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SURFACE MORPHOLOGICAL STUDY AND STRUCTURAL ANALYSIS OF POLYETHERSULFONE (PES) POLYMER IRRADIATED WITH 1.25 MEV GAMMA RAYS

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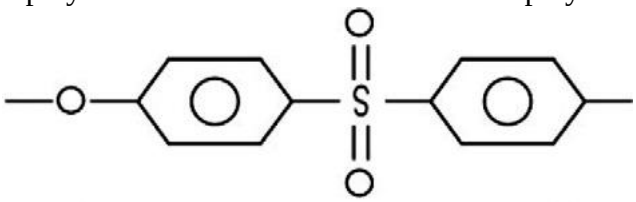
ABSTRACT
In the present paper we have studied the surface morphological changes induced by the gamma ray irradiation with energy of 1.25 MeV has been carried out using scanning electron microscopy (SEM) technique and the structural properties of the same polymer has been carried out using X-ray diffraction pattern (XRD).The morphology study shows that blisters of size 1 μm were observed on the surface of PES polymer due to 1.25 MeV gamma ray irradiated at ambient temperature, the effect of gamma irradiation and 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 Which indicates that amorphousness of the polymer increases due to irradiation but At the highest dose level of 300 kGy, the polymer shows a recovery characteristic which indicates that crystinity increases but the crystallite size also shows a recovery characteristic in the studied range of doses.

KEYWORDS: PES, SEM, XRD, Gamma radiation

INTRODUCTION
Polymer is one of the important classes of space materials in addition to metals, alloys and ceramics. On account of their rapid development of the physics and chemistry of polymers during last five decades a process of "polymer engineering" like "genetic engineering" has become a success story of practical applications. Polymer in addition to their inherent economical superiority can have vast variety of physical and chemical properties, wide range of mechanical parameters, flexibility lightness, optical transparency, easy processing etc. Which have made the polymer science as an attractive field. Polyethersulfone (PES) is a heat-resistant, transparent, amber, non-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 200ºC. 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 cause the photons to penetrate the material, breaking the polymer chains and creating free radicals. These free radicals can also recombine to create crosslinks between the adjacent molecules. Crosslinked materials have been found to improve their long term performance.

Different studies of effect of ion irradiation on polymers, reveal a variety of modifications of structural, of 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. 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. 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 1.25 MeV gamma ray induced modifications in PES polymer. The molecular structure PES polymer is shown below



Surface morphology (SEM)
The morphology of the surface of polymer was characterized by scanning electron microscope (SEM) using (JEOL, Model No. 3300) operating at 25 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.
EXPERIMENTAL DETAILS-

1. 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.

1.1 Morphological Study
To investigate the effect of gamma irradiation, On the surface morphological changes in 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 of polymer. Smooth surface of pure PES polymer is observed throughout the scanned region. Figure 1. (b) Shows the SEM micrograph of gamma irradiated polymer sample at 16 kGy. The blisters formation of size 1 μm has started at some regions may be due to the radiolytically evolved gases accumulating within the amorphous zone. The evolved gases sweep through crystalline zone but remaining portion is uniform as before. Exfoliation (removal of outer surface) of the surface can also be seen clearly. Next SEM micrograph of irradiated sample at 110 kGy is shown in Figure 1 (c). The blisters size is found to increase 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

1.2 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 2 (ABCDE). Figure 2.A. shows that the virgin polymer is non-crystalline in nature, which has three crystalline peaks at $2\theta = 44^{o}$, 51^{o} and 72^{o} and an amorphous hump at $2\theta = 20^{o}$. This semi-crystalline nature of polymer arises due to the systematic alignment of polymer chain folding [14]
Figure 2 (B) shows that the XRD pattern of gamma irradiated PES polymer sample, the overall peaks intensity decreases at 16 kGy. Figure 2 (C, D) shows that peak intensity increases at 110kGy and 142kGy. Again peaks intensity decreases at highest dose shown in Figure 2 (E).
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

$$L = K\lambda/\beta \cos\theta \quad (1)$$

where K=1, λ = 1.54 Å and β = FWHM in radian

The strain (ε) value can be evaluated using the relation

$$\varepsilon = (\beta \mathrm{Cos}\theta)/4 \quad (2)$$

The dislocation density (δ) may be calculated by using the formula

$$\delta = 15\,\varepsilon/(a \times D) \quad (3)$$

The quantity of increase and decrease of peak intensity, strain, dislocation, FWHM and crystallite size with doses are provided in Table 1. The results obtained in relation to the peak intensity, FWHM, strain ε, dislocation δ and the crystallite size results indicate the recrystallization of polymer due to gamma irradiation with increasing dose. This is in conformity with the earlier result [15].

CONCLUSIONS

The surface morphological and structural properties of Polyethersulfone (PES) Polymer samples under 1.25 MeV gamma radiation source of Co^{60} have been studied by SEM and XRD techniques. The following conclusions have been drawn:

- The morphology study suggests the blisters (size =1 μm) 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.

- An initial decrease in the crystallinity and a recrystallization characteristic has been observed with increasing dose. The peaks intensity, FWHM, strain ε, dislocation δ crystallite size also shows recrystallization with increasing dose.

In the experimental studies such as SEM, and XRD 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 due to irradiation.

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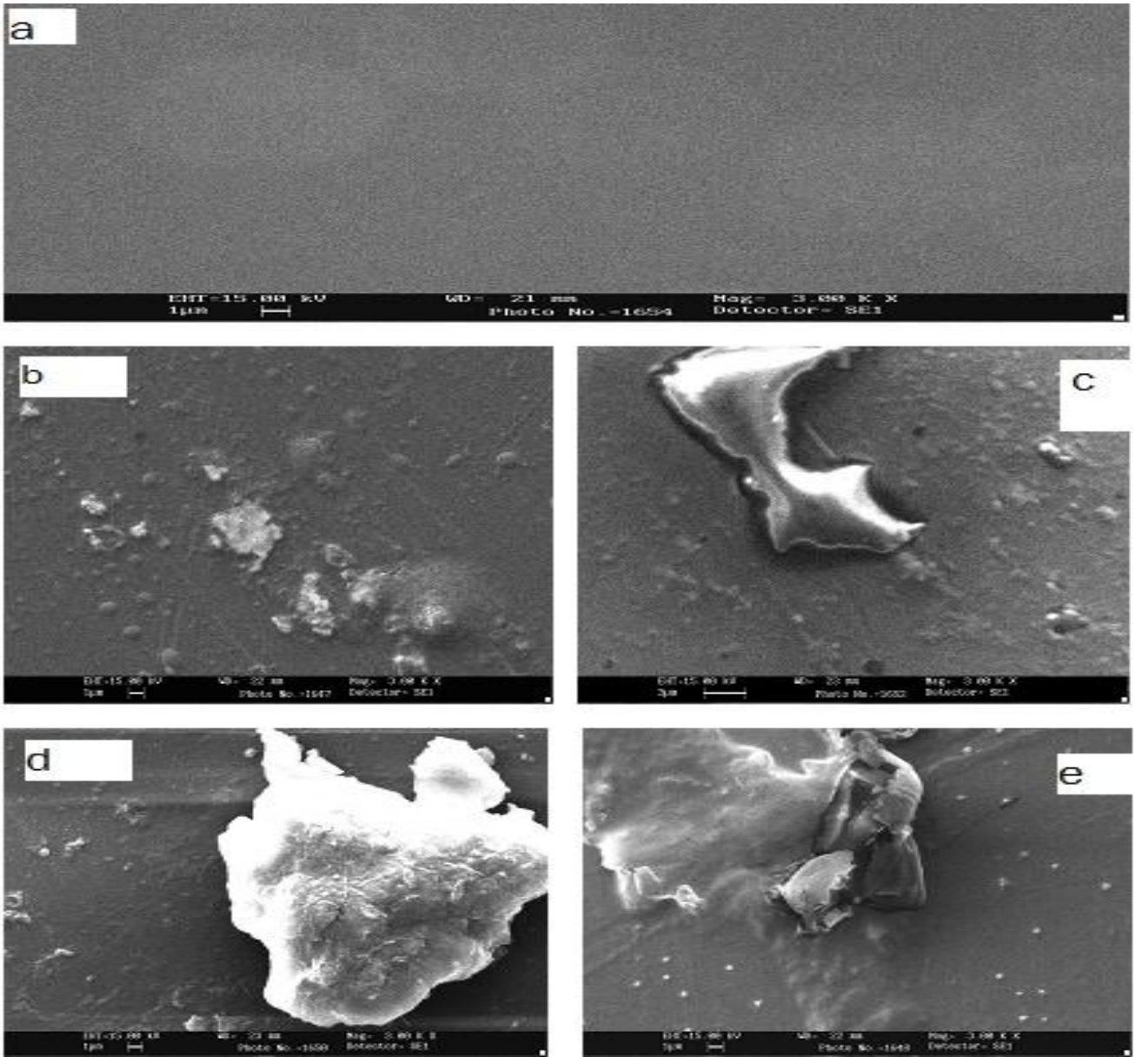

Fig. 1(Morphological changes induced by gamma Irradiation)

(X ray Diffraction Pattern Of PES Polymer Samples)

Table 1: X-ray diffraction spectra of 1.25 MeV gamma irradiated PES polymer at different doses

S.No.	Angle of Peak(2θ)	Dose (kGy)	FWHM (β)	Crystallite size (L) (A	Strain ε =(βCosθ)/4	Dislocation δ= (15 ε/ a x D)	Peak intensity	d-Value (A)
1.	44	0	0.400	3.74	0.10	0.087	12	2.0582
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
6.	51	0	0.800	1.921	0.20	0.339	07	1.7796
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
11.	72	0	0.240	1.472	0.06	0.026	13	1.2987
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

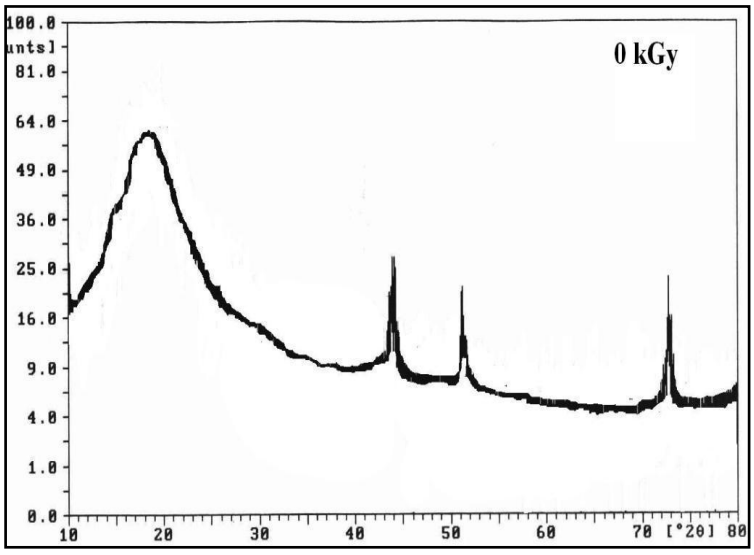

Fig 2A

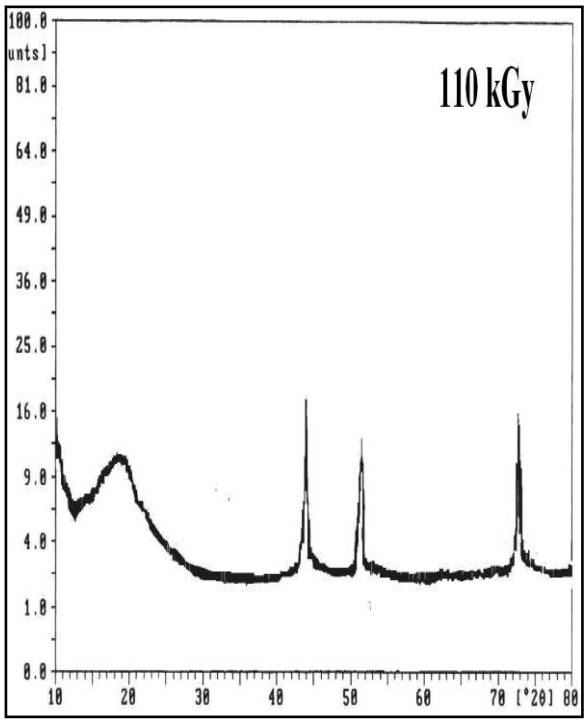

Fig 2B

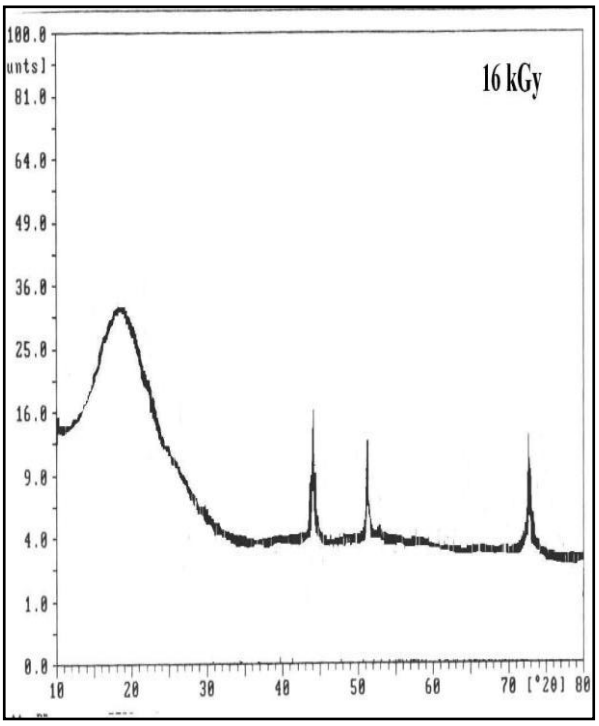

Fig 2C

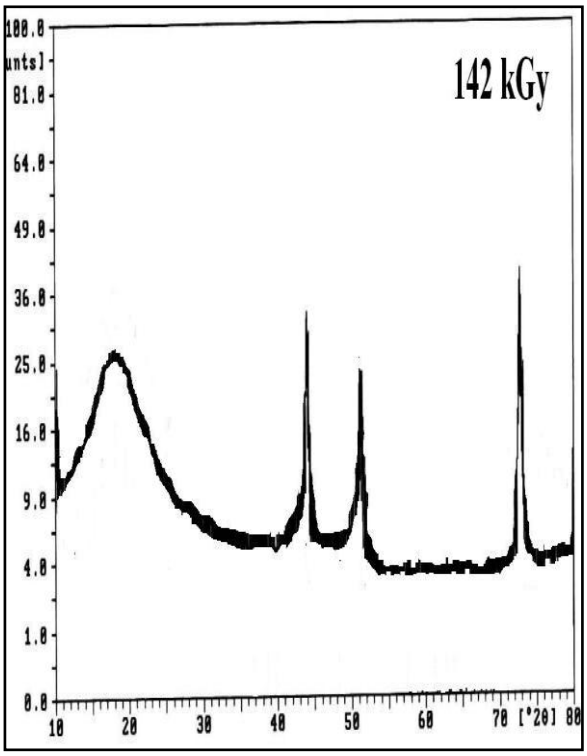

Fig 2D

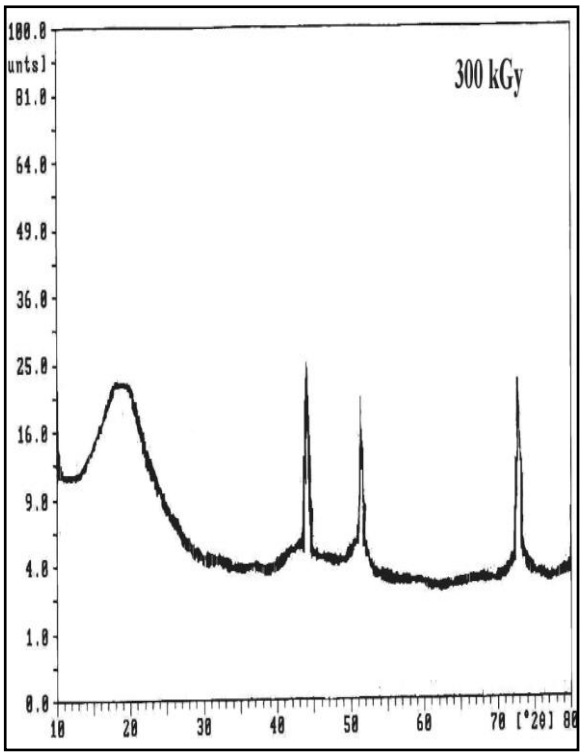

Fig 2E