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Electron beam and gamma ray irradiated polymer electrolyte films: Dielectric properties

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ABSTRACT

In this study, polymer electrolyte films were irradiated with electron beam (EB) and Gamma ray (GR) at 50 and 150 kGy. The induced chemical changes in films due to irradiations have been confirmed from the Fourier Transform Infra red (FT-IR) spectra. The X-ray Diffractometry (XRD) results show that crystallinity decreases by ~20% in EB and ~10% in GR irradiated films respectively compared to non-irradiated film. The micro structural arrangement was investigated by Scanning Electronic Microscopy (SEM) and the images reveal that there is a substantial improvement in the surface morphology in irradiated films. The real (ϵ') and imaginary (ϵ'') dielectric constant and AC conductivity are found to increase with increase in irradiation dose. Improved dielectric properties and conductivity (1.74×10^{-4} & 1.15×10^{-4} S/cm, respectively, for EB and GR irradiated films at room temperature) after irradiation and it confirm that EB and GR irradiation can be simple and effective route to obtaining highly conductive polymer electrolytes. From this study it is confirm that EB is more effectiveness than GR irradiation.

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1. Introduction

In recent years, polymer electrolytes have been attracting scientific and technological importance because of their potential applications in many areas such as Li-ion polymer solid state batteries, super capacitor, electro-chromic devices, etc. Wright and Fenton first proposed the idea of preparation of polymer electrolytes in 1973, but its technological significances were achieved and appreciated by Armand et al. a few years later [Armand, Chabango, and Duclot \(1998\)](#). As advanced technology keeps on developing every day, the demand for polymers is raising continuously, in particularly

polymer electrolyte because of its use in various promising applications [Khan and Qureshi \(1999\)](#). PEO based polymer electrolytes are still among the most extensively studied polymer ionic conductors due to fast ion transport, and they easily associate with other ions especially inorganic alkali and transition salts [Chiu, Chen, Kuo, Huang, and Chang \(2004\)](#). In particular to Cadmium chloride (CdCl_2)-doped PEO has attracted considerable attention because of fine tune their fundamental physical properties by simply varying the cluster size and composition makes them highly attractive for a variety of possible applications [Liu et al. \(2001\)](#). The main advantage of polymer electrolytes is the easy preparation of

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films of desired size and shape and its ability to form proper electrode/electrolyte contact in electro-chemical devices. These materials exhibit relatively complex multi-phase behavior in crystalline as well as amorphous phase. In the last few years, considerable efforts have been devoted to develop the volume of amorphous in polymeric materials, for which a number of different techniques such as doping, blending, plasticization, and ionizing radiation were used to reduce the crystallinity of polymer. In this case, EB and GR radiation methods were used for modify the material phase. Irradiation plays a prominent role in the modification of the physical properties of polymers because the radiation process directly provides the energy to the system. If ionizing radiation is passed through the matter, the energy deposit in the material causes irreversible changes in the macromolecular structure of the target material. Hence, the application of radiation in polymer technology has great importance to achieve desired modification in the physical properties polymeric materials.

Therefore, modification of polymer has become an important research area, mainly in industrial applications such as wire and cable, electronic devices, medical, and marine [Drobny \(2002\)](#), [Chmielewski, Haji-Saeid, and Ahmed \(2005\)](#) and [Sperling \(1992\)](#). On a large commercial scale, various radiation processing techniques such as EB, GR, UV, and X-rays or laser beams have demonstrated to be very effective means of improving the end-use properties of various polymers. In the present study, EB and GR irradiations with various dosages were used to expose the polymer electrolyte film, it interact with polymer chain could be undergo crosslink or chain scission leading to alteration in the macromolecular structure, which is most effective because no special additives are required. This structural modification in the irradiated polymer electrolyte is directly influence on physical properties; in this case the dielectric permittivity and electrical conductivity were observed increased with increasing the dose. The changes strongly depend on the physics and chemistry of the absorbed materials; as well as the chosen radiation energy and dose. The radiation effects may cause occurrence of ionization or excitation of the electrons in atoms, and displacement of atoms from their original site to adjacent lattice sites of the solid permit charges should be become free or loosely bonded to some trapping centers elsewhere in the material structure as a result cross-linking or chain scission [Bouffard, Balanzat, Leroy, Busnel, and Guevelou \(1997\)](#) and [Mujahid et al. \(2004\)](#). The presence of trapping charge within the material was influenced by the induced radiation, thus of the increase in the transport property [Zahran, Kander, Hegazy, and Kassem \(1993\)](#). Also, it is observed that alteration in the functional group of polymer causes a change in the physical properties of polymer films [Fink \(2004\)](#) and [Dworecki, Hasegawa, Sudlitz, and Wasik \(2001\)](#). In the past few years, the effects of EB and GR on polymers have been investigated individually and some of the results presented in the literature state that irradiation may reduce crystallinity, induce cross-linking, and produce a unsaturated carbonyl groups [Cleland, Parks, and Cheng \(2003\)](#), [Mishra et al. \(2001\)](#), [Perloni, Magistris, Chiodelli, Faucitano, and Buttafava \(1991\)](#), [Nasef, Saidi, and Dahlan \(2002\)](#) and [Shah et al. \(2008\)](#).

Dielectric measurements are performed to know the information about the molecular dynamics and the charge transportation mechanism in Polymer electrolytes films. Polymers with high dielectric permittivity have received increasing interest in recent years due to their potential applications in embedded capacitors, gate dielectrics and charge storage applications as they provide advantage in size and cost reduction. Literature survey reveals that, the dielectric studies on ionically conducting solids although important and valuable from scientific point of view, has not been studied extensively due to the limitations and complexity in data analysis.

Therefore, gaining a better understanding of the radiation influence on the ion dynamics and segmental motion of charges will facilitate to design new devices for required purposes. In addition, it is important to understand how the relaxation dynamics is affected by the dose. Thus, the effect of radiation on the physical and structural properties of such materials is required to be studied particularly, when such devices are being used in the radiation environment. The intention of this study is to investigate the effect of EB and GR on the dielectric properties of polymer electrolyte films.

2. Experimental

2.1. Materials

The Polyethylene oxide (PEO, $M_w = 5 \times 10^6$) powder was procured from M/s Sigma Aldrich Chemicals USA, Cadmium Chloride ($CdCl_2$) (M.W. 201.32) was obtained from SDFCL, Mumbai, India, and methanol (acetone free) was obtained from the NICE laboratory.

2.2. Methods

2.2.1. Polymer electrolyte film preparation

The film was prepared using the solution-cast technique. PEO: $CdCl_2$ (75:25%) was dissolved in methanol (CH_3OH) and stirred for approximately 6–8 hours at room temperature to obtain a homogenous viscous mixture. The stirred mixture was cast onto polypropylene dishes and allowed to evaporate at room temperature. The thickness of the film was measured using a Mitutoyo Dial thickness gauge and was determined to be 0.25 mm.

2.2.2. Irradiation source

The polymer electrolyte films were irradiated using electron beam accelerator with 8 MeV-20A current, pulse repetition rate of 50 Hz, pulse width 2.3 μs , and varying dose of 50, and 150 kGy in the air. They were also irradiated with gamma ray (^{60}Co source) at ambient temperature using a gamma cell at fixed dose rate of 9.5 kGy/hour and varying doses of 50 and 150 kGy.

2.2.3. FT-IR study

The FT-IR spectra of the non-irradiated and irradiated films were recorded in the transmission mode using model AIM-8800 FTIR Spectroscopy in the KBr medium. The spectrum of the transmittance as a function of wave number in the range

of $3500\text{--}500\text{ cm}^{-1}$ was recorded. The variation in transmittance before and after irradiation was compared, and the peaks were analyzed to study the chemical change.

2.2.4. XRD study

XRD patterns for non-irradiated and irradiated films were recorded (Rigaku miniflex 600-bench top X-ray diffractometer) at a scanning rate of $2^\circ/\text{min}$ in the 2θ range of $10\text{--}30^\circ$ to study the changes in the material phase.

2.2.5. Scanning electron microscopy

SEM images of non-irradiated and irradiated polymer electrolyte films were obtained using the SEM (JEOL Model JSM, 390LV model).

2.2.6. Dielectric measurements

The conductivity and dielectric parameters were measured using PC-based Precision Impedance Analyzer (Model 6500B) with PID temperature controlled having a frequency range of $40\text{ Hz}^{-1}\text{ MHz}$ and temperature range of $303\text{--}323\text{ K}$.

3. Results and discussion

3.1. FT-IR analysis

The FT-IR spectra of non-irradiated, EB and GR irradiated polymer electrolyte films are shown in Fig. 1. It reveals that some peaks intensity changed in the electron beam and gamma ray irradiated films in comparison with non-irradiated film. The bands at 940 and 1465 cm^{-1} is due to the CH_2 bending vibration, which correspond to the crystalline phase seem to decrease with irradiation dose. This may be attributed to the destruction of crystalline lamellar structure of polymer. The intensity of the bands at 1654 and 1100 cm^{-1} respectively represents the ketonic carbonyl groups $\text{C}=\text{C}$ and $\text{C}-\text{O}-\text{C}$ group.

The change in intensity and transmittance of these peaks indicates the occurrence of chain scission and cross-linking.

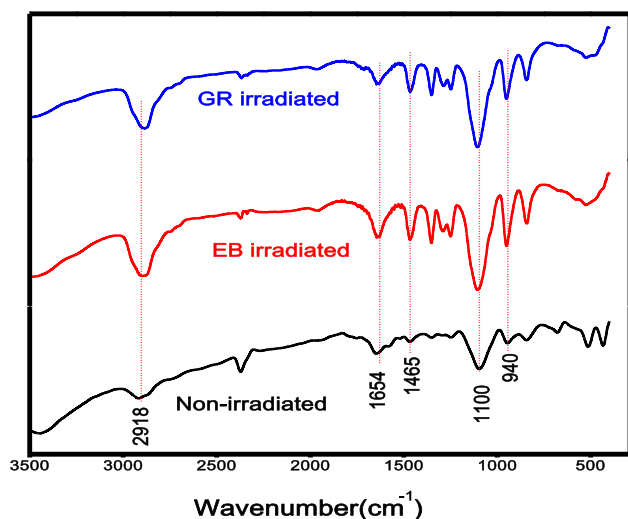


Fig. 1 – FT-IR spectra of (a) Non-irradiated, (b) Electron Beam, and (c) Gamma Ray irradiated films.

The peak at 2918 cm^{-1} is assigned the $\text{C}-\text{H}$ stretching; the increase in peak intensity in the irradiated films clearly indicates that the $\text{C}-\text{H}$ bond was affected because of the breaking cleavage of H -bonds. This might be attributed to the breakage of chemical bonds or formation/emission of low molecular gases and radicals due to irradiation. The peak intensity is increased and transmittance is decreased in EB irradiated films with respect to that of gamma irradiated films. Also, these results suggest that the affects are more pronounced in electron beam compared to that of gamma ray irradiation.

3.2. X-ray diffraction analysis

XRD analysis gives the effect of the radiation dose on the structural phase of polymer electrolyte films. The X-ray diffraction spectra of non-irradiated and irradiated films are shown in Fig. 2. The XRD spectrum of a non-irradiated film shows partly crystallinity and amorphous phase. The changes in the structural parameters after irradiation affect the crystalline phase of the polymer; estimated values are cited in the Table 1. The increase in FWHM (β) and peak intensity (I) is clearly represent the decrease in crystallinity.

However, it is interesting to note that the crystallinity decreased by $\sim 20\%$ in EB irradiated films and by $\sim 10\%$ in GR irradiated films compared to non-irradiated film. These results suggest that EB irradiation is more effective than gamma ray irradiation. Similar effects have been observed from dielectric permittivity, AC conductivity, and FT-IR results. Generally, the intensity and full-width half-maximum (FWHM) are found to increase and the crystallite size decrease. The average crystallite size (L) has been calculated using the Scherrer formula Cullity and Stock (2001). The variation of crystallite size, percentage of crystallinity, and average inter-chain separation are shown in Table 1. After irradiation, the average crystallite size, percentage of

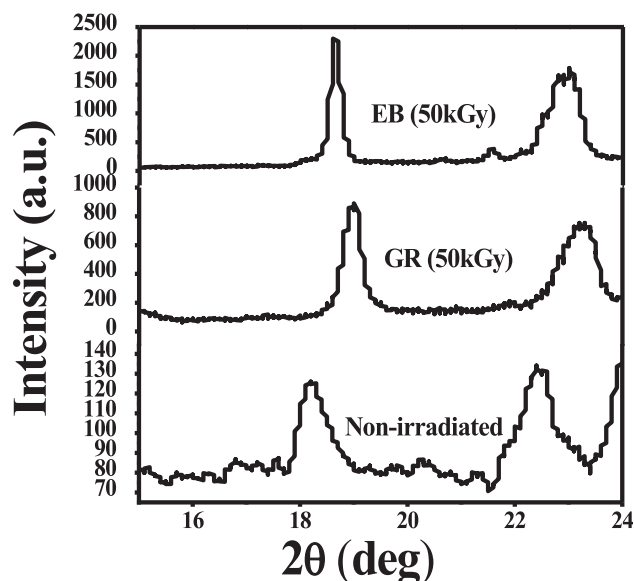


Fig. 2 – X-ray spectra of (a) Non-irradiated, (b) EB, and (c) GR irradiated films.

Table 1 – Structural parameters for non-irradiated, EB and GR irradiated films.

Sample	2 θ	d(A)	β	L (nm)	R (nm)	I	X _c (%)
Non-irradiated	18.19	4.87	0.2817	28.19	31.28	126.1	47.2
EB (50 kGy)	18.60	4.76	0.2885	27.53	30.57	2308.2	27.3
GR (50 kGy)	18.99	4.67	0.4915	16.16	29.94	890.9	35.0

crystallinity and average inter-chain separation are seen to decrease for the prominent peak (first peak) due to main chain scission and cross-linking effect. These changes leads to disorder in the irradiated films thus enhance the amorphous content in the irradiated films.

3.3. Surface morphology study

The SEM image analysis of the films provides the structural information of their topographical and chemical composition. The surface morphology of non-irradiated, EB and GR irradiated polymer electrolyte films are shown in Fig. 3. It can be seen that the surface of the non-irradiated film is smooth and the spherulites structures are clearly visible as well as minor surface cracks.

In irradiated films, it is clear seen that there is substantial improvement in the morphology i.e., the surface gets rough, increases in cracks depth and the spherulites structures almost disappear as a result of which the amorphous region would be dominant. The surface morphology seems to be more uniform in EB than GR irradiated films and more agglomeration is seen in GR irradiated films. It is seen that the cross-linking effect is more in EB irradiation than GR irradiation because of more electronic energy loss. These changes clearly indicate displacement of the polymer molecules under the impact of the incident irradiation. These obtained results suggest that the polymer get degraded due chain scissioning and cross-linking. Hence, these improvements favor to the ionic conductivity of films. These results well correlated with the XRD and dielectric results.

3.4. Dielectric permittivity study

One of the important electrical properties for a material is its dielectric permittivity (or relative permittivity, which is generally referred to as the dielectric constant). For most materials, the dielectric permittivity is independent of the

electric field strength below a certain critical level, at or above which the carrier injection into the material becomes important.

The dielectric permittivity depends strongly on the frequency of the applied alternating electric field or the rate of the change of the time varying field. Fig. 4 shows the frequency dependence of dielectric permittivity (ϵ' & ϵ'') at different temperatures for different irradiation dosage. The dielectric permittivity is found to be increase with increasing temperature and decrease monotonically with increasing frequency perhaps due to the tendency of the induced dipoles in the macromolecules to orient themselves in the direction of the applied field in the lower frequency. However, at high frequencies, the induced dipoles begin to lag behind in the applied field or the alignment dipoles with the applied oscillating field gradually failed as the frequency is increased and hence, the dielectric permittivity maintain constant [Reicha, El Hiti, El Sonbati, and Diab \(1991\)](#). This is a typical characteristic of the disordered materials. The dielectric permittivity ϵ' shows increase with temperature is emphasizing that whenever the temperature rises the dipoles orientation is facilitated and relaxed producing increased dielectric permittivity because of the decrease in the average structural time.

Also, it can be interpreted that the dielectric dispersion in ϵ' is more pronounced at low frequency(1 kHz) as the temperature increases, which may be the total polarization in polar dielectrics (dipolar, interfacial, and ionic polarization) [Saqan and Zihilif \(2003\)](#), [Fanggao, Saunders, and Lanbson \(1996\)](#), [Prasad and Varma \(2005\)](#). Similar results were observed by [Brosseau, Queffelec, and Talbot \(2001\)](#), [Kuo and Chang \(2001\)](#), [Saravanan, Anantharaman, and Venkatachalam \(2006\)](#). Fig. 4 (b, d & f) shows the temperature dependence of the dielectric loss (ϵ'') with frequencies. The ϵ'' shows an increase with the increasing temperature due to charges acquire more strength as a result of which become more flexible, hence dielectric loss increases with temperature. It can also be interpreted that the rapid response of the polar dipoles

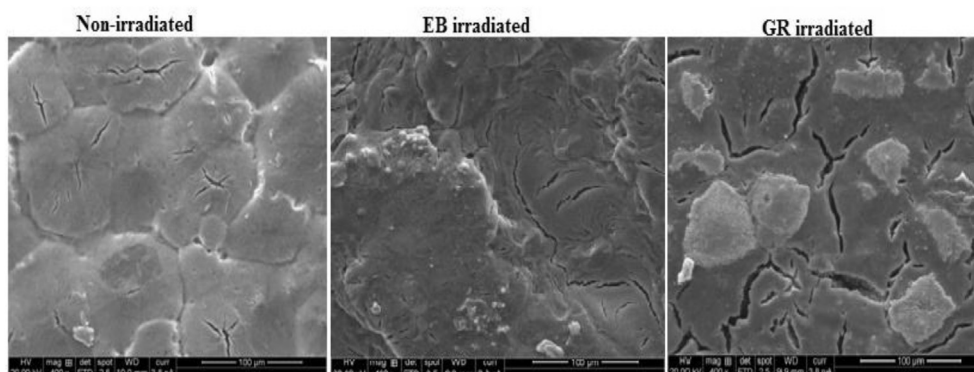


Fig. 3 – SEM images of Non-irradiated, EB and GR irradiated polymer electrolyte films.

subjected to an applied external field, i.e., increasing temperature decreases in the dipole relaxation time. From the figures, it is clear that ϵ' and ϵ'' increase with increasing temperature. This can be attributed to the temperature induced relaxation of the rigid structure, which is responsible for an increase in the dipole orientation.

The dielectric permittivity increases with the increase in the irradiation dose as shown in Fig. 5 (a & b). The dielectric behavior of the polymer is due to the presence of an appreciable number of defects in the form of chain scission, cross-linking, discontinuity, bending, breaking of bonds, etc. in the molecular chains. Upon irradiation, the increasing defects results in the formation of a greater number of dipoles in the polymer matrix that governs its dielectric properties. From Fig. 5, compare the influence of EB and GR irradiation on the dielectric permittivity of polymer electrolyte films and it is

clearly seen that EB effects is more pronounced than of GR irradiation because radiation tolerance level for EB exposure may be slightly higher than for GR exposure. Due to the higher dose rates and shorter exposure times of high energy EB irradiation, which have been shown to form high concentration of defects, free radicals, unsaturated bonds. Hence, favours the high dielectric permittivity than GR irradiation.

3.5. AC conductivity study

Fig. 6 shows the frequency dependent AC conductivity of non-irradiated and irradiated polymer electrolyte films. The increase in AC conductivity with frequency and temperature reveals that there may be more and more free charges are involved in the hopping process through the defective sites along the macro polymer chains.

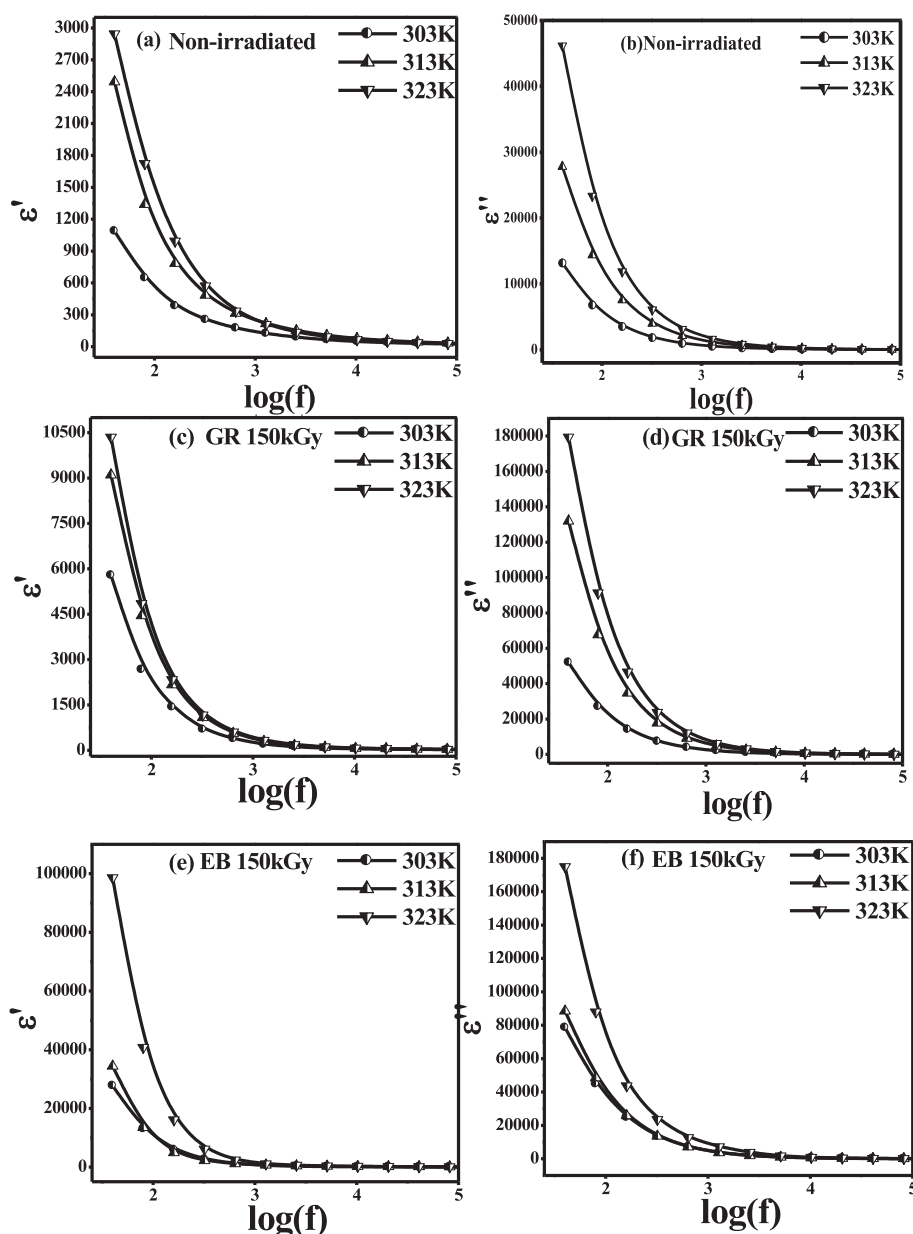


Fig. 4 – Variation of ϵ' & ϵ'' with frequency at different temperatures for EB and GR irradiated films.

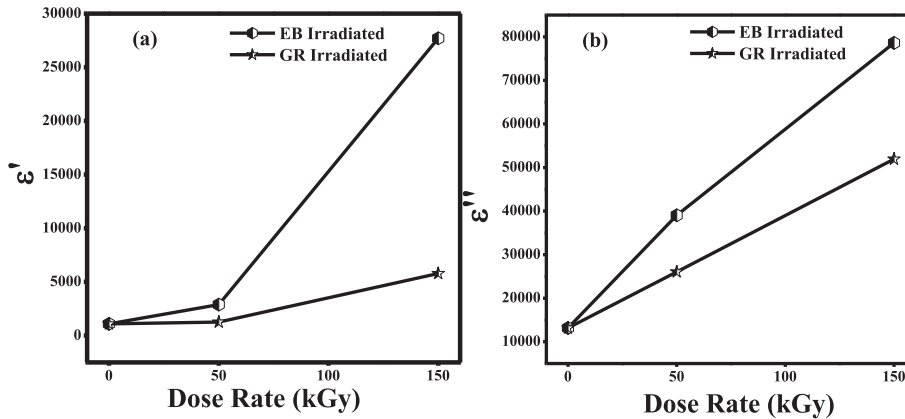


Fig. 5 – Variation of ϵ' & ϵ'' with irradiation dose for EB and GR irradiated films.

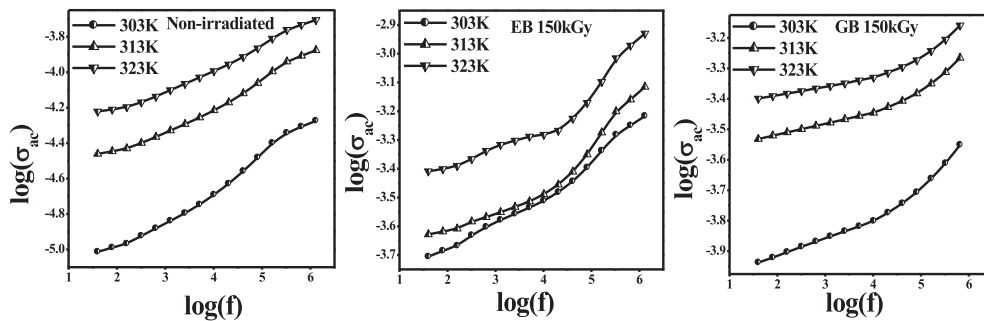


Fig. 6 – Frequency dependence conductivity of the non-irradiated and irradiated films at different temperatures.

In addition, it also emphasizes that the increase in AC conductivity with increasing temperature is due to the buildup of ordered polymeric chains or symmetry, which allows the charge transport to be still more effective. This is one of the common characteristics of disordered materials. These results suggest that conductivity changes significantly with the irradiation doses due to the occurrence of chain scissioning in polymer, which leads to faster ionic transport through the polymer matrix assisted by larger segmental motion of the polymer backbone Kumar, Deka, and Banerjee (2010). The EB

irradiated film shows high conductivity value compared to GR irradiated films as shown in Fig. 7. The total conductivity $\sigma(\omega)$ at a particular temperature can be expressed by the power law thus,

$$\sigma(\omega) = \sigma_{dc} + A\omega^S \tag{1}$$

Where, σ_{dc} is the dc conductivity and A is a constant dependent on temperature. The value of 'S' at every observed temperature is calculated from the slope of $\log(\sigma)$ versus $\log(f)$ plot (Fig. 6).

The frequency exponent 'S' value lies between 0 and 1. Such a universal dynamic response is found in a variety of disordered materials. It is observed that the frequency exponent (S) parameter decreases with increasing temperature as shown in Fig. 8.

This result was clearly represents that the AC conduction mechanism follows the correlated barrier hopping (CBH) model Elliott (1987). The AC conductivity found increases and the 'S' value decreases with the irradiation dosage. The values are cited in Table 2.

The temperature dependence of S, based on this model is

$$S = 1 - 6kT/W_M - kT\ln(1/\omega\tau_0) \tag{2}$$

Where, W_M is the effective barrier height at infinite intersite separation, τ_0 is the characteristic relaxation time, and k is the Boltzmann constant.

The values of W_M are obtained by the best-fitted parameters to Eq. (2) and are given in Table 2. As irradiation dosage enhances the value of W_M as well as conductivity were found to increase after irradiation. This is because the irradiation is

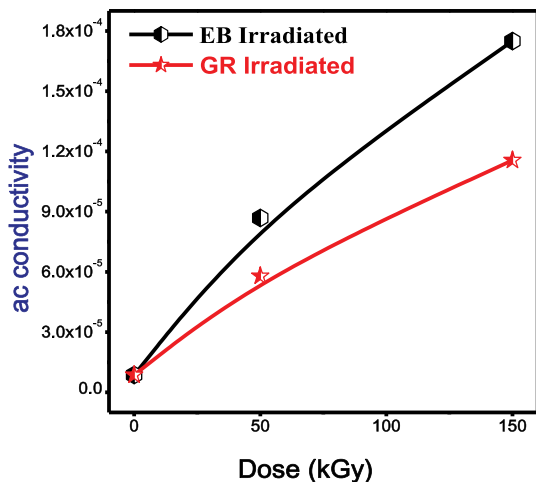


Fig. 7 – Dose dependent ac conductivity of EB and GR irradiated films at room temperature.

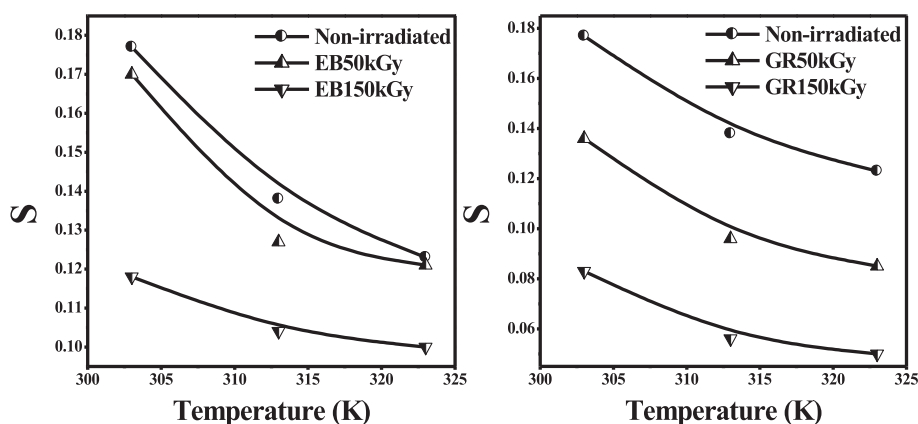


Fig. 8 – Temperature dependence of frequency exponent's' for non-irradiated and irradiated films.

Table 2 – AC conductivity and frequency exponent(S) values for EB and GR irradiated films at room temperature.

Dose (kGy)	$\sigma_{ac}(S/cm)$		S		$W_M(eV)$	
	EB	GR	EB	GR	EB	GR
0	8.626×10^{-6}	8.626×10^{-6}	0.177	0.177	0.191	0.191
50	8.087×10^{-5}	5.791×10^{-5}	0.170	0.136	0.211	0.202
150	1.749×10^{-4}	1.150×10^{-4}	0.108	0.083	0.574	0.313

expected to convert the polymeric structure into a hydrogen depleted carbon network. This carbon network is believed to make the polymer more conductive Wang, Curry, Tavenner, Dobson, and Giedd (2004).

4. Conclusions

We have investigated the EB and GR irradiation induced modifications in polymer electrolyte films using FT-IR, XRD, SEM, and impedance techniques. The results show the change in the crystallinity and chemical structures polymers after the irradiation by EB and GR radiation. The modification in the irradiated films was confirmed from FT-IR that the chain scissioning/branching occurs for both process, as the intensity of the wave number 1100 cm^{-1} enhances with incremental irradiation. Another remarkable peak observed at 1654 cm^{-1} corresponds to the presence of C=C group. An increase in the intensity of this peak suggests the occurrence cross-linking. The loss in crystallinity is slightly more in EB than in GR irradiation. In addition, the effects of EB and GR irradiation on structural morphology was investigated by SEM, and the images reveal that after irradiation there is improvement in the surface morphology of the films. The main results of this work are enhancement of the dielectric permittivity and AC conductivity of the polymer electrolytes after EB and GR irradiations. The dielectric behavior of the polymer arises due to the presence of an appreciable number of defects in the form of cross-linking, discontinuity, bending, breaking of bonds, etc. in the molecular chains. Upon irradiation, the increase in these defects results in the formation of a greater number of dipoles in the polymer matrix that governs the dielectric properties, and which leads to faster ionic transportation through the polymer matrix assisted by a larger segmental

motion of the polymer backbone. The exponent 's' decreases with increasing temperature, which clearly suggests that the AC conduction mechanism follows the correlated barrier hopping (CBH) model. Irradiation processes promoted an increase in the dielectric permittivity and conductivity value. The overall result shows that the effects are more pronounced in electron beam compared to that of gamma ray irradiation because of its more electronic energy loss in EB irradiation.

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