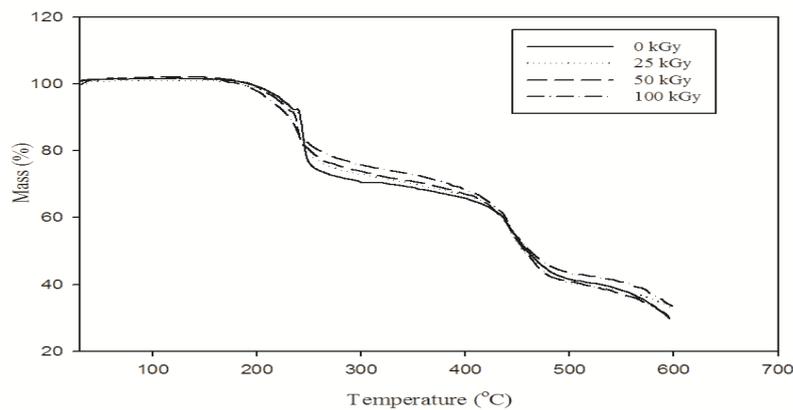


Fig. 6. TGA thermograms of irradiated PVC and PVC/NBR blend.

TABLE 3. Activation energy of neat PVC and its blend with NBR, 50/50wt%, at various radiation doses.

Sample Composition wt%	Dose (kGy)	Activation Energy E_a (kJ/mol)
PVC/NBR 100/0	0	7.42
	0	7.79
PVC/NBR 50/50	25	6.71
	50	6.15
	100	6.12

Differential scanning calorimetry

DSC scans were used to evaluate the effect of blend formulation and gamma-rays exposure on melting enthalpy. Thermal stability of PVC in blends is influenced by the compatibility of the polymer at the degradation temperature of the system, which can be investigated by measuring the melting enthalpy (T_m). Figure 7 and Table 4 show T_m values of the neat PVC and its blend with NBR, 50/50wt%. From the thermograms, pristine PVC reveals a melting temperature of $\approx 212.5^\circ\text{C}$, whereas its blend with NBR shifts to a significantly higher temperature value of $\approx 245^\circ\text{C}$. The experimental observations reflected three major suggestions: 1- A significant enhancement in melting temperature reveals that some microstructural changes have been imparted to the blend. In addition, the improvement in melting enthalpy enhances the ability of the materials to resist external deformation and decomposition under the effect of heat. Interestingly, this will eventually lead to better physical and chemical properties of the polymer matrix possessing special characteristics referred to as thermoplastic elastomer TPE_s. These results can safely be explained by interactions taking place between the PVC chains and the nitrile groups in the blend moiety. 2- A relatively higher presence of NBR may

act as a stabilizer towards the dehydrochlorination of PVC. A possible reason for this behavior can be again referred to the diffusion of HCl as it may be retarded due to interaction of the evolved HCl with polar nitrile groups. 3- Since incorporation of NBR remarkably impacts the crystalline phase in the matrix, as indicated by the increments in T_m , and provided that PVC chains located at crystallite surfaces are less ordered, therefore compounding PVC with NBR affords a desired status of compatibility.

Figure 8 DSC scans were also used to evaluate the effect of gamma-rays exposure on transition temperature (T_m). The reduction in T_m was explicitly noticed with dose. A lower T_m with radiation dose reflects a degree of scission in the tie molecules. The phenomenon may also suggest chain folds at the crystal surfaces of PVC molecules with a higher surface free energy. This may lead to the formation of shorter macromolecular PVC chains with a lower molecular weight beside other modifications in the crystal domains (Hassan, 2008). The discrepancy in crystalline population can be recognized to be at the expense of the blend compatibility, which in turn, may explain the lower onset temperatures as demonstrated by TGA, results shown in Fig. 5 and Table 2.

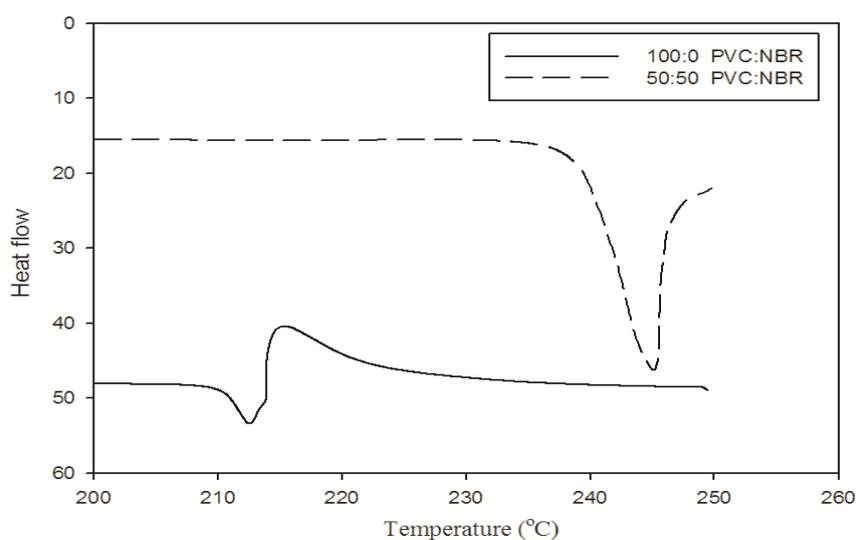


Fig. 7. DSC thermograms of PVC and PVC/NBR blend.

TABLE 4. Melting temperature of neat PVC and its blend with NBR, 50/50wt%, at various radiation doses.

Blend composition	Dose (kGy)	T_m (°C)
PVC	0	212.5
	0	245.0
PVC/NBR, 50/50wt%	25	244.3
	50	243.0
	100	238.6

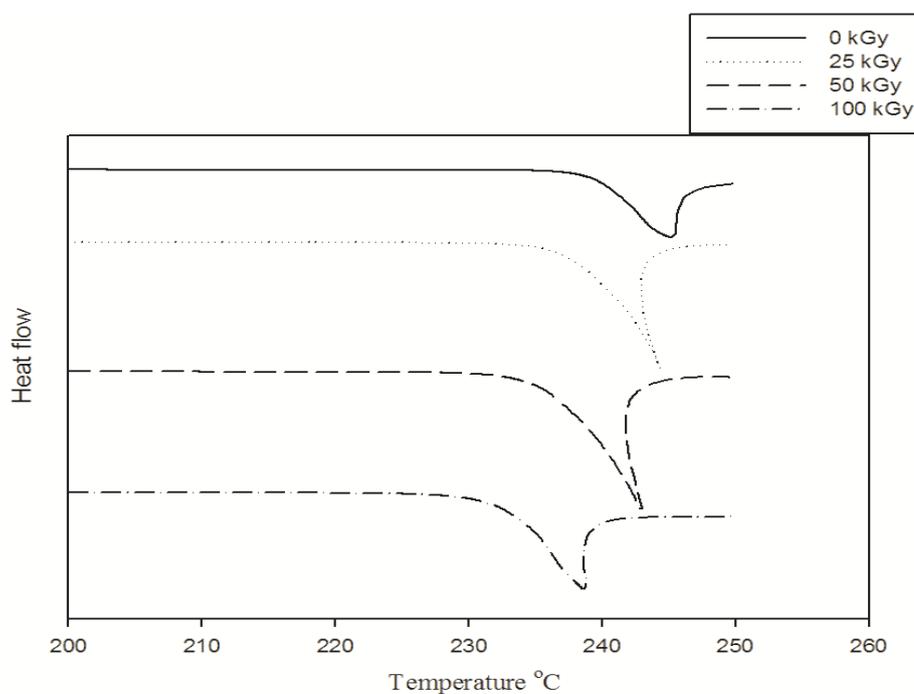


Fig. 8. DSC thermograms of irradiated PVC and PVC/NBR blend.

Conclusions

This article evaluates the oil resistance, thermo-mechanical and physical properties of polyvinyl chloride/vulcanized nitrile butadiene rubber blends cured by gamma irradiation. The penetration of brake and motor oils into the blend formulation decreased rapidly by irradiation up to 100kGy. The reported swelling results in brake oil comparatively revealed a stronger oil resistance behavior than that of motor oil due to the former higher viscosity parameter. The toughness of rubber vulcanizates has been generally increased due to the assimilation of PVC with in the NBR moiety. The trend of excessive increase in elongation % of the blend with PVC loading up to 30phr was noticeable.

The 50wt% NBR load in the blend improved the thermal stability of the neat PVC as indicated by the elevation in the onset temperature of degradation. Comparatively, a remarkable enhancement in the thermal stability was reported for the irradiated PVC/NBR, 50/50wt%, blend at 100kGy dose, as indicated by the $T_{0.25}$ decomposition. The improvement in the thermal stability supports the reported mechanical results, emphasizing significant increases in crosslink density by irradiation. Pristine PVC has a melting temperature of $\approx 212.5^{\circ}\text{C}$, whilst its unirradiated blend with NBR, 50/50wt%, shifts it to a higher value $\approx 245^{\circ}\text{C}$. The significant elevation reveals that some microstructural changes occur within the blend matrix.

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- (Received 10/10/2017; accepted 7 / 1 /2018)

الخصائص الحرارية الميكانيكية لمتراكبات البولي كلوريد الفينيل / مطاط النيتريل بيوتادين المعالجة بأشعة جاما

السيد فتحى السيد، منى يوسف النجار و مدحت محمد حسن
قسم كيمياء البوليمرات – مركز تكنولوجيا الإشعاع – هيئة الطاقة الذرية – القاهرة – مصر.

فى هذا البحث تم تحضير متراكبات من البولي فينيل كلوريد/مطاط الأكريل نيتريل بيوتادين بنسب مختلفة عن طريق الخلط الحرارى. بعد ذلك تم كبس العينات عن طريق المكبس الحرارى للحصول على أفلام يسهل تقطيعها لإجراء الإختبارات المعملية عليها. ثم تشيع المتراكبات المحضرة عند جرعات 25، 50، 100 و 150 كيلو جراى من اشعة جاما. تم قياس التحليل الوزنى الحرارى و أيضا قياس الشد والإستطالة والصلابة للمتراكبات المشععة وغير المشععة. علاوة على ذلك تم قياس مقاومة هذه المتراكبات للزيوت المستخدمة فى السيارات سواء كانت زيوت المحرك أو الفرامل والقابض. وقد اتسمت المتراكبات المشععة بتغير فى مختلف الخواص الفيزيائية والميكانيكية والحرارية. كذلك أظهرت الدراسة أهمية دور المزيج بين نوعى البوليمر فى الخواص الحرارية ومدى تحسينها وكذلك مقاومة الزيوت محل الدراسة.