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Effect of gamma radiation on semi-crystalline polyvinyl chloride polymer for low-voltage cable insulator

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ABSTRACT

This study explores the properties of semi-crystalline polyvinyl chloride (PVC) polymer as insulation material for low-voltage (LV) cables under high gamma radiation exposure. Test samples underwent gamma radiation (^{60}Co) at doses of 25, 50, 100, 200, 400, and 800 kGy. The evaluation encompassed surface morphology, electrical conductivity, thermal characteristics, and mechanical properties via tensile tests. Electron microscopy observation indicated surface smoothing and flattening occurred at an irradiation dose of 800 kGy. Gamma radiation with increasing doses results in similar thermogram profiles with slight differences in melting temperature and residue mass. The sample irradiated at a gamma dose of 25 kGy generates an increase in the percentage of crystallinity, indicating the occurrence of crosslinking, while other doses exhibit a decrease of crystallinity with increasing radiation dose. Tensile stress significantly dropped up to 400 kGy but increased at 800 kGy. Elongation at break (EAB) decreased with higher gamma radiation doses. Overall, materials up to 800 kGy remained non-brittle, serving as effective insulators and demonstrating thermal stability within high gamma radiation exposure conditions.

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1. INTRODUCTION

The environment that cables must contend with varies depending on their installation location and intended use, encompassing high temperatures, gamma radiation, humidity, corrosive environments, and mechanical stresses [1]. The most used insulating material for wiring in low-voltage (LV) services is polyvinyl chloride (PVC) [2], the second most produced polymer worldwide [3] and it is also among the most widely manufactured synthetic plastics [4]. This is attributed to its superior mechanical and dielectric characteristics, excellent chemical resistance, lightweight nature, ease of processing, and affordable manufacturing [5]–[7]. However, polymers are more readily degraded by ion radiation than are metals and inorganic materials. Following the first high-energy radiation reaction, which includes the ionization and excitation of outer electrons as well as the production of free radicals, polymer molecular chains undergo cross-linking and scission [8]. Chain scission, cross-linking, and oxidation are usually the most significant impacts of ionizing irradiation on polymers [9]. Both chain scission and cross-linking reactions can happen at the same time [8], [9]. After radiation exposure, most polymers experience deterioration in mechanical, thermal, and chemical stability, color change, and rise in electrical conductivity, which causes materials to fail functionally [8].

Thermoplastic semi-crystalline polymers are characterized by segmental crystalline regions interspersed with disordered amorphous regions. The degree of crystallinity, which quantifies the extent of crystallization within a polymeric system, correlates with the rate of crystallization for most polymers and significantly influences their mechanical, thermal, electrical, and optical properties. Furthermore, crystallinity primarily affects ductility over molecular weight, with higher crystallinity resulting in decreased ductility. The strength of a polymeric material depends not only on the strength of its -C-C bonds but also on factors such as molecular weight, crystallinity, and degree of crosslinking, with an optimal molecular weight range enhancing polymer strength [10]. The primary obstacles associated with the use of thermoplastic polymers are the wide variations in their mechanical and physical properties across different polymers and macromolecular features, as well as the effects of processing conditions on these properties.

Apart from resin, PVC material for electrical cable insulation is predominantly compounded with plasticizers, fillers, and thermal stabilizers. Plasticizers alter their chemical structure, reducing crosslinking by increasing chain mobility and facilitating recombination. Certain fillers like zeolites, metal silicates, calcium carbonate, barium sulfate, magnesium oxide, and zinc oxide prevent dehydrochlorination and crosslinking in PVC. Thermal stabilizers, including epoxy stabilizers, maintain color integrity when PVC products are exposed to γ-radiation. Gamma radiation induces PVC degradation via processes such as radical abstraction, oxidation, and dehydrochlorination, leading to the formation of double bonds. Radical recombination post-irradiation contributes to polymer aging, affecting properties like stiffness and modulus through chain scission and crosslinking [11]. Under the impact of high energy irradiation, such as γ-rays, PVC is prone to deterioration through the release of hydrochloric acid (HCl) [12].

In the proposed project, the aim is to characterize the mechanical and thermal properties of PVC insulation cable materials after gamma irradiation. These thermoplastic semi-crystalline polymer materials consist of a specific blend including the plasticizer additive dioctyl phthalate (DOP), stabilizer, external lubricant, internal lubricant, and filler. The cable is intended for use in a post-irradiation examination facility with a high gamma radiation dose. Although previous research has investigated the impact of gamma radiation on PVC, the composition of additives in PVC significantly influences its mechanical and thermal properties. Knowledge of radiation's impact on PVC is crucial for various applications. Even minor changes in the insulating material's chemical composition or physical structure due to ionizing radiation can result in irreversible alterations to its electrical and mechanical properties, leading to insulator degradation [12]. Therefore, it is essential to understand this aspect of domestic PVC products. The PVC compound used for cable insulation in domestic production differs from that in previous research. Consequently, it is necessary to investigate the effects of gamma radiation on its mechanical and thermal properties for appropriate application.

2. MATERIALS AND METHODS

A new PVC compound was prepared by mixing PVC resin (K 70), plasticizer additive of DOP (44 phr), stabilizer (10 phr), external lubricant (0.4 phr), internal lubricant (0.6 phr), and filler (29 phr) using a vertical and horizontal mixer with mixing rate of 120 and 50 rpm, respectively. Extrusion of the compound occurred in twin screw and single screw extruder with temperature ranges from 140 to 170 °C and speed of 37 rpm for the twin screw and 30 rpm for the single screw. By the presence of crystalline additives, PVC compounds become semi-crystalline, as the percentage of the total amorphous substance of PVC decreased, and with a composition of DOP plasticizer (44 phr), this PVC is also plasticized PVC. The PVC compound, in the form of granules, was processed into plate form with a thickness of 4 mm. The functionalized groups produced by radiolysis of the parent polymer granules may be destroyed during the heating and pressure process that turns the granules into a plate [12]. The sheet was then sliced into samples of various shapes. Tensile test samples and other samples were irradiated in a Gamma Cell irradiator with a ^{60}Co source at a dose rate of 3.4 kGy/hour. The dose variations were 25, 50, 100, 200, 400, and 800 kGy. The unexposed sample (0 kGy) was used as reference material.

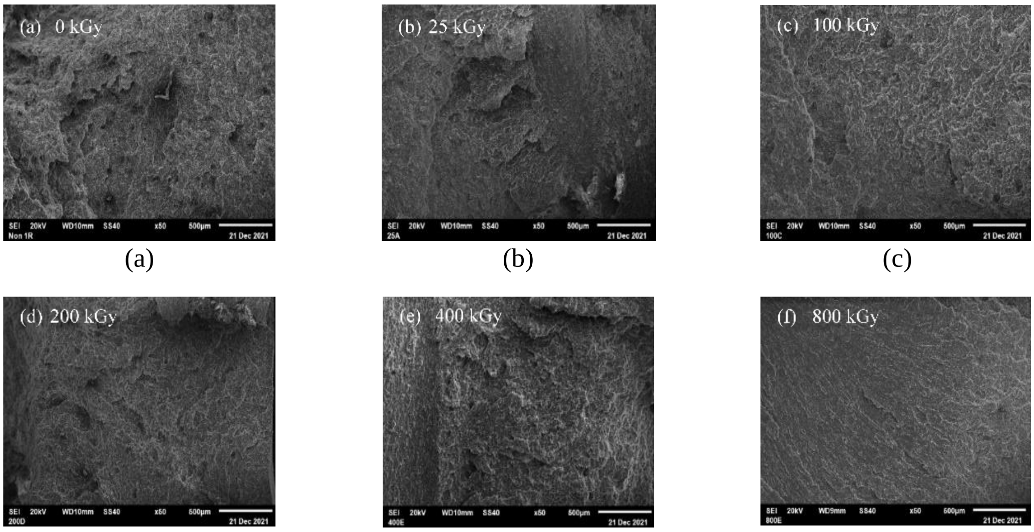
Surface morphology was observed using a scanning electron microscope (SEM) JEOL JSM-6510LA, with an electron beam voltage of 20 kV. A gold layer of 4-5 nm was deposited on the surface of the samples, to eliminate the effect of the dielectric properties of PVC. The secondary electron image of the samples was taken at magnifications of 50×, 250×, 500×, and 1000×. Thermal analysis was carried out using simultaneous thermal analysis (TG-DTA or TG-DSC) on a Labsys Evo by Setaram instrumentation in an Argon gas environment ultra high purity (UHP). The PVC samples were analyzed within a temperature range from ambient temperature up to 400 °C with a heating rate of 10 °C/min and a cooling rate of

20 °C/min. The TG-DSC apparatus automatically sets the gas flow rate after determining the cooling rate. The cooling process was continuously monitored, and the cooling rate was faster than the heating rate because no re-crystallization occurred. Electrical conductivity was measured using an LCR-meter (impedance, capacitance, resistance), Hioki 3522-50 LCR HiTester, and the frequency used was 3 kHz to 100 kHz with a voltage of 20 mV. The tensile test (BS EN 60811-501) was performed using a Shimadzu SCG-5kNA with a tensile speed of 20 mm/min.

3. RESULTS AND DISCUSSION

3.1. Surface morphology

A SEM at 50× magnification, as depicted in Figure 1 for each sample, revealed surface morphology. The secondary electron image at 50× provided clearer visibility than other magnifications (250×, 500×, and 1000×) due to smoothing effects. The non-irradiated sample showed a rough surface (Figure 1(a)), as well as exhibited by irradiated samples of 25, 100, 200, and 400 kGy (Figures 1(b) to (e)). The 800 kGy sample's surface (Figure 1(f)) exhibited smoothing and flattening, attributed to polymer flow under high radiation doses. Higher doses reduce crosslinks and matrix stiffness, increasing polymer chain mobility. This enhanced mobility allows the material to flow and reorganize under stress, resulting in a smooth surface morphology. Macromolecular breakdown also leads to surface smoothing because the material gets denser and more packed when short-chain fragments appear on the surface [1]. At lower doses of gamma radiation (up to 400 kGy), no surface smoothing occurred.



(a) (b) (c)

(d) (e) (f)

Figure 1. Surface morphology of the test samples with gamma radiation dose of; (a) 0 kGy, (b) 25 kGy, (c) 100 kGy, (d) 200 kGy, (e) 400 kGy, and (f) 800 kGy

3.2. Conductivity test

The ability of a material to transfer energy is known as conductivity. Ionic conductivity in electrolytes is dependent on the charged ions' capacity to flow through the medium. More mobility and a higher concentration of ions translate into stronger conductivity, which allows for the transmission of more energy [13]. The results of the conductivity test are presented in Table 1. Irradiated test samples exhibit lower conductivity compared to non-irradiated ones, which means that the material's insulating properties still function well and tend to improve after gamma irradiation.

Table 1. Conductivity test results (LCR)

Radiation dose (kGy)	Conductivity ($S.m^{-1}$)
0	$1{,}2552\times10^{-6}$
25	$1{,}7053\times10^{-6}$
50	$6{,}7236\times10^{-7}$
100	$1{,}5885\times10^{-9}$
200	$9{,}4406\times10^{-9}$
400	$6{,}9056\times10^{-7}$
800	$1{,}1389\times10^{-7}$

3.3. Thermal test

Quantifying the size of the crystalline portion in a polymeric system is known as the degree of crystallinity. For the majority of polymers, there is a correlation between the degree of crystallinity and the rate of crystallization. The degree of crystallinity affects mechanical, thermal, electrical, and optical properties. Polymer toughness and strength are improved by increased polymer crystallinity [10]. The degree of crystallinity (% crystallinity of PVC) can be calculated by measuring the amount of heat required to melt a sample using DSC (ΔH_m) and comparing it to the heat of fusion of perfect crystalline PVC ($\Delta Hm_{100\%}$), using (1) [14]–[17]:

$$\% \text{ crystallinity of PVC} = \left(\frac{\Delta H_m}{\Delta H_{m\ 100\%}}\right)\times 100 \qquad (1)$$

3.3.1. Thermogravimetric analysis

The TGA thermogram results (Figure 2) reveal a single-stage decomposition reaction in an inert gas environment, leading to the complete breakdown of the polymer. The loss of volatile chemicals resulted in an early mass loss before full decomposition [18]. Mass and heat transfer processes always coexist with chemical events during thermal degradation. Thermal analysis (TA) approaches measure changes in the physical quantities as a function of temperature (T) or time (t) during thermal degradation [19].

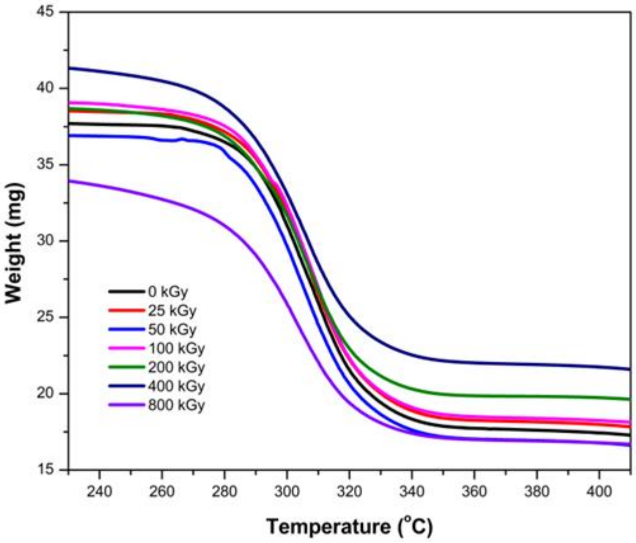


Figure 2. TGA thermograms of non-irradiated and irradiated samples with gamma doses of 25 kGy, 50 kGy, 100 kGy, 200 kGy, 400 kGy, and 800 kGy

Figure 2 displays the TGA curves of both irradiated and non-irradiated PVC. There are three main areas for weight loss. The first major loss occurred at 270 °C as a result of the loss of water content, causing the stable zone to extend up to 290 °C. The second major loss occurred in the range of 290–320 °C as a result of the loss of the dopant from the polymer matrix (fast decomposition zone), and the third major loss occurred between 320–350 °C as a result of the destruction of the polymeric chain backbone. In addition, it was shown that the initial breakdown temperature of radioactive PVC reduced as the radiation dose increased. With increasing the dose, the stable zone, fast decomposition zone, and residual decomposition zone should all shift towards lower temperatures. The oxidative breakdown of the polymer electrolyte causes a change in the molecular structure and weight as well as the production of free radicals [20].

Thermogravimetric (TG) data are summarized in Table 2, including the temperature at which the degradation rate is maximum (T_p), onset degradation temperature defined as the temperature at which 1% weight loss takes place ($T_{1\%}$), and weight loss at the end of the first degradation step around 400 °C (Wt_{400^oC}). There is no significant difference in Tp value between before and after irradiation, indicating that the dehydrochlorination mechanism is not very sensitive to increasing radiation doses. It is possible to compare the non-irradiated PVC results with those found in the literature. Non-irradiated PVC begins to decompose thermally around 266 °C, which is greater than the 183 °C found in Imene's study. The maximum decomposition rate occurs at 356 °C. The total weight loss recorded from ambient temperature to 400 °C is equal to 47 wt%, lower than the values determined by Matuschek (T_p=260 °C; $Wt_{400°C}$=64%), Slapak ($Wt_{400°C}$=62%), and Miranda (T_p=277 °C; $Wt_{400°C}$=64%) [21].

Table 2. Thermal degradation of plasticized PVC irradiated at various doses

Dose kGy	0	25	50	100	200	400	800
$T_{1\%}$ (ºC)	266	262	272	236	247	256	220
T_p (ºC)	356	356	355	359	354	353	356
Wt_{400^oC} (%)	47	48	46	47	52	52	48

The main effect of radio-oxidation is a reduction in the onset degradation temperature: the difference $T_{1\%}$ is not close to T_p for non-irradiated PVC and also for irradiated PVC, while a decrease of 46 ºC is recorded after irradiation at the highest dose. Thermograms also reveal that for pre-irradiated polymers, weight loss increases above 270 ºC. These findings suggest that there may be additional factors contributing to the weight loss of radio-oxidized PVC [21].

3.3.2. Differential scanning calorimetry

DSC is a convenient method for analyzing the thermal behavior of polymers. Figure 3 shows the PVC polymer's melting point at various doses with a sharp endothermic peak corresponding to the melting point (T_m) at 308°C (Figure 3(a)) for non-irradiated sample, and 308 °C (Figure 3(b), 25 kGy), 309 °C (Figure 3(c), 50 kGy), 311 °C (Figure 3(d), 100 kGy), 308 °C (Figure 3(e), 200 kGy), and 306 °C (Figure 3(f), 400 kGy) for irradiated samples. After irradiation at 800 kGy, the peak is reduced to 304°C (Figure 3(g), indicating a potential loss of crystallinity. DSC results in Figure 3 indicate the enthalpy change associated with the transition or reaction, calculated by considering calibration factors and sample mass. As an amorphous polymer, PVC can integrate any other polymer into its matrix. The absence of crystallinity in PVC makes it prone to heat instability and degradation during molding and sunlight exposure [3]. The crystallinity is determined by comparing the fusion heat with that of 100% crystalline PVC polymer, the maximum value is selected as 176 J/g [22]. It is around 75% before irradiation, as shown in Figure 4, and shows a decrease with the increase in gamma radiation dose.

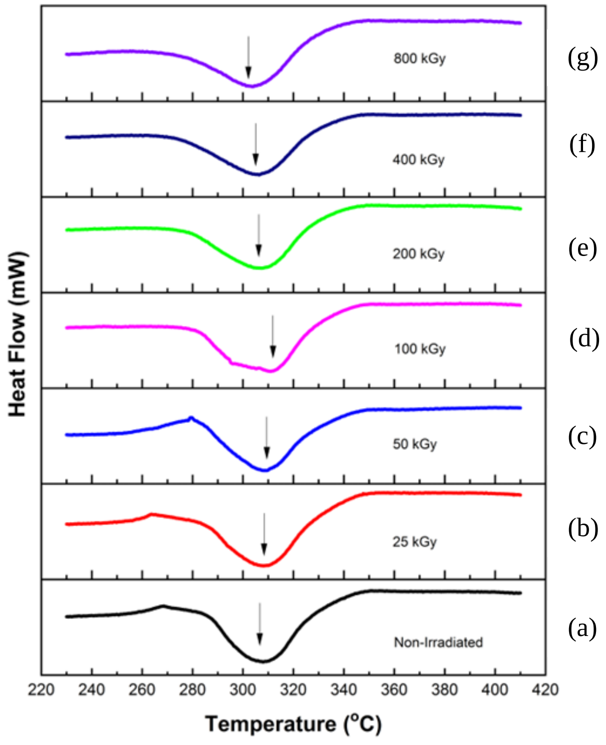


Figure 3. DSC thermogram of; (a) non-irradiated sample, and irradiated samples with gamma dose of, (b) 25 kGy, (c) 50 kGy, (d) 100 kGy, (e) 200 kGy, (f) 400 kGy, and (g) 800 kGy

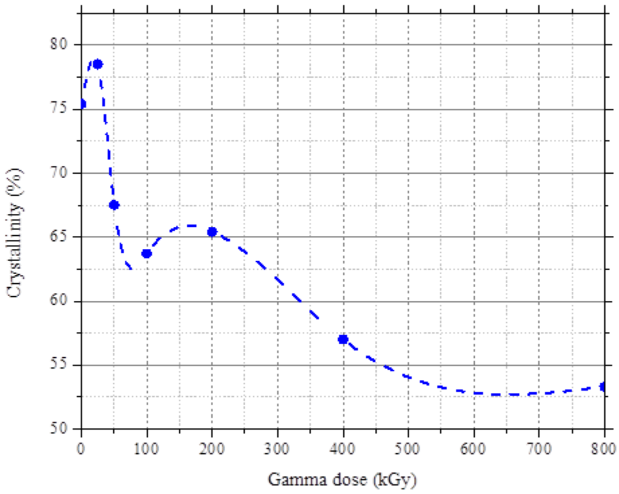


Figure 4. Crystallinity vs gamma dose

The relationship between crystallinity and mechanical properties is complex; increased crystallinity may enhance tensile strength but reduce elongation at break (EAB). The enthalpy of fusion (ΔH_fus) is obtained from DSC results, calculated as the integrated heat flow from the melting peak divided by the sample mass, indicating the heat absorbed per unit mass during material melting. It represents the energy needed for a substance to shift from a solid to a liquid state. A decrease in polymer crystallinity suggests a higher proportion of amorphous regions, which, due to a less ordered structure, facilitates the material's transition to a liquid phase upon heating. Typically, reduced crystallinity correlates with a lower melting temperature, but exceptions exist based on specific polymer characteristics. The physical characteristics of polymer materials are greatly influenced by the degree of crystallinity. The degree of crystallinity of polymers is connected to several of their physical properties, including mechanical, optical, thermal, and electrical properties [23]. Less tensile EAB resulted from increased crystallinity [24] and a reduction in mechanical characteristics may be caused by a decrease in crystallinity [25].

3.4. Mechanical test

Figure 5 depicts stress variations in PVC samples at different gamma irradiation doses up to 800 kGy. Tensile test data reveals that the 25 kGy sample exhibits higher stress compared to the non-irradiated sample. This indicates the increased ultimate tensile stress due to cross-linking and decreased stress in chain scission [26]. Radiation effects can alter polymer structure through chain scission, crosslinking, and degradation, impacting mechanical performance [27].

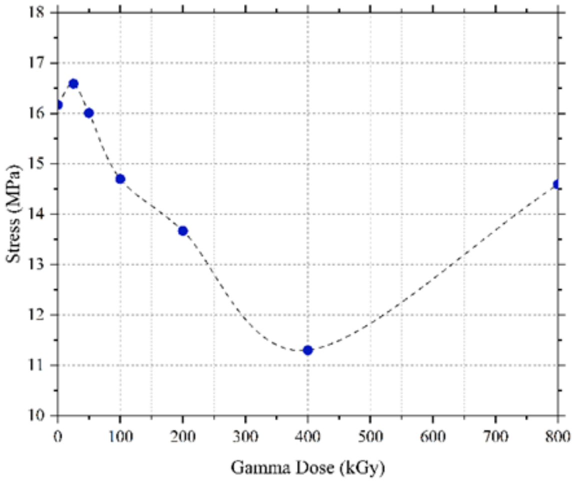


Figure 5. Stress vs gamma radiation dose

The data in Figure 5 shows increased tensile strength at doses of 0-25 kGy and 400-800 kGy, suggesting a cross-linking process. However, for other dose ranges, tensile strength degrades, indicating chain scission. At 800 kGy, the irradiated sample's stress is 14.6 MPa, compared to 16.2 MPa for the non-irradiated specimen. Figure 6 illustrates a decrease in EAB with increasing radiation doses. Even at 800 kGy, the sample retains some ductility with a 3% EAB. The aging time at which the EAB decreased to 50% of the starting value was chosen as the end-point, following IEC 60216-2: 2005, and by interpolation, the EAB decreased to 50% (6,6%) is at 165 kGy [28]. Degradation, involving chemical bond breakage and molecular chain cutting, may occur at doses of 50-400 kGy. Increasing radiation dose causes stress reduction, evident in Figures 5 and 6 EAB, possibly due to similar causes as stress reduction.

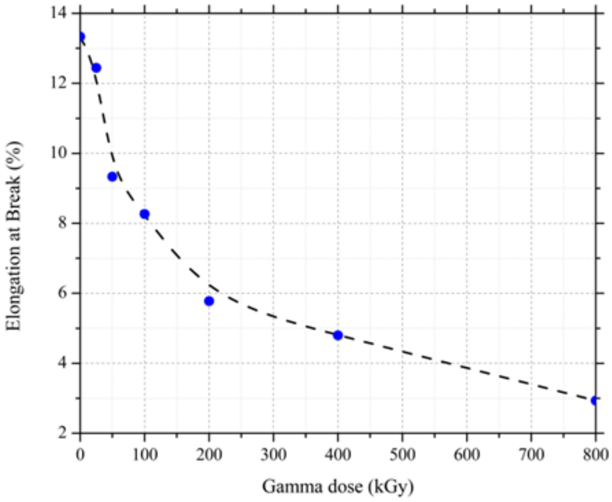


Figure 6. Percentage of EAB vs gamma radiation dose

4. CONCLUSION

Semi-crystalline PVC compound exposed to gamma irradiation was analyzed through several tests to compare its properties with non-irradiated samples regarding the application as insulation material for LV cables. Gamma radiation generally reduces stress and EAB in PVC insulation as the dose increases. Crystallinity decreases with higher radiation doses, but up to 800 kGy, the material remains functional with over 50% crystallinity. Thermal stability is minimally affected by gamma radiation. At 25 kGy, there's crosslinking and increased crystallinity, while other doses decrease the crystallinity of the compound. The aging time for 50% EAB reduction is 165 kGy. More research is needed to understand mechanical property changes under gamma radiation for polymer modifications. The author's suggestion for future work is to improve radiation resistance by applying nanofillers to obtain a PVC-based composite.

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