Morphological, Electrical, Structural and Optical Properties of Co⁶⁰ Gamma Rays Irradiated Polyethersulfone (PES) Polymer

Siddhartha^{1*}, Suveda Aarya¹, Mohd Shakir¹, A.K. Srivastava¹ and M.A. Wahab²

¹Department of Physics, Jamia Millia Islamia, New Delhi 110025, India ²Department of Physics, Sharda University, Greater Noida, UP. – 20130, India *Corresponding Author E-mail: siddharthasingh1@gmail.com

Abstract

The morphological, electrical, structural, and optical properties of Co^{60} gamma rays irradiation on Polyethersulfone (PES) polymer were studied by using Scanning Electron Microscope (SEM), High Frequency Analyzer Impedance Techniques, X-Ray Diffraction (XRD) and Ultraviolet/ Visible (UV/VIS) absorption spectroscopy. The morphology study shows that blisters of size 1 μ m were observed on the surface of PES polymer due to Co⁶⁰gamma ray irradiated at ambient temperature, the effect of Co⁶⁰gamma irradiation on conductivity behaviour of Polymer were investigated by the impedance spectroscopy at ambient temperature. The variation of dielectric permittivity and dielectric loss with frequency was carried out at ambient temperature. There is an exponential increase in a.c. conductivity with log frequency and the effect of irradiation is significant at higher doses. The dielectric permittivity and dielectric loss is observed to change significantly due to irradiation. It has been found that dielectric response in both virgin as well as irradiated samples obey the Jonscher's power law. The XRD pattern of virgin sample shows that polymer is semi-crystalline but due to irradiation, a decrease in the peak intensity and an increase in the FWHM up to the dose level 142 kGy have been observed. At the highest dose level of 300 kGy, the polymer shows a recovery characteristic. The crystallite size also shows a recovery characteristic in the studied range of doses. The band gap calculated from the UV-VIS spectra shows an appreciable change.

Keywords: PES, SEM, Dielectric, XRD, UV-VIS spectroscopy, Gamma radiation.

Introduction

Polymers have proven their potential in all fields of life and specially in the fields of medical science, electronics and space technology. Polyethersulfone (PES) is a heatresistant, transparent, amber, semi-crystalline polymer. The most characteristic feature of PES polymer is that it has better high-temperature properties than the conventional engineering plastics. Specifically, PES polymer remains in satisfactory condition in long-term continuous use without causing any dimensional change or physical deterioration at temperatures as high as 200°C. Use of radiation in polymers has great importance because it helps in achieving some desired improvements in the polymer properties. Gamma radiation treatment provides a unique way to modify the chemical, structural, optical, mechanical and electrical properties of the polymer by causing irreversible changes in their macromolecular structure [1-6]. Numerous studies reported in the past few decades demonstrate that the interaction of ionizing radiations-induced modifications with PES polymers leading to a wide variety of property changes [7-11]. Many investigators have studied the effect of different energy ion beam irradiations on the physical, electrical and chemical properties of PES polymer. Such irradiations may produce the electrons and low energy photons which are responsible for the modification of the material. The breaking of polymer chains and creating free radicals. These free radicals can also recombine to create crosslinks between the adjacent molecules. Different studies of effect of ion irradiation on polymers, reveal a variety of modifications of structural, electrical, optical and chemical compositions including processes such as main chain scission, intermolecular crosslinking, creation of unsaturated bonds, formation of volatile fragments and creation of carbonaceous clusters Kazuo et al. [12], Nilam et al. [13] Xianqiang et al. [14]. For ion beam irradiation, the following aspects are well established: (1) Energy loss (dE/dx) by charge particle in the material medium (electromagnetic interaction, high concentration of excited and ionized target atom) is differential in nature and hence energy deposited is non-homogeneous in nature Suveda et al. [4]. It is confined to the beam diameter only. However, the gamma irradiation have been found to have the ability to expose the whole area of the sample and hence expected to create homogeneous modification in it. Although a lot of work has been done to investigate the effect of ion irradiation on polymeric materials but the dependence of effect parameters related to ionizing radiation has not been completely understood so far. In this article, we report the results of morphology, electrical and chemical changes produced by Co⁶⁰ gamma ray induced modifications in PES polymer. The molecular structure PES polymer is shown below



Experimental

The Polyethersulfone (PES polymer sheets of thickness 250 µm were obtained from Messrs Good Fellow, Cambridge, U.K and were used without any further treatment.

Co⁶⁰Gamma ray irradiation

The polymer samples of size (1×2) cm² were cut from the commercially available sheet. One sample was kept as virgin and the other four samples were irradiated at various doses, in order to study the effect of radiation doses. The samples were irradiated in vacuum at the atmospheric pressure of 10⁻³ torr in ambient temperature, using Co⁶⁰ Gamma radiation source, in the radiation chamber (which is in a form of cylindrical chamber of 14 cm length and 10 cm diameter) with dose rate 4 kGy/h. The samples were irradiated in the dose range of 16 kGy to 300 kGy at UGC –DAE Consortium for Scientific Research, Kolkata Centre, Kolkata. The irradiated samples were characterized by using four analytical techniques like SEM, High Frequency Impedance Analyzer (HFIA), XRD and UV-VIS spectrophotometer to observe the possible modifications that could take place in the PES polymer by irradiation.

Surface morphology (SEM)

The morphology of the surface of polymer was characterized by scanning electron microscope (SEM) using (JEOL, Model No. 3300) operating at 30 kV accelerating voltage. Surface of the samples were coated with a thin layer of gold (3.5 nm) by using the vacuum evaporation technique to minimize sample charging effects due to the electron beam of SEM.

Electrical Response (PSM 1735 High Frequency Impedance Analyzer)

The electrical properties of all samples (virgin and irradiated) were also studied. The dielectric loss and dielectric constant measurements were carried out by using PSM 1735 High Frequency Impedance Analyzer over the frequency range 1 mHz to 10 MHz at ambient temperature. The a.c. conductivity was calculated using the relation

$$\sigma = (2\pi f C_p Dt) A^{-1} (\Omega^{-1} cm^{-1})$$
(1)

The dielectric constant was calculated using the relation $\epsilon = C_p / C_\infty$

where C_p is the capacitance measured using the PSM 1735 High Frequency Impedance Analyzer, *f* is the frequency, *D* is the dielectric loss and $\varepsilon = \varepsilon_0 A/t$, where ε_0 is the permittivity in vacuum and *A* and *t* are the cross- sectional area and thickness of the sample, respectively.

Structural Study (Powder X-Ray Diffractrometer)

The XRD data analyses of PES polymer samples were carried out by Powder X-Ray Diffractrometer (PW-1830) using monochromatic CuK_{α} (8.04 keV and λ = 0.154 nm) radiation.

Optical Response (UV-VIS spectroscopy)

The optical changes were analyzed by UV-VIS spectroscopy (UV 1601 PC) in the wavelength range 190–900 nm to observe the energy gap (E_g) variation with increase of dose.

Results and Discussion

To keep the treatment simple and unambiguous, the results of the present study have been discussed separately.

Morphological Study

To investigate fully the effect of gamma rays, the morphology of PES polymer were studied using scanning electron microscope (SEM). Figure 1(a-e) shows the SEM micrographs of (a) virgin and gamma irradiated (b) 16 kGy (c) 110 kGy (d) 142 kGy (e) 300 kGy PES at magnification 3 kx. From the figures it is observed that the gamma irradiation has caused significant modification on the surface morphology. Figure 1(a) shows SEM micrograph of the virgin sample of PES polymer. Smooth surface of pure polymer sample is observed throughout the scanned region. Figure 1(b) shows the SEM micrograph of gamma irradiated polymer sample at 16 kGy. The formation of blisters size 1 µm has started at some regions. Blistering is defined as the plastic deformation of the irradiated surface layer under cumulative influence of a large (critical) number of gas atoms that evolves near the surface region. Experimental and theoretical endeavours to understand these stages and to evolve the systematic of this phenomenon are set about since its discovery [15], however, very little has been achieved so far. It is observed that the nucleation density of bubbles saturates after a certain radiation dose, and prolonged irradiation results only in their growth [16]. As the radiation dose increases, the growing bubbles at a certain stage of their growth suddenly coalesce to form a disc shaped cavity parallel to the surface. When the gas pressure inside the cavity becomes large, the overlapping material layer is plastically deformed to produce dome shaped structure under the thrust of the gas pressure, known as exfoliation (removal of outer surface) which can also be seen clearly in Figs.1(c-e). Further, SEM micrograph of irradiated sample at 110 kGy is shown in Figure 1 (c). The blisters size is found to increases, may be due to the increasing gas pressure where the gases are accumulating. Figure 1 (d) shows the SEM micrograph of polymer sample irradiated at 142 kGy. The picture is quite different as compared to previous one. A very significant change has been found at this dose (cloudy type of feature with micro voids). Figure 1(e) shows the SEM micrograph of PES polymer sample irradiated at 300 kGy, the blister exfoliation can be seen at the highest dose of gamma radiation.





Figure 1: SEM images of Schematic representation of different stages in blister formation virgin and gamma irradiated PES polymer samples at(a) Virgin (b) 16kGy (c) 110 kGy (d) 142 kGy (e) 300 kGy.

Electrical Response

The a.c. conductivity measurement was performed with in a months of irradiation for virgin and irradiated PES polymer samples at ambient temperature and the corresponding graph is shown in figure 2. A sharp increase in conductivity at high frequencies has been observed in virgin as well as irradiated PES polymer samples as dose increases. The increase in conductivity at a given frequency due to irradiation may be attributed to scissioning of polymer chains and the migration of long lived radicals which are trapped mostly in crystalline region to amorphous region, resulting in an increase of free radicals, unsaturation, etc. An a.c. field of sufficiently high frequency may cause a net polarization, which is out of phase with the applied field. This results in a.c. conductivity and it appears at frequencies greater than that at which traps are filled or emptied [17, 18]. Figure 3 shows a plot of tan δ (dissipation factor) vs log frequency for virgin and irradiated PES polymer samples. It is observed that loss factor decreases as frequency increases moderately with increasing dose. The increase in loss factor with dose may be due to accumulation of stable radiolytic products, resulting in an increase of free radicals unsaturation, etc. Figure 4 shows a

plot of dielectric constant (ϵ) vs log frequency at ambient temperature for virgin and irradiated polymer samples. As evident from the graph, the dielectric constant remains almost constant up to 117 kHz and then decreases at higher frequencies. At lower frequency, the mobility of the free charge carriers is constant and thus the dielectric constant remains uniform. As the frequency increases the charge carriers migrate through the dielectric and get trapped against defect sites and induce an opposite charge in its vicinity. At these frequencies, the polarization of trapped and bound charges cannot take place and hence the dielectric constant decreases. The decrease in dielectric constant at higher frequency can also be explained by Jonscher's power law [17].



Figure 2: The a.c. conductivity vs log of frequency plot for virgin and irradiated PES Polymers.



Figure 3: Variation of tan δ with log of frequency for virgin and irradiated PES polymers.



Figure 4: Plot of dielectric constant vs log of frequency for virgin and irradiated PES polymers.

UV-VIS Spectral Studies

The absorption of light energy by polymeric materials in UV and visible regions involves transition of electrons in σ , π and n- orbitals from the ground state to the higher energy states. Ultraviolet-visible (UV/VIS) spectrophotometer has become an important tool for investigating these electronic transitions. It is used to estimate the value of optical energy gap (E_g) in polymers. The results of absorption studies with UV/VIS spectrophotometer carried out on virgin as well as irradiated PES polymer samples are shown in Figures 5, 6 and 7, respectively. The optical response of virgin and irradiated PES polymer samples at various doses is shown in figure 5. It is clear from the figure that the absorption edge is shifting in non symmetric manner in the range of 415 to 450 nm. This shift may be correlated with the formation of conjugated bonds showing the possibility of formation of carbon clusters. This type of transition occurs in non bonding electron containing compounds and also in aromatic compounds due to bond cleavage and reconstruction [8, 19-24].



Figure 5: UV/VIS spectra of virgin and Co⁶⁰ gamma-irradiated PES polymers.



Figure 6: The dependence of $(\alpha hv)^{1/2}$ on photon energy (hv) for virgin and gamma irradiated PES polymers.



Figure 7: The dependence of $(\alpha hv)^2$ on photon energy (hv) for virgin and gamma irradiated PES polymers.

Urbach Rule

The absorption coefficient near the band edge for semi-crystalline materials shows an exponential dependence on the photon energy (hv) given by the expression known as Urbach formula [25]

$$\alpha(v) = \alpha_0 \exp(hv/E_u)$$
⁽²⁾

The exponential absorption edge (known as Urbach's rule) observed in most materials is interpreted in terms of thermal fluctuations in the band-gap energy. In eq. 2, α_0 is a constant, E_u is an energy which is interpreted as the width of the tail of localized state in the forbidden band gap, v is the frequency of radiation and h is Planck's constant. The origin of E_u is supposed to be due to thermal vibrations in the lattice [22]. The logarithm of the absorption coefficient $\alpha(v)$ was plotted as a function of the photon energy (hv) for irradiated PES polymer samples with different doses of gamma rays as shown in Figure 8. The value of the Urbach energy E_u in each case was calculated by taking the reciprocal of the slope of the linear portion in the lower photon energy region of these curves and is listed in Table 1 and also shown in Figure 9. The increases in Urbach's energy in the case of PES polymer may be due to decreases in the amorphous nature of the polymer at higher doses.



Figure 8: The dependence of natural logarithm of α on photon energy for virgin and gamma irradiated PES polymers.



Figure 9: Variation of Urbach's energy with irradiation doses.

Determination of Band Gap

The absorption edge of semi-crystalline materials gives the measure of the band strength or band gap E_g , and the position of the sharp absorption edge is

$$E_{g} = hc/\lambda_{g}$$
(3)

where h is the Planck's constant, c is the velocity of light and λ is the wavelength. The present polymer obeys the rule of indirect transition as well as direct transition [27]. The equation 4 gives the relationship among the optical band gap, absorption coefficient and incident photon energy as

$$\alpha (hv) = B (hv - E_g)^n / hv$$
(4)

where hv is the photon energy of the incident photons, B is constant, E_g is the value of the optical energy gap between the valance band and the conduction band, *n* is the power, which characterizes the electronic transition, whether it is direct or indirect during the absorption process in the k-space. Specially, *n* is 1/2, 3/2, 2, and 3 for direct allowed, direct forbidden, indirect allowed and indirect forbidden transitions, respectively.

The variation of $(\alpha hv)^{1/2}$ with photon energy (hv) for PES polymer is shown in figure 6. The value of band gap has been determined by taking the intercept on the x-axis. It is clear that the value of optical indirect band gap decreases at 16 kGy and increases at 142 kGy and again decreases from 142 kGy to 300 kGy. The variation of energy gap with the irradiation dose is provided in Table 1.

The variation of $(\alpha hv)^2$ with photon energy (hv) for PES polymer is shown in figure 7.

It is clear that like the value of optical indirect band gap the value of optical direct band gap also decreases at 16 kGy and increases at 110 kGy and again decreases from 142kGy to 300 kGy. Taking into account the linear portion of the fundamental absorption edge of the UV/VIS (shown in Figures 5) such plots have been presented in Figures 6 and 7, respectively. By extrapolating the straight parts of this relation to the hv axis, indirect and direct band gaps have been determined for virgin and gamma irradiated PES polymer samples. The different transition energies are shown in figure 10 and are also provided in Table 1. This, in turn, clearly indicates the simultaneous existence of indirect and direct band gaps in the PES polymer samples with decreasing tendency at higher gamma ray dose. These results confirm that the irradiation produces faults in PES polymer structure (band rupture, free radical, etc.) which consequently increase the electronic disorder inducing the creation of a permitted state in the forbidden (interdict) band or the deformation of valence band.



Figure 10: Plots for transition energy (eV) as a function of the gamma absorbed dose (kGy).

Furthermore, the values of the indirect band gap have been found to be lower than the corresponding values of direct band gap as given in Table 1. The coexistence of indirect and direct band gaps in the PES polymer has not been reported so far, but such results have been observed in some other materials also [28-30].

The number of carbon atoms per conjugation length N (i.e. the number of carbon hexagonal atoms in a cluster) can be calculated by Robetson relation Robertson *et al.* [31].

$$N = 2\beta \pi / E_g$$
(5)

Here 2β gives the band structure energy of a pair of adjacent π sites. The value of β is taken to be -2.9 eV as it is associated with $\pi \rightarrow \pi^*$ optical transitions in -C=C-structure. A shift in the absorption edge can be attributed to an increase of the conjugation length. The number of carbon atoms per conjugation length is found to increase up to 7 at the highest dose i.e., 300 kGy. The increase of unsaturated conjugation length in polymer samples results in their discoloration i.e., the PES polymer samples turn light yellow from transparent with increasing gamma radiation dose. Gamma radiation can induce the formation of unsaturated bonds in polymers and contribute to coloration, since as a rule, the conjugated double bonds are responsible for the colour of most organic compounds [32].

From the optical transmission spectra as shown in Figure 11 of virgin and irradiated polymer samples some variation in the transmission window was observed due to gamma irradiation with increasing dose in all PES polymer samples. The virgin and irradiated polymer samples have low UV cut off range 380-400 nm. In terms of percentage of transmission, we observe that for virgin and irradiated polymer samples the optical transmission has been increased in visible as well as infrared regions. The increase of optical transparency indicates the decreases of band gap with increasing dose. The small peaks in the near infrared region are the characteristic absorption inherent to the individual dose of polymer samples.



Figure 11: Gamma radiation dose dependence of optical transmittance spectra virgin and Irradiated PES polymer.

Powder XRD Studies

The XRD pattern of virgin and gamma irradiated at different doses of radiation (16, 110,142, and 300 kGy) PES polymer samples are shown in Figure 12 (ABCDE). Figure 12.A. shows that the virgin polymer is semi-crystalline in nature, which has three crystalline peaks at $2\theta = 44^{\circ}$, 51° and 72° and an amorphous hump at $2\theta = 20^{\circ}$. This semi-crystalline nature of polymer arises due to the systematic alignment of polymer chain folding [33]

Figure 12 (B) shows that the XRD pattern of gamma irradiated PES polymer sample, the overall peaks intensity decreases at 16 kGy. Figure 12 (C, D) shows that peak intensity increases at 110kGy and 142kGy. Again peaks intensity decreases at highest doses shown in Figure 12 (E).



Figure 12: X-ray diffraction pattern of virgin and gamma irradiated PES at various doses (16,110, 142 and 300 kGy).

It is concluded that the peak intensity has recovered with increase of dose. The full width at half maximum (FWHM) is generally associated with the crystallite size which can be obtained from Scherrer's formula

$$L = K\lambda/\beta \cos\theta \tag{6}$$

where K=1, $\lambda = 1.54$ Å and $\beta = FWHM$ in radian	
The strain (ϵ) value can be evaluated using the relation	
$\varepsilon = (\beta Cos \theta)/4$	(7)

The dislocation density (
$$\delta$$
) may be calculated by using the formula
 $\delta = 15 \epsilon / (a \times D)$ (8)

Various observed and calculated parameters, such as the peak intensity, strain, dislocation, FWHM and crystallite size with irradiation dose are provided in Table 2. The results indicate the occurrence of recrystallization in PES polymer samples due to increase of gamma irradiation dose.

Table 1: The variation of the band gap energy and Urbach's energy in the pristine and gamma irradiated PES, alongwith the number of carbon atoms (N) per conjugated length.

γ-radiation	Absorption	Direct Band	Indirect Band	Urbach's	(N)	(N)
dose (kGy)	$edge(\lambda_g)$	Gap Energy	Gap Energy	energy	Direct	Indirect
	(nm)	(eV)	(eV)	(eV)		
0	421	2.92(±0.10)	2.60(±0.10)	0.56	6~	7~
16	416	2.89(±0.10)	2.45(±0.10)	0.67	6~	7~
110	428	2.94(±0.10)	2.75(±0.10)	0.41	6~	7~
142	428	2.93(±0.10)	2.72(±0.10)	0.43	6~	7~
300	416	2.72(±0.10)	2.71(±0.10)	0.61	7~	7~

Table 2: X-ray diffraction spectra of Co⁶⁰ gamma irradiated PES polymer at different doses.

S.No.	Angle of	Dose	FWHM	Crystallite	Strain ε	Dislocation	Peak	d-
	$Peak(2\theta)$	(kGy)	(β)	size (L)	$=(\beta Cos\theta)/4$	δ= (15 ε/ a	intensity	Value
				(A	, v	x D)		(A)
						-		
1^{st}	44	0	0.400	3.74	0.10	0.087	12	2.0582
Peak								
1.								
2.	44	16	0.560	2.66	0.14	0.171	07	2.0505
3.	44	110	0.240	28.0	0.06	0.007	12	2.0567

	-							
4.	44	142	0.320	14.60	0.08	0.017	22	2.0571
5.	44	300	0.320	14.60	0.08	0.017	18	2.0551
2^{nd}	51	0	0.800	1.921	0.20	0.339	07	1.7796
Peak								
6.								
7.	51	16	0.480	7.390	0.12	0.053	04	1.7830
8.	51	110	0.640	2.400	0.16	0.217	05	1.7790
9.	51	142	0.320	4.802	0.08	0.054	14	1.7812
10.	51	300	0.640	7.390	0.16	0.071	06	1.7783
3rd	72	0	0.240	1.472	0.06	0.026	13	1.2987
Peak								
11.								
12.	72	16	0.320	5.355	0.08	0.048	06	1.2989
13.	72	110	0.320	5.355	0.08	0.048	10	1.2649
14.	72	142	0.320	5.355	0.08	0.048	22	1.2983
15.	72	300	0.640	2.679	0.16	0.195	15	1.2967

Conclusions

The morphology, electrical, optical and structural properties of Polyethersulfone (PES) Polymer samples under gamma radiation source of Co⁶⁰ have been studied by SEM, High Frequency Impedance Analyzer, UV-VIS spectroscopy and XRD techniques. The following conclusions have been drawn:

The morphology study suggests the blisters (size =1 μ m) formation on the surface of the PES polymer samples. They ascribe to the rupture of chemical bonds and the formation of low molecule gases. These gases are accumulated inside the polymer at a depth where maximum radiation damage takes place. When the pressure of accumulated gas crosses the mechanical strength of PES samples, it deforms and results as blisters.

The results of ac. conductivity, dielectric loss (δ) and dielectric constant (ϵ) of PES polymer samples shown an increasing trend with an increase in gamma ray radiation doses.

The optical band gap (E_g) , and Urbach energy (E_u) were determined from the optical absorption spectra. It is concluded that the values of indirect band gap are lower than the corresponding values of direct band gap in virgin and gamma irradiated PES Polymer samples. Also the band gap (E_g) decreases with the increase of gamma irradiation dose, due to the photo-degradation of PES polymer and the formation of defects and clusters in the material, while the cluster size (number of carbon atoms N per conjugated length) increases with the increase of absorbed dose.

Optical transmittance has been found to increase in visible as well as infrared regions. The increase of optical transparency indicates that it depends greatly on energy band gap and radiation dose on polymer.

The average of peak intensity is found to increase with increasing dose. It shows that crystalline nature of the PES polymer increases. The crystallite size is also found to increase with increasing dose. The peak intensity, strain (ϵ) and dislocation (δ) are found to show recovery characteristics with increasing dose.

In the experimental studies such as SEM, High Frequency Impedance Analyzer, XRD and UV-VIS measurements, the observed modifications of Polyethersulfone (PES) polymer were found with increasing radiation dose. They attribute to scissioning and crosslinking of the polymer chains, leading to production of free radicals and unsaturated bonds in the polymer matrix upon irradiation.

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