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Modifications in polymeric properties due to different doses of gamma irradiation ranging from 10¹ Gy to 10⁶ Gy: An account

Dipak Sinha

Department of Chemistry, Nagaland University, Lumami, Nagaland, India

ABSTRACT

When polymeric materials like Polyallyl diglycol carbonate (PADC), Polycarbonate (PC), Polyvinyl Chloride (PVC), Polypropylene (PP) etc. are exposed to gamma radiation, they show pronounced changes in their physical and chemical properties. Interestingly, the changes due to irradiation are varied in nature, proving that all polymers do not undergo similar type of modifications under similar irradiation conditions. Thus, while polymers like PADC and PVC do not reveal any change in the IR spectral pattern, the IR spectrum of polypropylene polymer confirms the total destruction of isotactic arrangements of the polymer leading to the formation of alcoholic and ketonic groups. The etch-rates are drastically enhanced due to irradiation at the highest dose for all types of PADC detectors which is not the case for polycarbonates. For polyacetate polymer, elimination of carbon dioxide takes place due to destruction of the ester group. Interestingly, in polycarbonate polymer it was observed that at the dose of 10° Gy, phenolic groups form due to cleavage of ester bonds. Thermal stability of PADC (American-Acrylics) is drastically reduced at the doss of 10^6 Gy, whereas for polypropylene polymer, the stability is greatly enhanced. It is also observed that due to etching, thermal stability of PADC detectors decreases. Dielectric studies reflect the fact that at a gamma dose of higher than 10^4 Gy, PADC detector may not behave as an ideal nuclear track detector. This paper accounts the modifications on different types of polymeric track detectors due to gamma irradiation. Variations in different properties such as track properties, structural modifications, thermal properties, electrical properties etc. through a dose range of 10^1 Gy to 10^6 Gy are discussed

Key words: Polymer, Gamma Dose, Gy

INTRODUCTION

The application of radiation on polymeric materials is of great importance as it allows achievements of desired modifications in polymer properties. Irradiation of polymeric materials with gamma rays, X-rays, accelerated electrons and ion beams for example leads to the formation of reactive intermediates such as free radicals, ions and excited states which then lead to reactions like chain scission, chain aggregation, formation of double bonds and molecular emission. The outcomes of these reactions are formation of oxidized products, grafts, scission of main chain (degradation) or cross-linking. Often the two processes (degradation & cross-linking) occur simultaneously, and the final outcome of the process is determined by a competition between the reactions [1,2]. Oxidation and degradation occur gradually with increasing irradiation dose. Different polymers have different responses to radiation, which are intrinsically related to the chemical structures of the polymers. As a consequence of this, physico-chemical properties of the polymer become modified [3-10].

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Responses of track detectors are different for different types of radiation, and the applications of the detectors are dependent on the type of radiation they are exposed to. This property can be utilized to modify the polymer properties, as can be accessed from the fact that a lot of work has been reported on the influence of gamma radiation on nuclear track detectors [11-18]

Our group has been involved with the study of polymer modification through gamma radiation for the last few years, which has led to some understanding of the type of modifications that takes place in the irradiated polymers. This paper presents an insight of the behavioral trends of the modifications caused in polymer materials by different doses of gamma radiation.

MATERIALS AND METHODS

Seven samples of the polymeric material/detector of various thickness and sizes $(3 \times 3 \text{ cm}^2 \text{ and } 2x2 \text{ cm}^2)$ were exposed to ⁶⁰Co gamma source having a dose rate of 3.0 k Gy/h. The exposure time varied from 12 s to nearly 14 days in order to deliver the required doses in the range of 10^1 to 10^6 Gy. The errors in doses range from 8% for low dose (10 Gy) to about 1% for high doses.

For track studies, one set of each sample was first exposed at normal incidence to alpha and fission fragments from a 252 Cf source (having a half-life of 2.65 years and activities of 5.7×10^3 fission/min and 1.84×10^5 alpha particles/min). The pre-exposed samples, along with the unexposed second set of samples, were then irradiated with various doses of gamma radiation in the dose range of 10^{1} – 10^{6} Gy. Subsequent to the gamma exposure, the second set was exposed to the 252 Cf source for alpha and fission fragment detection. The first and the second sets are referred to as the post- and pre-gamma sets, respectively in this work.

The bulk etch rate (V_G) and track etch rate (V_T) were determined from the following expressions

 $V_G = D_{ff}/2t$, $V_T = V \times V_G$, Where $V = 1 + x^2/1 - x^2$, $x = D_{ff}/D_a$,

where $D_{\rm ff}$ is the diameter of the fission tracks and D_{α} the diameter of the alpha tracks [11]

RESULTS AND DISCUSSION

This paper is a report of the modifications that are caused on different polymers/polymeric track detectors due to gamma irradiation. The modifications had been earlier assessed by different analytical techniques [19-35] and a compressive account is now presented.

On PADC Detectors:

Gamma effect on four different types of PADC (Polyallyl diglycol carbonate) detectors was studied [19-29]. The PADC detectors used are classified on the basis of their origin, as follow

Name of the detector	Manufractured by	Thickness
PADC	Homalite	1500 µm
PADC	American Acrylics	650 µm
PADC	Pershore	1000µm
PADC	Trastrack	650 µm

The chemical structure of PADC is given below



Since PADC is known to be one of the most widely used track detectors, modifications on track properties were studied for these detectors. Track studies revealed that in almost all the cases, both the bulk and track etch rate start

increasing significantly at a dose higher than 10^4 Gy. This increase in etch rates was observed in both pre- and postgamma exposed detectors. Since the etch rate increase is generally due to scissioning of the molecular chain, it may be assumed that scissioning of the molecular chain starts at a dose of 10^4 Gy. With increasing dose, the intensity of chain scissioning increases. It was also observed that post-gamma irradiated detectors had higher etch rate values than pre-gamma irradiated samples. This could be due to further scissions caused by gamma radiation. The more scissioning of the polymeric chain, the more is the reduction in the average molecular weight of the polymer, and thus the etch rate values increase. Another explanation might be that the post-gamma exposure hinders the annealing of fission product radiation damage, which ultimately increases the etch rates. Table 1 shows the bulk-etch rates for pristine and irradiated detector at a dose of 10^5 Gy and 10^6 Gy, for the etching temperature of 60^0 C, of both pre and post-gamma detectors. The detailed modifications on different PADC detectors are available in our earlier reports [19-21].

Detector	Etching Temperature	No Dose	10 ⁵	Gy	$10^6 \mathrm{Gy}$	
			pre-gamma	post-gamma	pre-gamma post-gamma	
Homalite	60^{0} C	0.69 ± 0.03	0.75 ± 0.03	0.76 ± 0.03	9.33 ± 1.2 11.20 ± 1.2	
Pershore	$60^{\circ}C$	1.50 ± 0.03	4.20 ± 0.03	5.90 ± 0.03	15.60 ± 1.2 22.40 ± 1.2	
Trastrack	$60^{\circ}C$	2.50 ± 0.03	3.80 ± 0.03	5.60 ± 0.03	$21.00 \pm 1.2 \ \ 26.20 \pm 1.2$	
American-Acrylics	$60^{0}C$	0.59 ± 0.06	1.22 ± 0.10	1.58±0.10	18.90+1.3 26.95 +1.8	

Table1. Bulk etch rate (V _G) in	µm/h for gamma-irradiated PADC detectors
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Since the etch rate increase is generally correlated with scissioning of the molecular chain, there is possible of cleavage of Polyallyl linkage with diethylene glycol, as has been reported earlier [20]. This scissioning of the chain lead to the formation of radical (Fig.1) as was detected in PADC (American-Acrylics) at a gamma dose of 10^6 Gy [20]







Figure.2 TGA thermogram of gamma irradiated PADC (American- Acrylics) [20] shows that at the highest dose, thermal stability decreases

Gamma irradiation does not seem to have significant effect on thermal stability of PADC detectors [23]. However, only in the case of American-Acrylics detector, thermal resistance decreases drastically due to gamma exposure (Fig.2) at the highest dose of 10^6 Gy [20, 23].

This decrease in thermal stability in American Acrylics can be attributed to the scissioning of the polymer chain. However for other types of PADC detectors (Fig.3), thermal stability does not seem to be modified due to irradiation.



Figure.3 TGA thermogram of gamma irradiated PADC (Homalite) [23] does not show any significant modifications

Further it was observed that chemical etching causes the thermal stability of the PADC detector to decrease to a significant extent. Fig.4 shows the TGA thermogram for etched and un-etched detector, which clearly evidences the decrease in thermal stability of the etched detector [28].



Fig. 4. TGA thermogram of gamma irradiated un-etched and etched PADC (American-Acrylics) detector [28]

The dielectric response of gamma irradiated PADC (American Acrylics) samples indicates an increase in dielectric constant values at a dose of 10^6 Gy (Figure 5). An increase has been observed at all measured frequencies implying an improvement in the orientational polarisation of the PADC molecules with the applied electric field. Considering the ion explosion spike model, it could be possible that at this highest dose, the detector may not behave as a good track detector [22].

On Polycarbonate Detectors

An account of the different types of polycarbonate detectors as given below is reported here, based on results of earlier studies [30,31].

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Name of the polymeric detector	Manufactured by	Thickness
Makrofol-E	Bayer AG, FRG	300 µm
Polycarbonate	Bayer AG, FRG (transparent)	300 µm
Lexan	GEC, USA	500 µm
Polycarbonate	Bayer AG, FRG (semitransparent)	100 µm



Fig.5 Variation of dielectric constant with frequency in pristine and irradiated PADC (American Acrylics) [22]

The Chemical structure of polycarbonate polymer is given below



Polycarbonate polymer is known as bisphenol-A polycarbonates, having two benzene rings in the monomer unit. These aromatic groups are known as radiation stabilizers, and have the ability to reduce the polymer irradiation sensitivity due to delocalization of the excitation energy. Probably the presence of aromatic groups in these polymeric films makes them radiation insensitive and hence the back-bone structure remains intact. This possibility explains the fact that no significant change in the bulk-etch rate of these detectors is observed even after exposure to gamma radiation as high as 10^6 Gy [30]. Table 2 presents the etch rates of different types of polycarbonate detectors at different doses [30].

Table 2. Bulk etch rate (V_G) in μ m/h for g	gamma-irradiated Polycarbonate detectors
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Detector	Etching Temperature	No Dose	10^{3}	Gy	10 ⁶ Gy	
			Pre	post	Pre	post
Makrofol-E	$60^{0}C$	$0.73 \pm .06$	$0.72 \pm .06$	$0.78\pm.06$	$0.90 \pm .06$	$1.03 \pm .06$
Lexan	60^{0} C	$0.64 \pm .03$	$0.59\pm.03$	$0.64\pm.03$	$0.73 \pm .03$	$0.85 \pm .03$
Polycarbonate (transparent)	60^{0} C	$0.62 \pm .03$	$0.62\pm.03$	$0.63\pm.03$	$0.74 \pm .03$	$0.73 \pm .03$
Polycarbonate (semitransparent)	60^{0} C	0.67 ±.03	$0.66 \pm .03$	$0.67\pm.03$	$0.84 \pm .03$	$0.89 \pm .03$

Even though there was no apparent bond breaking of the total polymer structure, ESR studies reveal some information about the cleavage of C-H bond of the benzene rings [R]. ESR spectra show the formation of radical in the polymer irradiated at the highest dose (Fig.6).

ESR studies give further information about the cleavage of C-H bond of the benzene rings. The radical which is formed from the carbon atoms delocalizes in the ring system and the hydrogen radical formed probably combines with other hydrogen radicals to form hydrogen molecules.

Fig.6. ESR spectra of gamma irradiated Makrofol-E detector, shows the radical signal at a dose of 106 Gy[30]

The thermal resistance of these detectors is not found to be modified after irradiation [23,30]. TGA thermogram of polycarbonate (300 μ m), ascertains this (Fig.7). Only it was observed that at the highest dose of 10⁶ Gy, the polymer thermal stability decreases to some extent and weight loss process starts at lower temperature i.e. at 420°C and continues up to 700°C.

Interestingly, in case of one polycarbonate polymer (100 μ m), it was observed that glass transition temperature decreases (Fig.8) with increasing gamma dose [31].

Fig.8 Dose-dependent variation of Tg for gamma-irradiated polycarbonate polymer [31]

IR spectra of polycarbonate (100 μ m) indicate that owing to gamma exposure, polycarbonate polymer forms phenolic group at the dose of 10⁶ Gy. This phenolic group forms due to cleavage of ester bonds as explained by us earlier [31].

Fig.9 XRD spectra of gamma-irradiated polycarbonate polymer [31]

XRD studies indicate the presence of two types of nano-crystalline zones which are dispersed in an amorphous matrix. With increasing gamma dose the intensity of both peaks increases. These results reflect that the amorphous character of the polymer decreases and crystallinity increases with increase in dose, which may be due to scissioning of the polymer chains, by which the polymer undergoes some spatial rearrangement. The small fragments may rearrange themselves towards new crystalline zone. Figure 9 shows the XRD spectra of pristine and gamma irradiated polycarbonate polymer ($100 \mu m$).

Polyacetate Polymer

Gamma effect on different types of polyacetate polymer as given below were studied [32,33]

Name of the polymer	Manufactured by	Thickness
Triafol-TN (cellulose triacetate)	Bayer AG. Leverkusen, FRG	100 µm
Triafol-BN (cellulose acetate butyrate)	Bayer AG. Leverkusen, FRG)	200 µm
Polyacetate	Bayer AG. Leverkusen, FRG	770 µm

The bulk-etch rate (V_G) was determined by the track diameter technique at different etching temperatures (55, 60, 65 and 70^oC). The bulk-etch rates for both pre- and post-gamma exposed Triafol-TN detector in the temperature range from 55^o C to 70^oC is increased by about 10 -15% up to a dose of 10⁵ Gy. In the case of Triafol-BN, the bulk-etch rate is practically independent of the gamma dose up to 10⁵ Gy. However, at 10⁶ Gy a steep increase in V_G is observed which is 30 to 50 times higher than the average V_G at 10⁵ Gy. This marked increase in V_G may be due to scissioning of the molecular chains caused by gamma radiation. It was also observed that the etch-rate remains invariant for both pre- and post-irradiated samples[32] Table 3 and Table 4 lists the bulk-etch rate values for Triafol-TN and Triafol-BN for pre-gamma set at different doses and at different etching temperatures.

Table 3. Bulk- Etch Rate (V_G) in µm/h For Triafol-TN for pre gamma exposure

Temperature	No Dose	10 ¹ Gy	10 ² Gy	10 ³ Gy	10 ⁴ Gy	10 ⁵ Gy
55°C	$1.75 \pm .07$	$1.74 \pm .07$	$1.77 \pm .07$	$1.89 \pm .07$	$1.80 \pm .07$	$1.95 \pm .07$
60°C	$2.35 \pm .12$	2.39±.12	2.42±.12	2.41±.12	2.40±.12	2.42±.12
65°C	$2.76 \pm .09$	2.83 ± 09	2.84±.09	$2.84 \pm .09$	2.88±.09	2.86±.09
70°C	$3.42 \pm .13$	3.61±.13	3.62±.13	3.75±.13	3.77±.13	3.83±.13

Temperature	No Dose	10 ¹ Gy	10 ² Gy	10 ³ Gy	10 ⁴ Gy	10 ⁵ Gy	10 ⁶ Gy
55°C	0.38±.04	0.39±.04	$0.38 \pm .04$	0.39±.04	0.39±.04	$0.50 \pm .04$	14.66±1.6
60°C	0.64 ± 05	$0.65 \pm .05$	$0.65 \pm .05$	$0.65 \pm .05$	$0.65 \pm .05$	$0.68 \pm .05$	31.38±2.9
65°C	0.70±.06	0.73±.06	0.73±.06	0.72±.06	$0.72\pm.06$	$0.75 \pm .06$	40.61±4.2
70°C	0.82±.06	$0.82 \pm .06$	$0.83 \pm .06$	0.84±.06	$0.84 \pm .06$	1.10±.06	49.10±4.2

Table - 4 Bulk- Etch Rate (V_G) in µm/h for Triafol-BN for pre gamma exposure [32]

UV-Vis study of Triafol-BN (Fig.10) clearly reflects the oxidation of polymer at the dose of 10^6 Gy, which could be due to removal of antioxidants present in the polymer as explained by the disappearance of the peak (shoulder) at 310 nm [32]. At the dose of 10^6 Gy, this shoulder disappears implying that the antioxidant was removed by radiation from Triafol-BN and thus oxidation was favored and the detector became brittle.

Fig. 10. UV -VIS absorption spectra of gamma irradiated Triafol-BN detector [32]

IR studies some information about the ester group destruction of the Triafol-BN polymeric film at 10^6 Gy gamma dose as reported earlier [32]. This destruction leads to a decrease in the average molecular weight and consequently the bulk-etch rate increases.

Due to gamma exposure the dielectric constant of the polyacetate polymer increases at a dose higher than 10^4 Gy (Fig.11). The increase is visible in the lower frequency region i.e. in the range of 42-1000 Hz. The increase in dielectric constant value is probably due to irregularity in the polymer chains caused by irradiation. This irregularity gives rise to a hopping mechanism which enhances the polarization in the polymer matrix at a dose higher than 10^4 Gy [33].

Fig. 11. A comparison of dielectric constant value of pristine and irradiated polyacetate polymer at different frequencies[33]

Polypropylene (PP) Polymer

Interestingly it is observed that due to gamma exposure at the dose of 10^6 Gy, oxidation takes place by removing the antioxidant present in the film which probably facilitates random destruction of the polymeric chain with the formation of alcoholic and ketonic groups. The detail mechanism about the formation of alcoholic and kenotic group

is explained in our earlier report[34]. Figure 12 show the IR spectra of the pristine and gamma irradiated polypropylene polymer irradiated by gamma radiation.

Fig.12 FT-IR spectra of pristine and irradiated Polypropylene polymer [34]

With the formation different unsaturated groups in the polymer matrix, the optical band-gap of polymer decreases with increasing dose and it is prominent above the dose of 10^3 Gy. The optical band gap from 4.59 eV from pristine sample reduces to 2.07 eV for the sample irradiated at 10^6 Gy. The UV-Vis spectra of the irradiated polymer at different doses are shown in Fig.13, which clearly reflects the shift of absorption edge towards higher frequency due to decrease in band gap [34].

Figure 13. UV-VIS spectra of gamma irradiated Polypropylene polymer shows that due to increase of gamma dose, adsorption edges to higher wavelength [34]

TGA study of the polypropylene polymer clearly concludes that due to gamma exposure at the dose of 10^6 Gy, the thermal resistance of the polymer increases (Fig.14). Up to the dose of 10^5 Gy, the decomposition of the polymer starts at around $260-270^{\circ}$ C and completes at around $330-335^{\circ}$ C. But at the dose of 10^6 Gy, decomposition starts at a much lower temperature, i.e., around 178° C, and continues up to about 550° C and the polymer is found to decompose in four different steps.

Fig.14 TGA thermogram of pristine and gamma irradiated polypropylene polymer [34]

Polyvinyl Chloride (PVC) Polymer

Gamma irradiation of Polyvinyl Chloride shows that at a dose higher than 10^3 Gy, optical band gap for the PVC polymer decreases with increasing dose [35]. This decrease is more significant at higher gamma doses and at the dose of 10^6 Gy observable scissioning of the C-Cl bonds takes place as follows [35].

This scissioning of the C-Cl bond leads to formation of double bonds as can be seen in IR spectra. This reduces the thermal stability of the PVC and the irradiated polymer decomposes at temperatures lower than those for the pristine polymer. The heat evolved due to the decomposition of the polymer decreases to a large extent at the dose of 10^5 Gy. At higher dose (10^6 Gy), the exothermic behavior of the polymer disappears due to the scissioning of C-Cl bond [35] as can be seen in Figure 15.

Fig.15 DSC thermogram shows the disappearance of exothermic peak at dose of 10⁶ Gy [35]

CONCLUSION

On the basis of the study had been made on different types of polymeric materials the following conclusions are made

a. Due to gamma exposure etch-rates of the PADC detectors increases significantly at a dose of 10^6 Gy and this increase is more pronounced for post-gamma exposed detectors.

b. Thermal stability of PADC-American Acrylics decreases drastically at a dose of 10^6 Gy, which is not the case of other types of PADC detectors. Due to chemical etching, the thermal stability of PADC detector decreases more. c. Dielectric constant of the PADC detector increases at the highest dose of 10^6 Gy.

d. Gamma irradiation does not seem to have any significant effect on bulk-etch rates of polycarbonate detectors.

e. Amorphous character of the polycarbonate polymer decreases and crystallinity increases with increase in gamma dose

f. Owing to gamma exposure, polycarbonate polymer forms phenolic group at the dose of 10^6 Gy.

g. Bulk-etch rate of Triafol-BN increases significantly at the highest dose of gamma exposure which is not the case for Triafol-TN detector.

h. Due to gamma exposure the dielectric constant of the polyacetate polymer increases at a dose higher than 10⁴ Gy

i. Gamma exposure leads to formation of alcoholic and ketonic groups in polypropylene polymer

j. Thermal stability of polypropylene increases due to gamma exposure

k. Gamma irradiation leads to formation of polyenes in PVC polymer due to scissioning of C-Cl bonds

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