Effect of Electron Beam Irradiation on Mechanical and Dielectric Properties of Polypropylene Films

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Received 11 November 2008; accepted 13 May 2010
DOI 10.1002/app.32805
Published online 2 August 2010 in Wiley Online Library (wileyonlinelibrary.com).

ABSTRACT: In the present investigation the effect of electron beam irradiation on the mechanical properties of thin films of Polypropylene (PP) were measured. The dielectric properties of PP films were carried out in the frequency range from 20 Hz to 1 MHz at various DC bias potential. All measurements were carried out at room temperature. It is found that the electron beam irradiation caused an increase in Young's Modulus of PP film dose of up to 70 kGy were applied, but tensile strength and % elongation at break were found to be decrease with the increasing dose rate. The significant changes were observed in the case of dielectric constant and dielectric loss for electron irradiated PP films. The DSC results indicate that irradiation on PP films changes the thermal behavior. Minor differences in FTIR spectra were observed after irradiation treatment. It is observed that, the effect of radiation damage results in improvement in mechanical strength of the films. The increased dielectric constant and dependence of the bias voltage in case of irradiated and unirradiated PP films has been reported. It is suggested that, the PP films modified with the electron beam irradiation can be used as a good dielectric material for different electronic devices. J Appl Polym Sci 119: 1220–1229, 2011

Key words: polypropylene; electron beam irradiation; mechanical properties; dielectric properties; thermal properties

INTRODUCTION

Polypropylene (PP) is low cost and high performance engineering polymer. For health care and packaging application PP has the advantage of being nontoxic and inert to liquids and drugs. PP is widely used in medical products and pharmaceutical fields.[1] During its processing, additives such as antioxidants, antiUV or lubricants are added, that protect the polymer during sterilization and storage. PP is used as a dielectric material in many types of capacitors because of its very low dielectric loss and excellent dielectric strength.[2] The advantage of electron beam irradiation over chemical doping lies in the fact that cannot be applied because of their low validity and/or insolubility in suitable solvents. The polymer simultaneously crosslinks and degrades with irradiation and the extent of each process. Other factor being equal is apparently dependant upon the degree of crystallinity. The radiation processing of PP is of limited use as it undergoes predominantly chain scission when subjected to high energy radiation.[3] Although it is known for a long time[4,5] that an ionising radiation influences the mechanical properties of polymeric materials, still this problem is intensively being studied.[6–8] To a large extent, the tendency and range of changes in the material properties depend on whether crosslinking or degradation of a polymer dominates during the irradiation of a given material.[9–11] The progress in the engineering of low-energy or high-energy electron accelerators opens new possibilities for the radiational modification of polymers films that can be used in packaging.[12–14] Apart from the tensile strength and % strain at break, the Young's modulus is also an important functional parameter of a plastic.

Fujii et al.[15] have reported that the nonlinear behavior of dielectric loss in current flows in polymer materials contains much information about dielectric property. These properties were evaluated by using film sample of nonpolar dielectric materials. Singh et al.[2] explained that the ion irradiation of PP films leads to chain scission and cross linking and as a result there are changes in the dielectric properties. He reported that the value of capacitance and dissipation factor shows the moderate increase with respect to the temperature. Benderly et al.[16] reported that within the range of irradiation levels used, the dielectric constants of all the PP film samples were unaffected whether irradiation was carried out in air or nitrogen. Abdel-Hamid[17] has reported that electron beam modification of PP is expected to have further potential applications in radiation processing. Differential scanning calorimetry (DSC) is a major tool for studying the crystallization kinetics.

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Journal of Applied Polymer Science, Vol. 119, 1220–1229 (2011)

Thermal stability includes the glass transition and melting temperature of polymers. The thermal treatment or thermal history of a semicrystalline polymer will be reflected in its melting behavior. Many authors have explained this double melting behavior with the melt-crystallization model. This model suggest that the low-temperature and high-temperature peaks in the DSC melting curve can be attributed to the melting of some amount of original crystal and to the melting of crystals formed through the melt-crystallization process during a heating scan, respectively.[18] Deping and Guan[19] indicated that isotactic polypropylene (iPP) irradiated by electron beam in air mainly exhibits chain scission that cause the melt index to increase with increasing dose. Upon irradiation within the 500 kGy dose range, the crystallinity thermal degradation temperature and thermal stability of (iPP) increase with the irradiation dose. Zenkiewicz[20] studied the tensile strength, % elongation at break and tear resistance of electron beam irradiated polymer films. He measured the mechanical properties of polymer films in both directions i.e., machine direction (MD) and transverse direction (TD) with draw ratios of 5 : 1 and 8 : 1, respectively.

One of the most widely use of toughening PP is blending with certain elastomer. Although blending of the polymer improves the mechanical strength compatibility of the material. In the present investigation the effect of electron beam irradiation on PP film were studied. The objective was to find suitable radiation dose for significant improvement in the mechanical and dielectric properties.

EXPERIMENTAL

Materials

Polypropylene used in this work was received as commercial film from Reliance industry, India. The size of each sample was 100 mm × 20 mm and thickness was ≈ 150 μm. Acquired polypropylene thin films are 92–97% isotactic and it has a number average molecular mass of 40,000–60,000 with a polydispersity of 6–12.

Electron beam irradiation

Irradiation was carried out using linear electron beam accelerator from Department of Atomic energy, Bhabha Atomic Research Centre, Mumbai, India. The maximum electron energy was ~1.6 MeV, current was ~10 mA, and conveyor speed 13 mms^{-1}. Samples were irradiated in the dose range 5–70 kGy.

Characterization

The tensile strength, young Modulus, and % elongation of PP films of thickness (≈ 150μm) were tested on a Lloyd, USA made LR 10K Universal Tensile Machine (UTM). Thin films of length 4 cm and width 1 cm (ASTM D638) was gripped between the two jaws of the tensile machine. The cross head speed was kept at 5 mm/min. The average value was determined from the set of minimum 10 repeats.

The maximum force was 10 kN for tension and compression. The measuring accuracy of UTM is better than ±0.5%.

Irradiated and unirradiated PP thin films were cut in circular shape of (diameter 8 mm) and used for dielectric measurements. Electrical contact was made by applying as air drying type of conducting silver paste to the both side of thin films.

The Parallel capacitance (C_p), Dielectric loss (έ), Quality factor (Q), Conductance (G), Capacitance in series (Cs), Resistance in parallel (Rp), Resistance in series (Rs) and Phase angle (θ) were measured by using the LCR precision meter (Model 4284A, Agilent Technologies). Both irradiated and unirradiated films were pasted from both sides with conducting silver paste for better electrode contact. The relative dielectric constant confirmed by measuring the equivalent parallel capacitance (C_p), Thickness of film (t), and space permittivity (ε_0) with the following formula.[18,21]

$$\text{Dielectric constant } (\varepsilon) = \frac{C_p}{C_o} = \frac{t \times C_p}{A \times \varepsilon_0}$$

All the measurements were carried out within the frequency range 20 Hz to 1MHz for various DC bias potential from 0 to 40 volts. The specialties of this impedance analyzer were inbuilt DC bias potential variation facilities, which enabled us to apply simultaneous DC potential along with alternating-current (AC) signal for measuring the overall electrical parameters. The fixture assembly attached to this instrument was designed to take observations under NTP conditions only.

The thermal analysis was performed with a Perkin-Elmer DSC-7 differential scanning calorimeter at a heating rate of 10°C/min under a nitrogen atmosphere in the temperature range of 30–200°C.

FTIR spectra were recorded using Perkin Elmer, FTIR spectrophotometer (Model PARAGON-500). The comparative FTIR spectra of the treated and untreated samples were recorded in the range 4000–40 cm^{-1}. The FTIR spectra were taken in transmittance mode.

RESULTS AND DISCUSSION

Mechanical properties of irradiated and unirradiated PP films

Table I represents the results of Maximum load (N), Deflection at maximum load (mm), and Work to

TABLE I
Maximum Load (*N*), Deflection at Maximum Load (mm), and Work to Break (*J*) of Electron Beam Irradiated and Nonirradiated PP Thin Films

Electron beam irradiation dose rate to PP films	Maximum load (*N*)	Deflection at maximum load (mm)	Work to break (*J*)
0 kGy	51.79	509.23	19.49
5 kGy	42.01	459.30	13.23
10 kGy	45.57	552.96	16.98
15 kGy	40.02	420.99	14.36
20 kGy	38.57	510.84	12.23
25 kGy	34.46	6.32	12.92
30 kGy	35.30	7.52	5.28
35 kGy	35.73	6.80	4.80
40 kGy	34.69	5.87	2.17
45 kGy	31.97	6.56	3.67
70 kGy	29.30	6.22	0.76

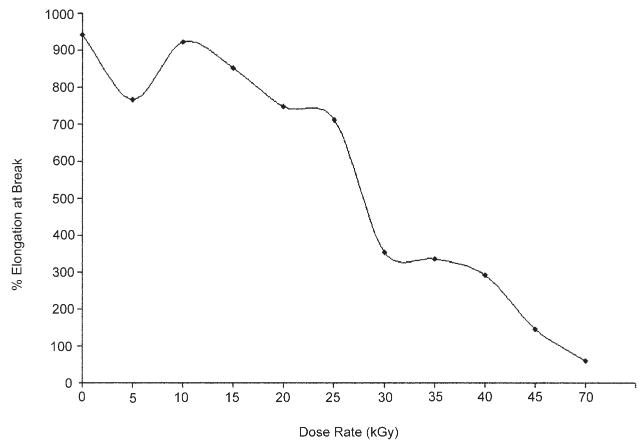

Figure 2 Effect of electron beam irradiation dose on % strain at beak of PP films.

break (*J*). It is observed that all values of mechanical property parameters decreases at higher irradiation doses. The required load to peel the sample is automatically adjusted in UTM machine.

Maximum deflection can be expressed as

$$\delta = 5\,q\,L4 = E\,I\,384$$

where

δ = maximum deflection (m, mm, in)

E = modulus of elasticity [Pa (N/m^2), N/mm^2, psi]

q = uniform load (N/m, N/mm, lb/in)

L = length of beam (m, mm, in)

The higher dose of electron beam causes more degradation in the mechanical properties of PP thin films. Experimental values of Young's Modulus, % strain at break, and tensile strength carried out for the control and electron beam irradiated PP thin films. Young's modulus (Fig. 1) shows an initial decreasing behavior at low irradiation doses. However, after the dose of 10 kGy it increases further with higher irradiation doses. This can be explained based on the destruction of the crystallites, which act as "physical crosslinks"[22]. This kind of crosslink has an effect on the modulus greater than the chemical crosslinking produced; whereas at higher radiation doses, the increase in the crosslinking density compensates for the further loss of crystallinity and leads to an increase in the modulus which is very similar to that of the density. Initially a fall occurs that corresponds to the destroyed, density rises as a result of the structural changes in the polymer, i.e., the formation of the double bonds and crosslinks, which bind the molecules more closely together and lead to a tighter packing of the polymer chains.[23,24]

Values for % strain at break are shown in Figure 2, presenting a decreasing trend with slight variations. After 10 kGy dose rate of irradiation, % strain at break was found to decrease with the increase in radiation dose. Number of crosslinks increased with irradiation dose which can be attributed to radiation induced chain scission process in PP matrix.[25,26] Tensile Strength decreases as the irradiation dose increases, which is shown in Figure 3. It was

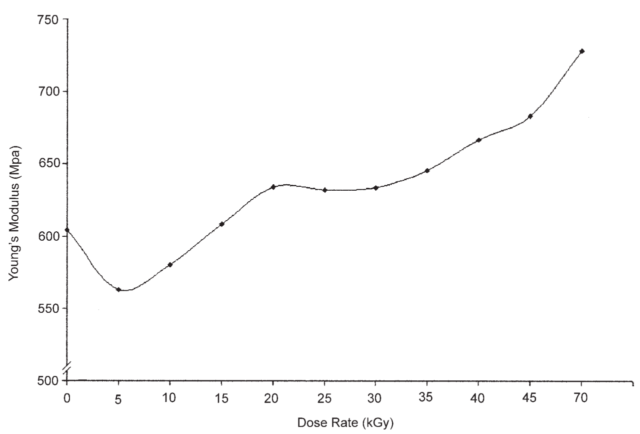

Figure 1 Effect of electron beam irradiation dose on Young's Modulus of PP films.

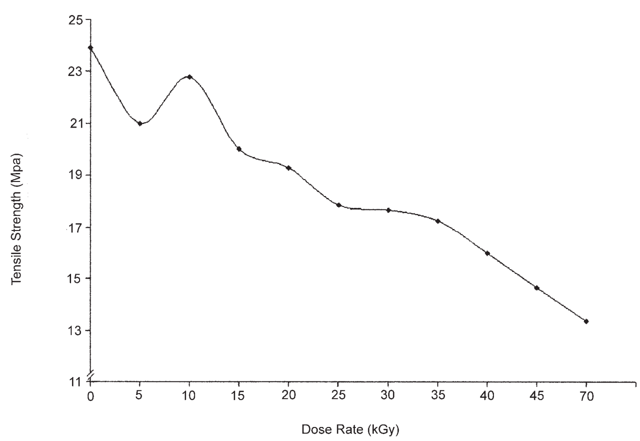

Figure 3 Effect of electron beam irradiation dose on tensile strength of PP films.

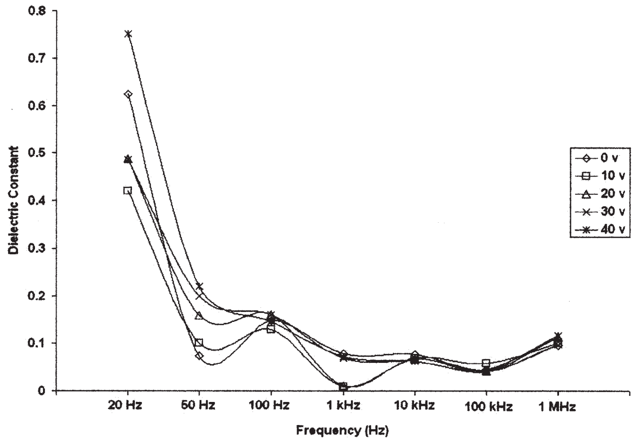

Figure 4 ε as a function of frequency for pure PP films.

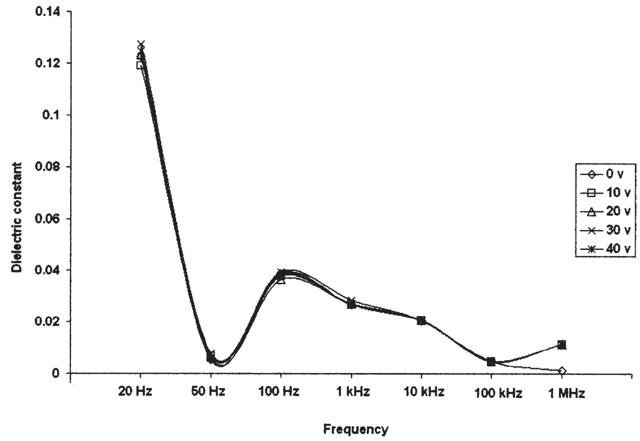

Figure 6 ε as a function of frequency for electron beam irradiated PP films with dose rate of 5 kGy.

observed that the maximum tensile strength obtained for pure PP film sample i.e., 24.24 MPa. This value slightly decreases up to 21 MPa for 5 kGy dose rate. The maximum value of the tensile strength reaches 22.84 MPa at 10 kGy dose rate but not higher than pure PP sample. Tensile Strength value was found to be decrease with the increasing irradiation dose rate after 10 kGy. The difference between unirradiated and electron beam irradiated samples was due to the degrading and crosslinking nature of PP.[27,28] In the second region (25 to 35 kGy), the tensile strength decreases relatively slow. The crystallinity is decreased with increasing radiation dose.[29] This behavior can be explained by the degradation effect of electron beam on PP.

Dielectric measurements of unirradiated and irradiated PP films

Role of DC bias potential

The application of DC bias potential (influence of direct field) from 10 to 40 V along with AC signal, clip the peaks of an AC.[30,31] The comparative electrical parameters were recorded by LCR meter as a function of frequency at various DC bias potential for Pure PP and irradiated PP films at NTP (normal temperature and pressure) conditions.

The dielectric constant of pure PP films is shown in Figure 4. The dielectric constant value ($\varepsilon = 0.75$) was found to be maximum at 20 Hz frequency for 40 V DC bias potential. There is a sharp decreasing trend observed at 50 Hz frequency. Then further increase in frequency the dielectric constant value was decreased for all applied DC bias potential. The dielectric loss of pure PP films is shown in Figure 5. The maximum dielectric loss value was ($\tan^{-1} = 6.46$) was observed at 1 MHz frequency for 30 V DC bias potential. The first peak position was obtained at 50 Hz frequency with loss factor ($\tan^{-1} = 3.65$) for applied 30 V bias potential. At 10 kHz frequency, the third peak value was observed for 30 V DC bias potential. The dielectric constant of electron irradiated PP films with dose rate of 5 kGy is shown in Figure 6. The maximum dielectric constant value

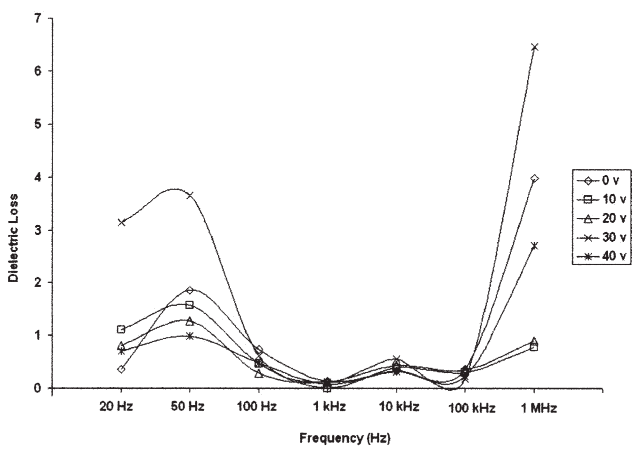

Figure 5 Tan^{-1} as a function of frequency for pure PP films.

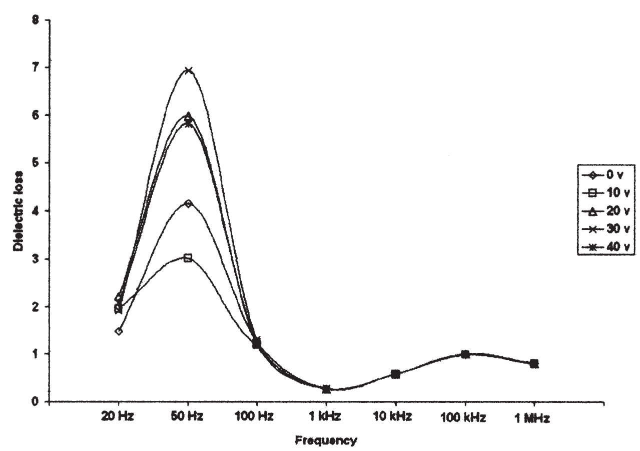

Figure 7 Tan^{-1} as a function of frequency for electron beam irradiated PP films with dose rate of 5 kGy.

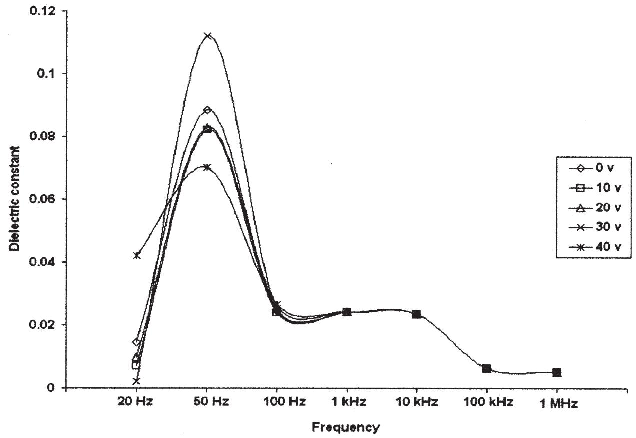

Figure 8 ε as a function of frequency for electron beam irradiated PP films with dose rate of 10 kGy.

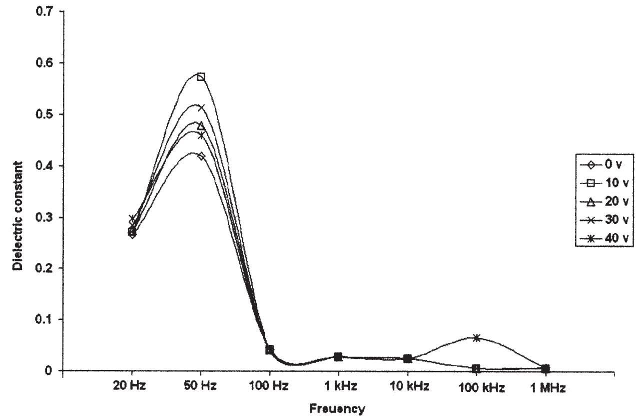

Figure 10 ε as a function of frequency for electron beam irradiated PP films with dose rate of 15 kGy.

(ε = 0.12) was observed at 20 Hz frequency for 30 V DC bias potential. There is only one peak observed at 100 Hz frequency with dielectric constant value ε = 0.039. Thereafter there is no effect on dielectric constant value due to increase in the DC bias potential and frequency. The dielectric loss of electron irradiated PP films with dose rate of 5 kGy is shown in Figure 7. The highest dielectric loss value ($\tan^{-1}$ = 6.93) was observed at 50 Hz for 30 V DC bias potential. The plot is shows only one peak position at 50 Hz frequency and then the loss factor decreases with further increase in frequency.

The dielectric constant of electron beam irradiated PP films with dose rate of 10 kGy is shown in Figure 8. The maximum dielectric constant value was plotted at 50 Hz frequency and for 30 V DC bias potential with value (ε = 0.11). There is remarkable decrease in dielectric constant value at 100 Hz frequency. It was observed that dielectric constant decreases with further increase in the frequency. The maximum dielectric loss value of irradiated PP films with 10 kGy dose rate was observed at 20 Hz frequency and for 40 V DC bias potential with value (ε = 4.41). There is a sharp decrease in loss value at 1 kHz frequency. Another peak was observed at 100 kHz for all applied DC bias potentials as shown in Figure 9. The maximum dielectric constant value of irradiated PP films with 15 kGy dose rate was observed at 50 Hz frequency and for 10 V DC bias potential with value (ε = 0.57). There is a sharp decrease in dielectric constant value at 100 Hz frequency. It was observed that dielectric constant does not change with further increase in the frequency as shown in Figure 10. The dielectric loss of electron beam irradiated PP films with dose rate of 15 kGy is shown in Figure 11. There are two peaks, observed at 100 Hz and 100 kHz. The highest loss value ($\tan^{-1}$ = 1.24) was obtained at 100 Hz frequency for 0 V DC bias potential.

The dielectric constant of electron beam irradiated PP films with dose rate of 20 kGy is shown in Figure 12. The dielectric constant value (ε = 0.26) was found to be maximum at 20 Hz frequency for 30 V DC bias potential. There is a sharp decrease in the dielectric constant values at 50 Hz frequency and

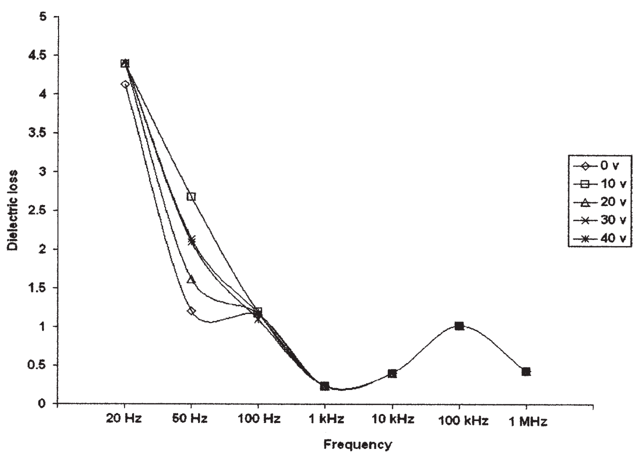

Figure 9 Tan^{-1} as a function of frequency for electron beam irradiated PP films with dose rate of 10 kGy.

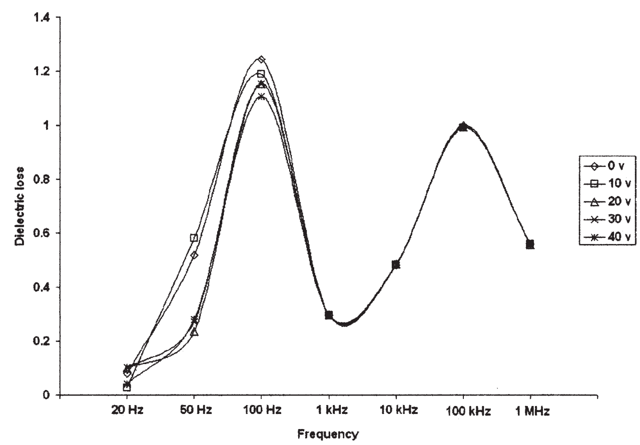

Figure 11 Tan^{-1} as a function of frequency for electron beam irradiated PP films with dose rate of 15 kGy.

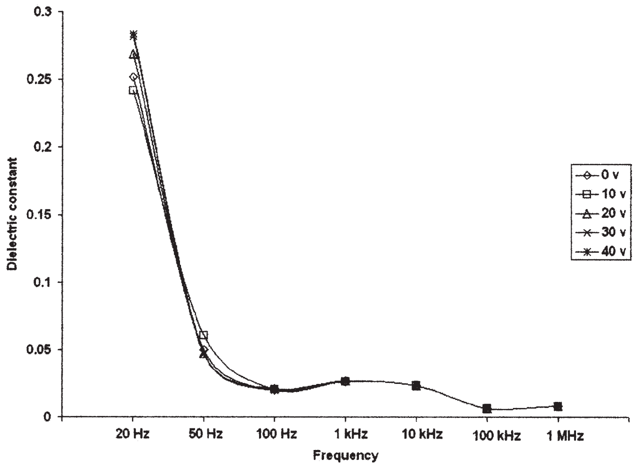

Figure 12 ε as a function of frequency for electron beam irradiated PP films with dose rate of 20 kGy.

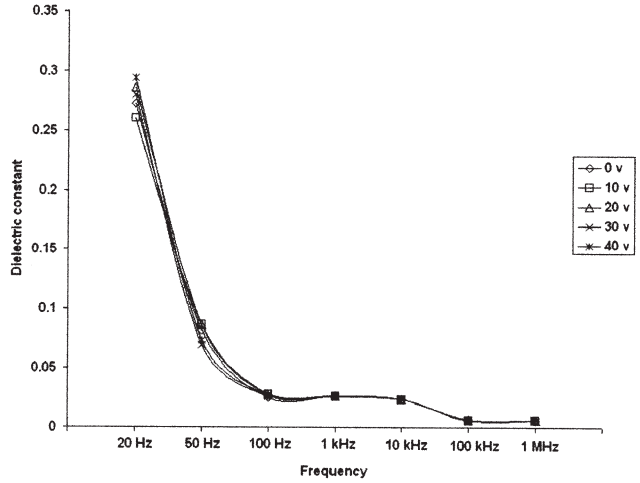

Figure 14 ε as a function of frequency for electron beam irradiated PP films with dose rate of 25 kGy.

further decreases with increase in frequency for all DC bias potentials. The dielectric loss of electron beam irradiated PP films with dose rate of 20 kGy is shown in Figure 13. There are two peaks observed at 50 Hz and 100 kHz. The highest loss value ($\tan^{-1} = 2.87$) was obtained at 50 Hz frequency for applied 10 V DC bias potential and the lowest dielectric loss value was observed at the same frequency with value ($\tan^{-1} = 1.61$) at 30 V DC bias potential. The dielectric constant value ($\varepsilon = 0.29$) of irradiated PP films with 25 kGy dose rate was found to be maximum at 20 Hz frequency at 40 V DC bias potential. The dielectric constant value decreases at 50 Hz and then it decreases with further increase in frequency as shown in Figure 14. The dielectric loss of electron irradiated PP films with dose rate of 25 kGy is shown in Figure 15. The maximum dielectric loss value was observed ($\tan^{-1} = 1.98$) at 100 Hz frequency for 0 V DC bias potential. There is a remarkable decrease in loss value at 1 kHz frequency and then it increases with further increase in frequency.

The dielectric constant of electron irradiated PP films with dose rate of 30 kGy is shown in Figure 16. The dielectric constant value ($\varepsilon = 0.32$) was found to be maximum at 20 Hz frequency for 0 V DC bias potential. There is a sharp decreasing trend observed at 100 Hz frequency. There is one peak observed at 1 kHz frequency with dielectric constant value $\varepsilon = 0.027$. Thereafter there is no effect on dielectric constant value due to increase in the DC bias potential and frequency. The maximum dielectric loss value of irradiated PP films with 30 kGy dose rate was observed at 20 Hz frequency and at 0 V DC bias potential with value ($\tan^{-1} = 1.83$). As frequency increases up to 100 Hz; first peak was observed with dielectric loss value $\tan^{-1} = 1.33$ for 0 V DC bias potential. Another peak value was observed at 10 kHz frequency and the dielectric loss value decreases with further increase in applied frequency is shown in Figure 17. The dielectric constant of electron irradiated PP films with dose rate of

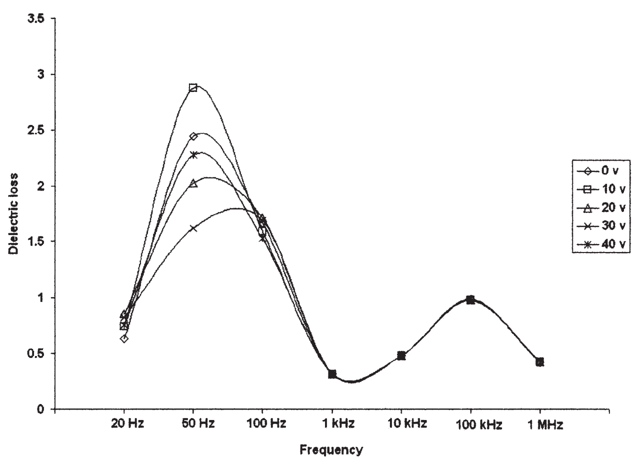

Figure 13 Tan^{-1} as a function of frequency for electron beam irradiated PP films with dose rate of 20 kGy.

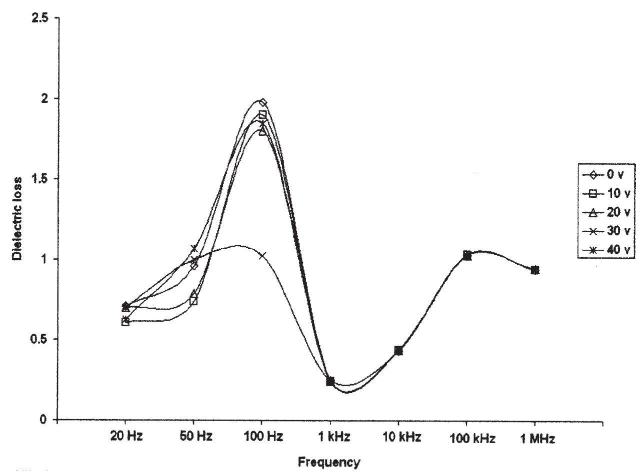

Figure 15 Tan^{-1} as a function of frequency for electron beam irradiated PP films with dose rate of 25 kGy.

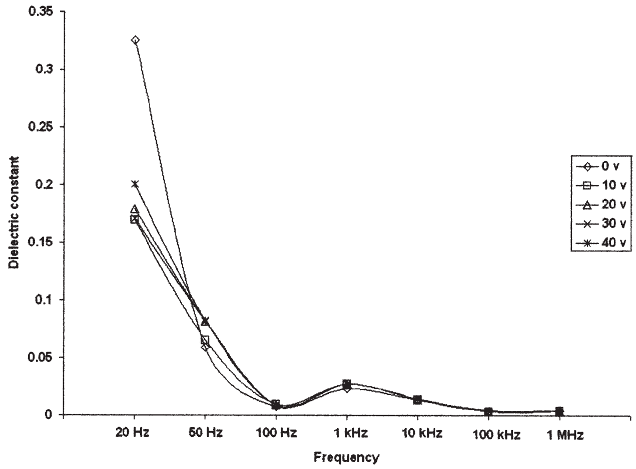

Figure 16 ε as a function of frequency for electron beam irradiated PP films with dose rate of 30 kGy.

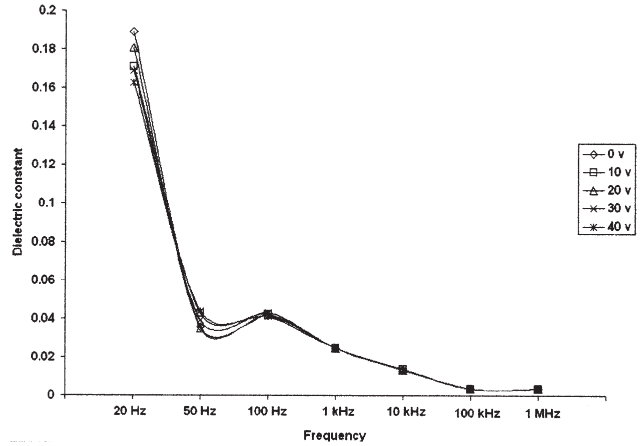

Figure 18 ε as a function of frequency for electron beam irradiated PP films with dose rate of 40 kGy.

40 kGy is shown in Figure 18. The dielectric constant value (ε = 0.18) was found to be maximum at 20 Hz frequency at 0 V DC bias potential. There is only one peak observed at 100 Hz frequency with dielectric constant value ε = 0.042. Thereafter dielectric constant value decreases with further increase in frequency. The dielectric loss of electron irradiated PP films with dose rate of 40 kGy is shown in Figure 19. The maximum dielectric loss value ($\tan^{-1}$ = 4.013) at 50 Hz frequency for 0 V DC bias potential. The lowest dielectric loss value at 50 Hz frequency is ($\tan^{-1}$ = 3.001) for 10 V DC bias potential. There is a remarkable decrease in loss value at 100 Hz frequency and then it increases with higher frequencies.

The dielectric constant value (ε = 0.17) of irradiated PP films with 45 kGy dose rate was found to be maximum at 20 Hz frequency at 30 V DC bias potential. The dielectric constant value decrease at 100 Hz and then it decreases with further increase in frequency as shown in Figure 20. The highest dielectric loss value of irradiated PP films with 45 kGy dose rate was observed at 50 Hz frequency and for 10 V DC bias potential with $\tan^{-1}$ =1.85. The second peak was observed at 10 kHz for all applied DC bias potential. Thereafter there is no effect on loss value due to increase in the DC bias potential and frequency as shown in Figure 21. The dielectric constant of electron irradiated PP films with dose rate of 70 kGy is shown in Figure 22. The dielectric constant value (ε = 0.202) was found to be maximum at 20 Hz frequency for 0 V DC bias potential. There is a sharp decreasing trend observed at 100 Hz frequency. There is one peak observed at 1 kHz frequency with dielectric constant value ε = 0.023. Thereafter there is no effect on dielectric constant value due to increase in the DC bias potential and frequency. In Figure 23, the maximum dielectric loss value of irradiated PP films with 70 kGy dose rate was observed at 100 Hz frequency and for 40 V DC bias potential with value ($\tan^{-1}$ = 1.13). As frequency increases up to 10 kHz; second peak was observed with dielectric loss value $\tan^{-1}$ = 0.95. The third maximum loss factor ($\tan^{-1}$ = 0.87) was

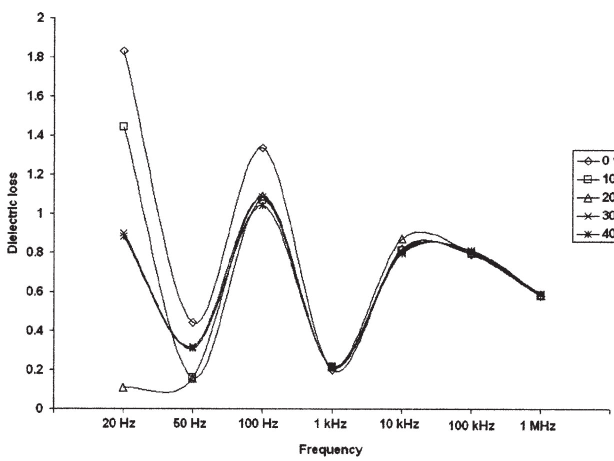

Figure 17 Tan^{-1} as a function of frequency for electron beam irradiated PP films with dose rate of 30 kGy.

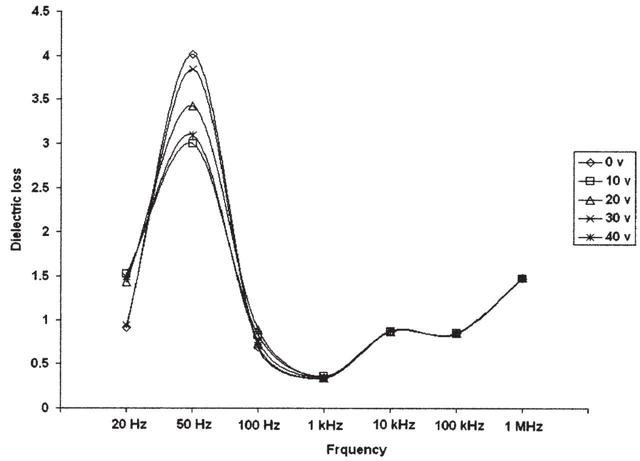

Figure 19 Tan^{-1} as a function of frequency for electron beam irradiated PP films with dose rate of 40 kGy.

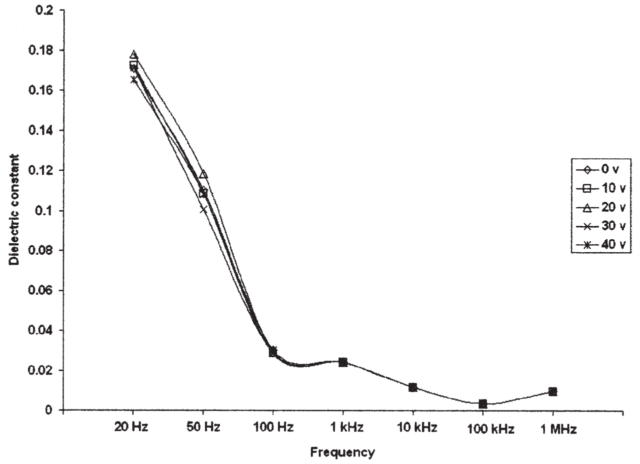

Figure 20 ε as a function of frequency for electron beam irradiated PP films with dose rate of 45 kGy.

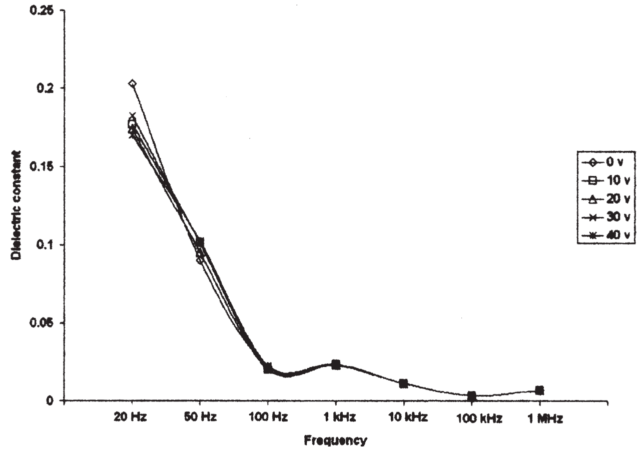

Figure 22 ε as a function of frequency for electron beam irradiated PP films with dose rate of 70 kGy.

observed at 20 Hz frequency for 40 V DC bias potential.

Figure 24 shows the DSC thermograms for nonirradiated PP films by electron beam irradiation at 10, 20, 40, and 70 kGy. In the DSC curves, the endothermic peaks corresponding to melting of PP can be seen at 30–220°C. The peaks corresponding to melting temperature (T_m) of all irradiated PP films can be observed in the same range as the (T_m) of control PP. Melting temperature increased after electron irradiation (159.24°C at 0 kGy, 160.5°C at 20 kGy, and 159.7°C at 40 kGy). However, at 10 and 70 KGy melting temperature decreased after electron irradiation. The lower melting temperature (155.71°C) was observed at higher irradiation dose i.e., 70 kGy. On basis of these facts, highly reactive radicals and energetic species may be responsible for the variation occurred in the properties of polymer.

Representative FTIR spectra of nonirradiated and irradiated (electron – beam irradiation) samples are shown in Figure 25. FTIR spectra are practically identical indicating that any changes observed in molecular structure as a result of irradiation are not easily detected with FTIR spectroscopy. The trend shows no variation in this study comparing with (Fintzou et al.) where electron beam irradiation in the presence of air was used. So our study can be explained by the fact that in the presence of air, oxygen reacts with the free radicals produced in the polymer matrix as a result of irradiation leading to the formation of carbonyl group.[32]

The progress in the irradiation of electron beam opens new possibilities for radiational modifications in polymer matrix. This kind of crosslink has an effect on the dielectric properties greater than the chemical crosslinking produced; whereas at higher radiation doses, the increase in the crosslinking density. The formation of free radicals; recombination and scission of bonds; crosslinking and different oxidation reactions are taken place during irradiation process on polypropylene. Due to this molecules are closely bind together lead to tighter packing of

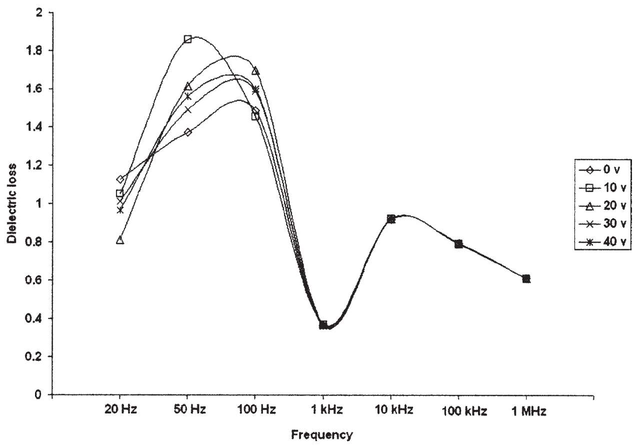

Figure 21 Tan^{-1} as a function of frequency for electron beam irradiated PP films with dose rate of 45 kGy.

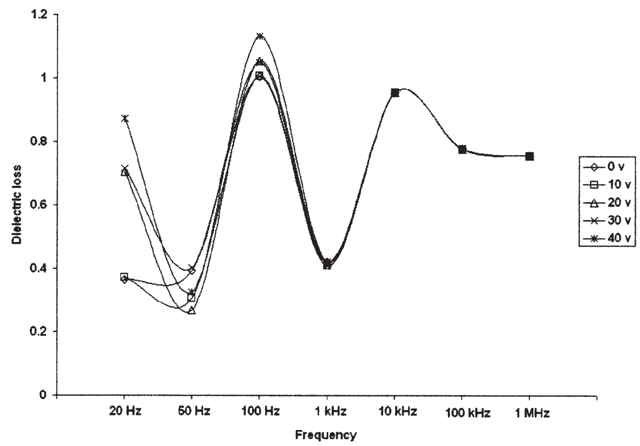

Figure 23 Tan^{-1} as a function of frequency for electron beam irradiated PP films with dose rate of 70 kGy.

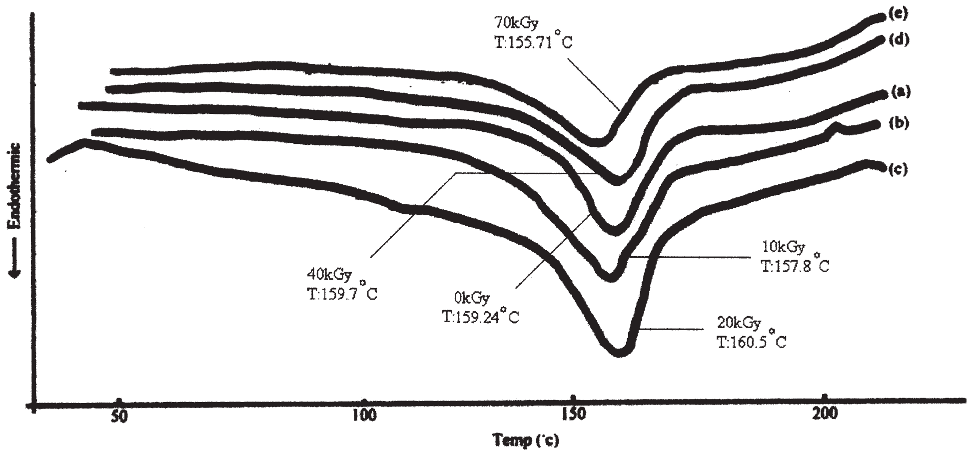

Figure 24 Effect on the DSC thermograms of PP thin films, at different doses: (a) 0 kGy, (b) 10 kGy, (c) 20 kGy, (d) 40 kGy, and (e) 70 kGy.

polymer chains. It is noteworthy that in this work, the variation occurred in dielectric properties due to the electron beam irradiation process. These highly reactive free radicals and other transferred energetic species may be acts as crosslinking agents for the polymer.

CONCLUSION

PP irradiated by electron beam in air mainly exhibit crosslinking that causes the mechanical properties to increase with increasing dose. The Young's Modulus of pp irradiated at 5 kGy is 562.98 MPa which is less than that of pure PP whereas at 20 and 70 kGy, the Young's Modulus is increased up to 634.034 and 728.68 MPa, respectively. These facts indicate that low dose irradiation is sufficient to improve the properties of polymers. Thus due to the highest impact strength of irradiated Polypropylene, it can be used as good alternative for Nylon rope. After irradiation the breaking of a limited but critical number of chains are connecting crystalline regions of polymer. The DSC results shows that deterioration occurred in the thermal properties of PP. FTIR spectra analysis showed that, the structural properties of the polymer was not destroyed by electron irradiation even at 70 kGy.

The electron beam irradiation of PP films leads to crosslinking and as a result there are changes in the

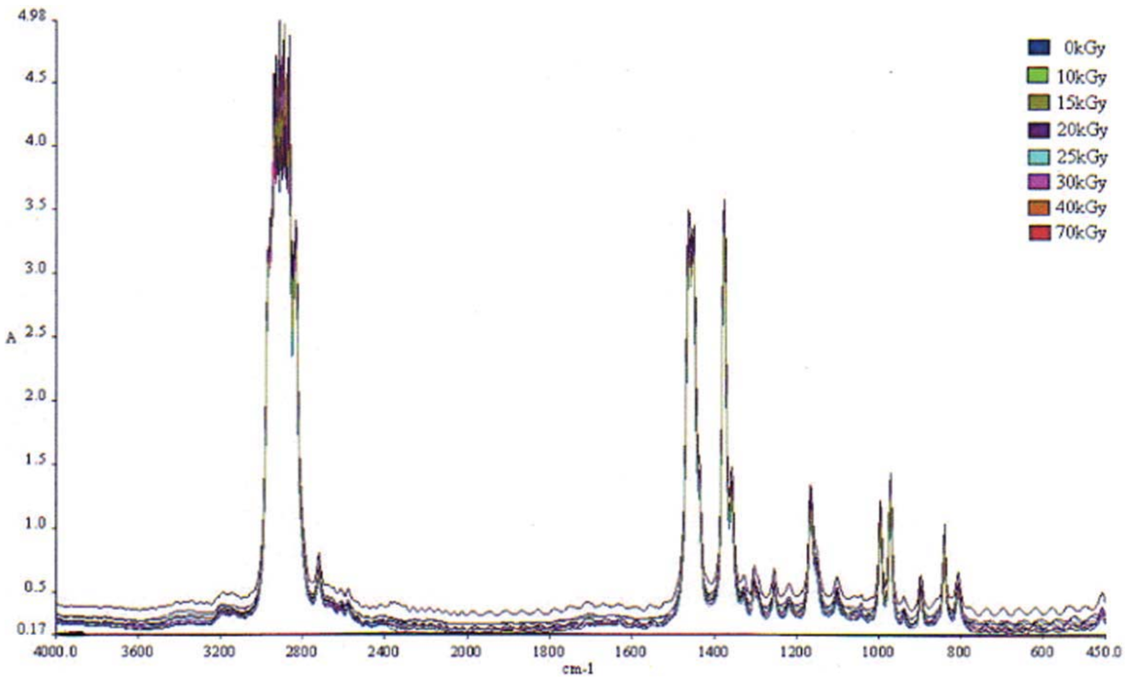

Figure 25 FTIR spectra of the PP films, electron beam irradiated at different doses. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

dielectric properties. There are significant changes in both dielectric constant and dielectric loss resulted from irradiation of electron beam. The value of loss factor increases with increases in irradiation dose rate. The loss factor shows a moderate increase up to 100 kHz frequency for all irradiated PP films. This suggests that PP film can be used as capacitors, may be useful below 100 kHz frequency. Electron beam irradiated PP with dose rate of 20 kGy can be used as a good capacitor dielectric because of its very low dielectric loss and excellent dielectric strength.

Authors wish to thank Mr. K.S.S. Sarma for his assistance in electron beam irradiation experiments in the facilities of BARC – BRIT Complex (Navi Mumbai -400705, India).

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