Chapter 4

Electron Beam Irradiation Effects on Dielectric Parameters of SiR–EPDM Blends

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Additional information is available at the end of the chapter

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Abstract

The survival of an electrical system is mostly governed by the endurance limit of the dielectric material employed in it. The five different compositions of SiR–EPDM blends were prepared. Electron beam radiation has been widely used in the cable manufacturing industries in order to increase the life of the cable. Hence, the five blends were irradiated to 5, 15 and 25 Mrad dose levels by electron beam accelerator. The dielectric parameters such as breakdown voltage (BDV), dielectric strength (DS), dielectric constant (DC), and dissipation factor (DF) were measured as per ASTM/IEC standards. This chapter evaluates the effect of electron beam irradiation on dielectric parameters of SiR–EPDM blends.

Keywords: silicone rubber (SiR), ethylene propylene diene monomer (EPDM), breakdown voltage (BDV), dielectric strength (DS), dielectric constant (DC), dissipation factor (DF), electron beam irradiation

1. Introduction

Electron beam irradiation has been effectively utilized in power cable industry and identified as one of the most advanced processing techniques. The products processed with electron beam radiation, experience shorter exposure time, which could result in less oxidative effects on certain materials. It is essential to investigate the effect of electron beam irradiation upon the dielectric parameters of the five different compositions of SiR–EPDM blends [1–4, 9]. Hence, the samples of SiR–EPDM were irradiated to 5, 15 and 25 Mrad dose levels by electron beam accelerator. The new functional groups formed during blending and after the electron beam irradiation were investigated through physicochemical investigation techniques like Fourier transform infrared spectroscopy (FTIR). To observe the morphological changes and

also to identify the elemental composition, scanning electron microscope (SEM) analysis and energy dispersive X-ray analysis (EDXA) were performed on SiR–EPDM blends. The effects of electron beam irradiation on the dielectric parameters of various compositions of SiR–EPDM blends were reported.

2. Experimental

2.1. Preparation of SiR–EPDM blends

Commercially, available SiR and EPDM were used. Type of SiR-polydimethyl siloxane (PDMS). The composition of EPDM is ethylene-65%; propylene-25%; diene monomer-10%. Diene type is ethylidene norbornene (ENB). They are supplied by M/S Joy Rubbers, India. The five different compositions of SiR–EPDM blends were prepared [1–4, 9].

1. Blend A—SiR 90%/EPDM 10%.
2. Blend B—SiR 70%/EPDM 30%.
3. Blend C—SiR 50%/EPDM 50%.
4. Blend D—SiR 30%/EPDM 70%.
5. Blend E—SiR 10%/EPDM 90%.

2.2. Electron beam irradiation

The five different compositions of SiR–EPDM blends were irradiated up to 5, 15 and 25 Mrad doses using an electron beam accelerator of 1.5 MeV rating at M/S Siechem Industries, Pondicherry, India.

3. Characterization of SiR–EPDM blends

3.1. Dielectric characterization

In order to analyze the dielectric behavior of SiR–EPDM blends in harmful environments, blends have been tested as per ASTM/IEC standards.

3.1.1. Breakdown voltage and dielectric strength

As per standard ASTM D 149 (IEC 60243), BDV and DS were measured. The sample dimensions of 5 × 5 × 0.3 cm were placed between two electrodes, and the voltage was increased at a fixed rate of 500 V/s. The voltage at which dielectric breakdown occurs was measured as BDV. DS was calculated.

3.1.2. Dielectric constant and dissipation factor

DC and DF were measured as per ASTM D 150 (IEC 60250) at 1 MHz. The sample dimensions were 5 × 5 × 0.3 cm.

3.2. Physicochemical investigations

Various physicochemical techniques such as FTIR, EDXA and SEM were used to identify the nature of changes in the electron beam irradiated samples of SiR–EPDM blends.

3.2.1. Fourier transform infrared spectroscopy (FTIR) analysis

FTIR spectra of electron beam irradiated samples were taken using Perkin Elmer spectrophotometer, in the wave number ranging from 500 cm^{-1} to 4000 cm^{-1}. The number of scans for each IR spectrum was 4**.**

3.2.2. Energy dispersive X-ray (EDXA) analysis

EDXA analysis has been performed using EDXA analysis setup (Make HITACHI), in order to determine the elemental composition of the materials at the surface of the electron beam irradiated samples of SiR–EPDM blends.

3.2.3. Scanning electron microscopy (SEM) analysis

SEM analysis was performed using a scanning electron microscope (Make HITACHI) with a magnification of 5–300,000, in order to study the morphology of the surface of electron beam irradiated samples of SiR–EPDM blends.

4. Results

4.1. Dielectric characterization of virgin SiR–EPDM blends

The virgin SiR rich blends (A and B) have higher breakdown voltage (BDV) and dielectric strength (DS), when compared to remaining blends. This may be due to the occurrence of maximum self cross-linking during the blending process itself. During the blending process, the cross-linking reaction has taken place between the side chains of SiR and EPDM. The blend C and EPDM rich blends (D and E) were found to have lesser values of BDV, DS, and higher values of DC and DF in comparison with SiR rich blends (A and B).

4.2. Effect of electron beam irradiation on dielectric behavior of SiR–EPDM blends

4.2.1. Effect on breakdown voltage and dielectric strength

Figures 1 and **2** depict the variations in breakdown voltage and dielectric strength of five different compositions of SiR–EPDM for various doses of electron beam irradiation.

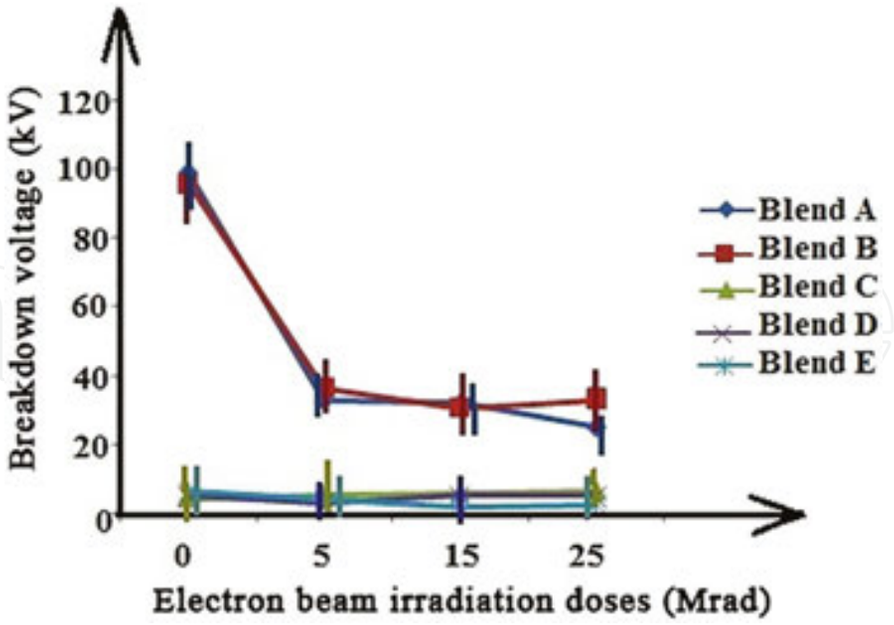

Figure 1. Variations in breakdown voltage of SiR–EPDM blends for various doses of electron beam irradiation.

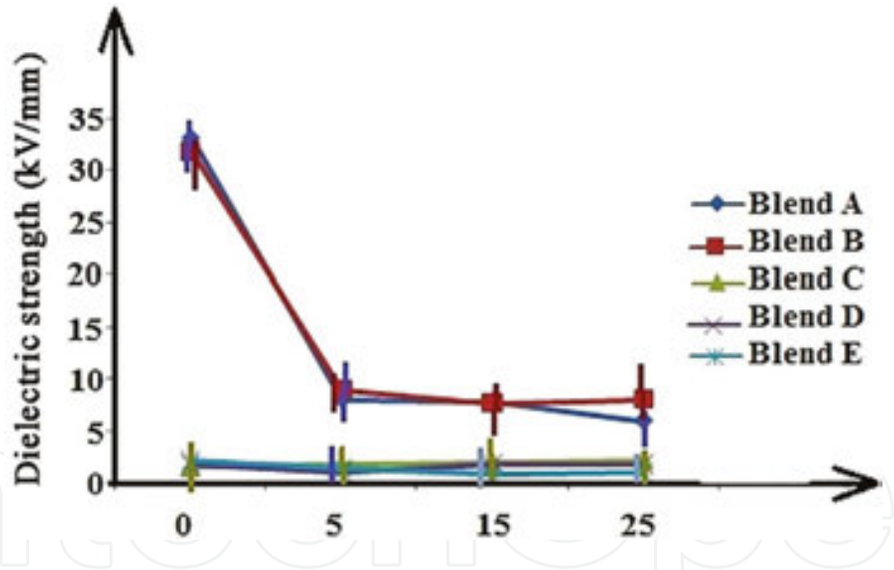

Figure 2. Variations in dielectric strength of SiR–EPDM blends for various doses of electron beam irradiation.

The BDV and DS of SiR-rich blends (A and B) and EPDM-rich blends (D and E) reduced for all doses of electron beam. The BDV and DS of the blend C improved for all doses of electron beam. The DC of the blend D and E has been improved at 5 and 5/25 Mrad respectively.

4.2.2. Effect on dielectric constant and dissipation factor measurement

Figures 3 and **4** depict the variations in dielectric constant and dissipation factor of five different compositions of SiR–EPDM blends for various doses of electron beam irradiation.

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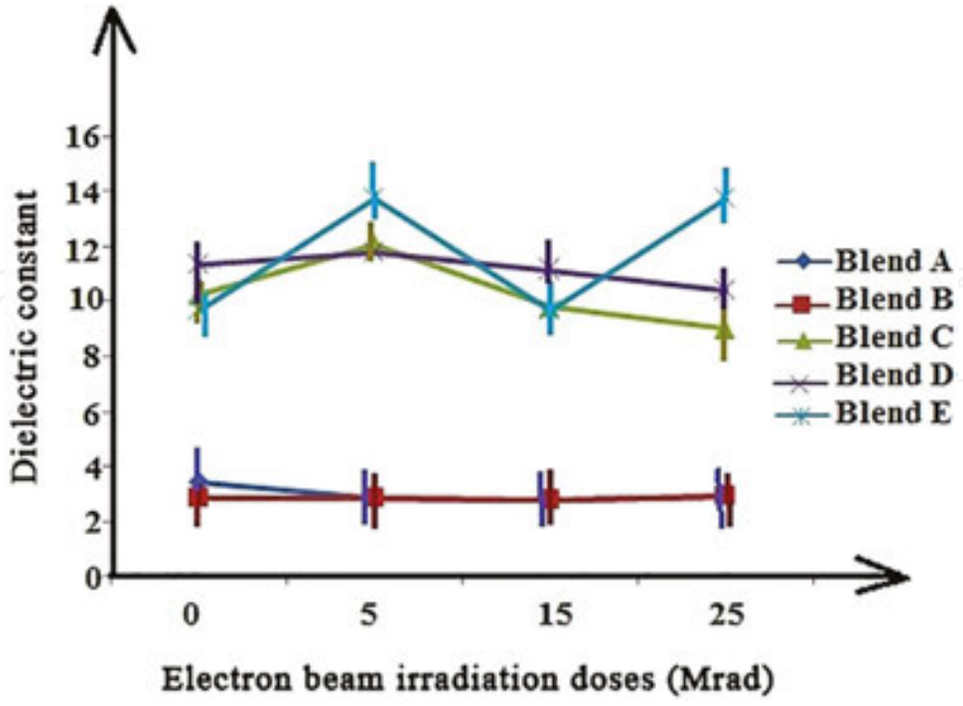

Figure 3. Variations in dielectric constant of SiR–EPDM blends for various doses of electron beam irradiation.

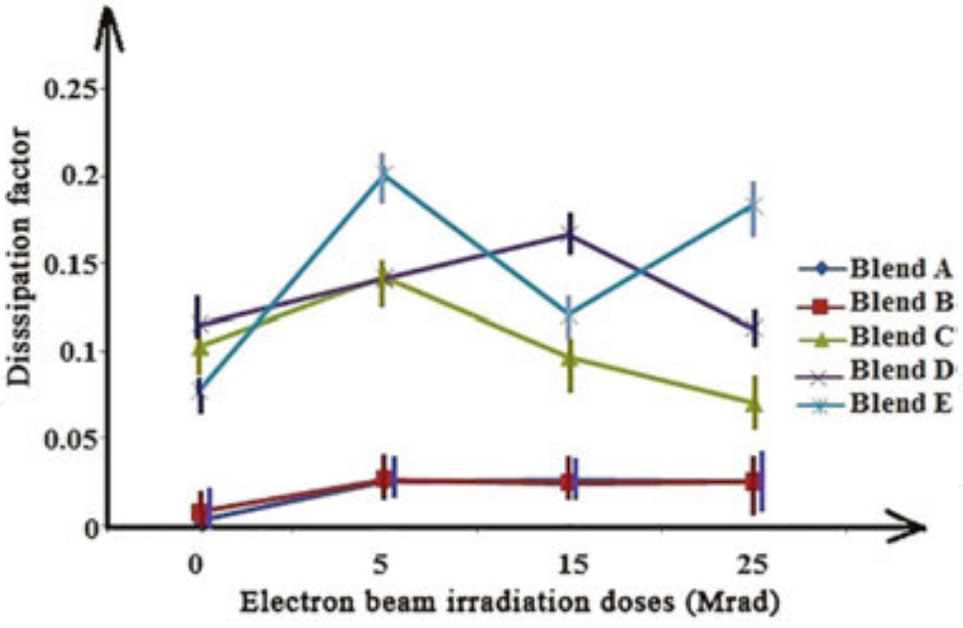

Figure 4. Variations in dissipation factor of SiR–EPDM blends for various doses of electron beam irradiation.

The DC of the blend A reduced for all doses of electron beam. The DC of blend B reduced at 5 and 15 Mrad. The DF of SiR-rich blends (A and B) reduced for all doses of electron beam. The DC and DF of the blend C improved at 5 and 15 Mrad respectively. The blend D has the improved DF at 25 Mrad.

5. Discussion

5.1. Dielectric performance of virgin and electron beam irradiated SiR–EPDM blends

For SiR–EPDM blends, it has been observed that cross-linking and chain scission may modify the macromolecular chains of the material. The consequence is the change in the dielectric parameters of the material. The effect of dominant mechanism can be noted from the changes in dielectric parameters.

5.2. FTIR analysis

FTIR spectra of electron beam irradiated samples of SiR–EPDM blends were obtained to identify the mechanism for the change in dielectric parameters after the electron beam irradiation. FTIR spectra of the virgin and electron beam irradiated samples of three compositions of SiR–EPDM blends are depicted in **Figures 5–7** respectively.

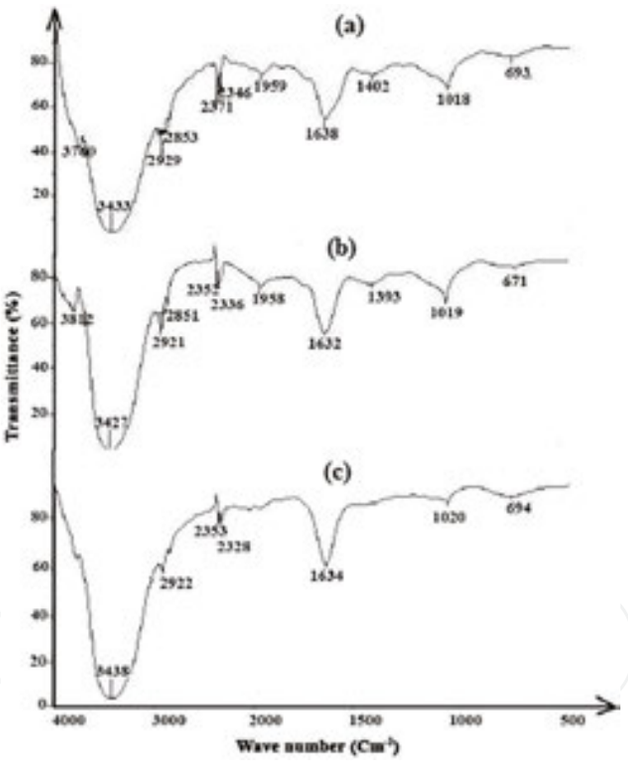

Figure 5. FTIR spectra of electron beam irradiated samples of blend A. (a) 5 Mrad, (b) 15 Mrad and (c) 25 Mrad.

The FTIR investigations on electron beam radiated samples revealed that the radiation has induced the chemical and morphological changes. The variation in dielectric parameters was validated through FTIR spectra. It depicts the occurrence of new functional groups along with the % absorbance and the corresponding wave number. **Tables 1** and **2** list the correlation of variation in dielectric parameters of electron beam irradiated samples of SiR rich blends and EPDM rich blends and blend C using FTIR respectively.

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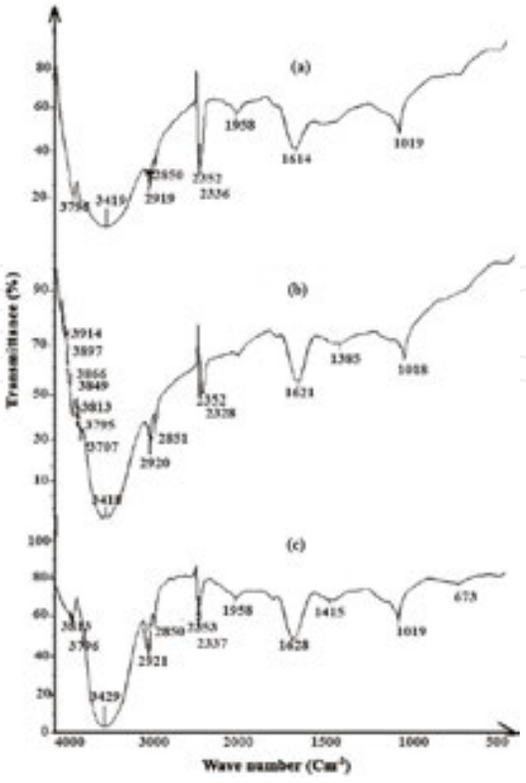

Figure 6. FTIR spectra of electron beam irradiated samples of blend C. (a) 5 Mrad, (b) 15 Mrad and (c) 25 Mrad.

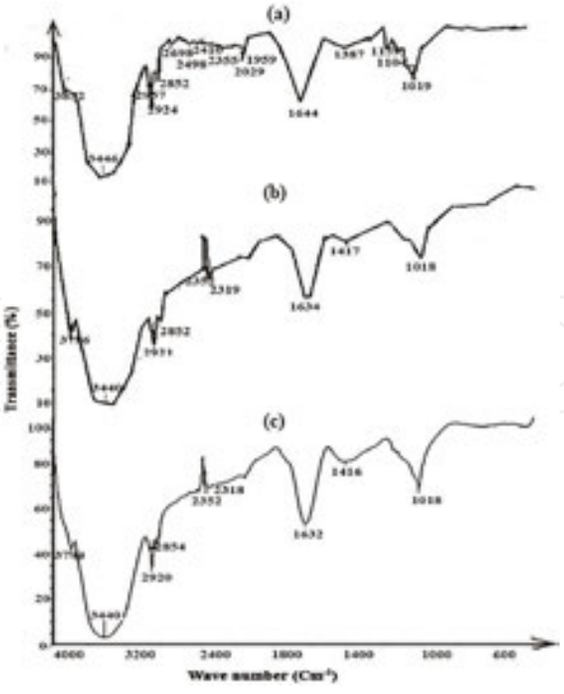

Figure 7. FTIR spectra of electron beam irradiated samples of blend E. (a) 5 Mrad, (b) 15 Mrad and (c) 25 Mrad.

Behavior of dielectric parameter/doses	5 Mrad	15 Mrad	25 Mrad
B → Improvement in DC at 25 Mrad	Alcohol(OH) bonded, strong, broad A → 3433 cm^{-1} with 150% B → 3427 cm^{-1} with 116% Si–O–Si A →1018 cm^{-1} with 18% B →1018 cm^{-1} with 10%	Alcohol(OH) bonded, strong, broad A → 3427 cm^{-1} with 150% B → 3446 cm^{-1} with 150% Si–o–Si A → 1019 cm^{-1} with 15% B → 1018 cm^{-1} with 2%	Alcohol(OH) bonded, strong, broad A → 3438 cm^{-1} with 150% B → 3429 cm^{-1} with 150% Si–O–Si A → 1020 cm^{-1} with 4% B → 1018 cm^{-1} with 49%
Reduction in BDV, DS and DF A → Reduction in DC	Absence of alcohol (OH)-free, strong, sharp group and acid (COOH) group		

Table 1. Correlation of variation of dielectric parameters of electron beam irradiated samples of SiR rich blends using FTIR.

The BDV, DS, and DF of the SiR rich blends (A and B) found to reduce for all doses of electron beam irradiation. This is due to the disappearance of acid (COOH) group in them. The BDV and DS of the blend C is improved for all doses of electron beam irradiation. This is due to the appearance of Si–O–Si group at 1019, 1018, and 1019 cm^{-1} with 29, 20, and 23% absorbance in it. The dielectric constant is improved at 5 Mrad. This may be due to the appearance of =C–H (Alkene, bending, strong). The DF has been reduced at 15 Mrad. This may be due to the disappearance of =C–H (Alkene, bending, strong). The maximum improvement in DC of blend D occurred at 5 Mrad. This may be due to the increase in Si–O–Si group at 1018 cm^{-1} with 34% absorbance and also due to the shifting of alcohol (OH)-free group to higher wave number [16–18]. The maximum improvement in DC of blend E has occurred at 25 Mrad. This may be due to the increase in alcohol (OH)-free group at 3795 cm^{-1} with 138% absorbance.

Behavior of dielectric parameter/doses	5 Mrad	15 Mrad	25 Mrad
C (50:50) Improvement in BDV, DS Improvement in DF except at 15 Mrad D → 3445 cm^{-1} Improvement in DC at 5 Mrad	Absence of =C–H (Alkene), bending strong	Improvement in	=C–H (Alkene), bending strong at 673 cm^{-1} with 11% Si–O–Si group at 1019 cm^{-1} with 23% (C–H) alkane group at 1416 cm^{-1} with 16%
EPDM rich blends (D and E)	Absence of =C–H	Alcohol(OH) free,	Alcohol(OH) bonded, strong,

Behavior of dielectric parameter/ doses	5 Mrad	15 Mrad	25 Mrad
D → DC except at 15/25 Mrad Improvement in DF except at 5/15 Mrad E → Improvement in DC except at 15 Mrad	(Alkene), bending strong Increase in Si–O–Si group at 1018 cm^{-1} with 34% absorbance *Shifting of alcohol (O–H) free group to higher wave number	strong, sharp D → 3813 cm^{-1} with 15% E → 3796 cm^{-1} with 45% Alcohol(OH) bonded, strong, broad D → 3434 cm^{-1} with 150% E → 3440 cm^{-1} with 150%	broad with 150% E → 3440 cm^{-1} with 150%

Table 2. Correlation of variation of dielectric parameters of electron beam irradiated samples of EPDM rich blends and blend C using FTIR.

5.3. EDXA analysis

Figures 8–10 show the EDXA curves of the electron beam irradiated samples of blends A, C, and E respectively. The inferences from EDXA curves of all the irradiated samples of SiR–EPDM blends are listed in **Tables 3**and **4**.

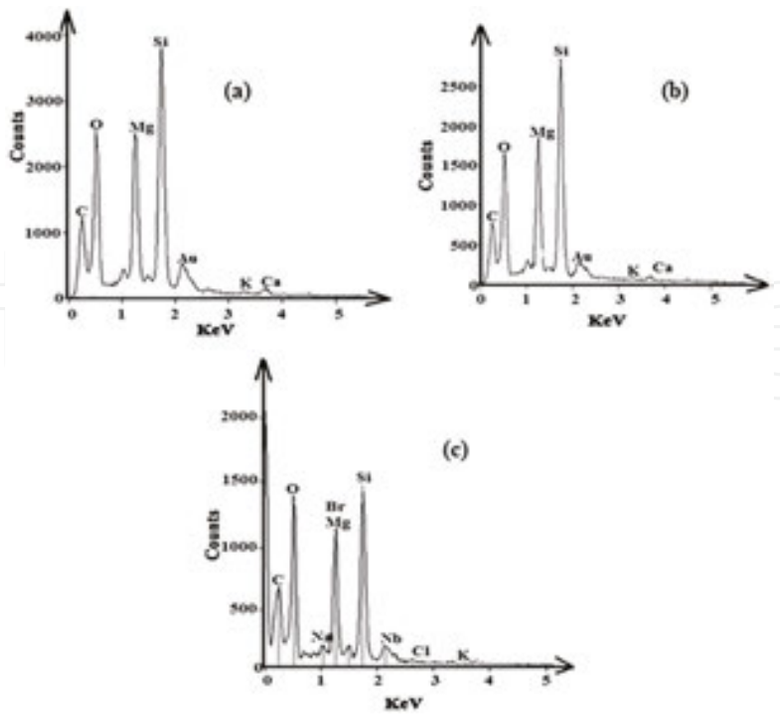

Figure 8. EDXA curves of electron beam irradiated samples of blend A. (a) 5 Mrad, (b) 15 Mrad and (c) 25 Mrad.

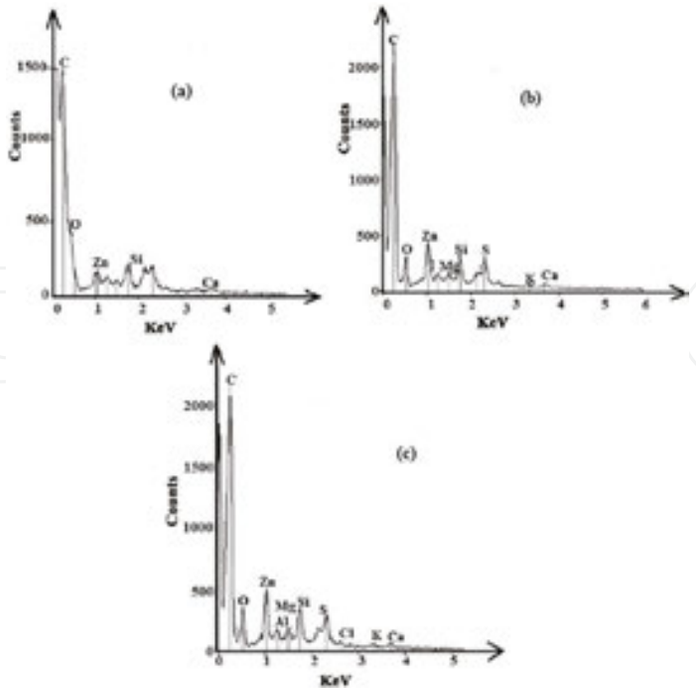

Figure 9. EDXA curves of electron beam irradiated samples of blend C. (a) 5 Mrad, (b) 15 Mrad and (c) 25 Mrad.

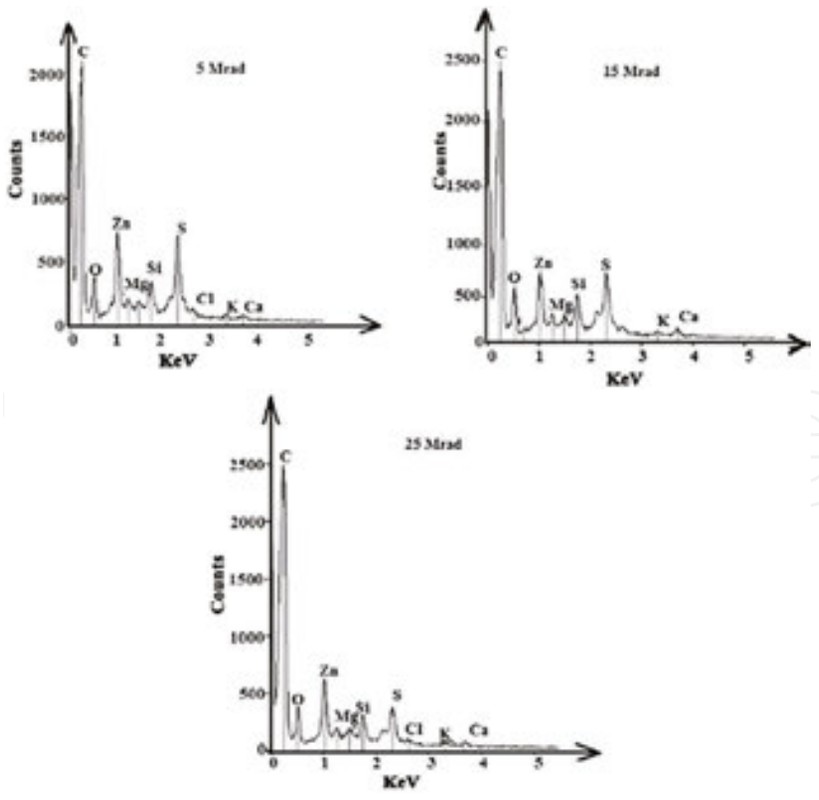

Figure 10. EDXA curves of electron beam irradiated samples of blend E. (a) 5 Mrad, (b) 15 Mrad and (c) 25 Mrad.

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Doses/elements	Carbon wt (%)		Silicon wt (%)		Oxygen wt (%)	
	A	B	A	B	A	B
0 Mrad	38.82	54.2	13.17	6.51	28.69	19.26
5 Mrad	39.35	40.21	14.91	11.32	27.6	30.3
15 Mrad	46.01	40.85	10.7	12.08	36.38	31.41
25 Mrad	39.92	38.87	11.17	11	36.72	32.07

Table 3. Inferences from EDXA curves of electron beam irradiated samples of SiR rich blends (A and B).

Doses/elements	Carbon wt (%)			Silicon wt (%)			Oxygen wt (%)		
	C	D	E	C	D	E	C	D	E
0 Mrad	71.42	84.99	89.13	2.11	0.76	0.8	11.84	3.78	3.14
5 Mrad	78.47	77.56	73.78	3.83	1.83	1.29	0.77	8.26	8.73
15 Mrad	82.43	81.66	79	1.26	1.62	1.63	5.21	6.86	6.69
25 Mrad	77.09	77.63	78.36	2.06	1.73	1.34	9.23	7.9	7.96

Table 4. Inferences from EDXA curves of electron beam irradiated samples of EPDM rich blends (D and E) and blend C.

5.4. Correlation of EDXA results with FTIR

The interpretations between EDXA and FTIR of the electron beam irradiated samples of SiR–EPDM blends are listed in **Tables 5–7**.

Inference from EDXA	5 Mrad	15 Mrad	25 Mrad
Increase in carbon content	C=C (alkene, stretch variable)/C=C (asymmetric, stretch, strong)/C–H (alkane)/=C–H (alkene) bending strong	C=C (alkene, stretch variable)/C=C (asymmetric, stretch, strong)/C–H (alkane)	C=C (alkene, stretch variable)/)/=C–H (alkene) bending strong
Decrease in silicon content except at 5 Mrad	Presence of Si–O–Si/Si–CH_3–CH_2 and Si–H		
Increase in oxygen content except 5 Mrad	Increase in Alcohol (–OH) free group content Occurrence of Si–O–Si group		

Table 5. Interpretation between EDXA and FTIR of the electron beam irradiated samples of blend A.

Inference from EDXA	5 Mrad	15 Mrad	25 Mrad
Increase in carbon content	Increase in CH_3–CH_2–CH		
	Appearance of C=C (asymmetric, stretch, strong)	Appearance of C–H (alkane), bending ,strong	Appearance of C=C (asymmetric, stretch, strong)/C–H (alkane), bending, strong/=C–H (alkene), bending, strong
Decrease in silicon content except at 5 Mrad	Absence of Si–H (amorphous Si)		
Decrease in oxygen content	Absence of acid COOH group		

Table 6. Interpretation between EDXA and FTIR of the electron beam irradiated samples of blend C.

Inference from EDXA	5 Mrad	15 Mrad	25 Mrad
Decrease in carbon content	Absence of acid (–COOH) group		
Increase in silicon content	Appearance of Si–H (amorphous Si) Increase in Si–CH_3–CH_2/Si–H content	Increase in Si–CH_3–CH_2/Si–H content	
Increase in oxygen content	Increase in alcohol (–OH)-free group content		

Table 7. Interpretation between EDXA and FTIR of the electron beam irradiated samples of blend E.

5.5. SEM analysis

Figure 11 (a1, b1, c1, d1, e1) and (a2, b2, c2, d2 , e2) are the SEM micrographs of the electron beam irradiated samples of SiR–EPDM blends exposed to 15 Mrad dose of electron beam irradiation for a magnification of 500 and 4000 respectively.

Figure 12 (a1, b1, c1, d1, e1) and (a2, b2, c2, d2, e2) are the SEM micrographs of the electron beam irradiated samples of SiR–EPDM blends exposed to 25 Mrad dose of electron beam irradiation for a magnification of 500 and 4000 respectively.

It is observed from **Figure 1**1(a1, a2) that the surface of SiR rich blend (A) has larger number of cracks. This may be due to the decrease in silicon content for 15 Mrad dose of electron beam irradiation (inferred from EDXA analysis), but the surface of blend B and EPDM rich blends (D and E) has smaller number of cracks. This is validated through the increase in oxygen and silicon concentrations (inferred from EDXA analysis). The surface of blend C has smaller cracks. This may be due to the reduction in oxygen and silicon concentrations. The availability of white particles on the surface of the blends B, D, and E may be due to the decrease in carbon content in them (inferred from EDXA curves).

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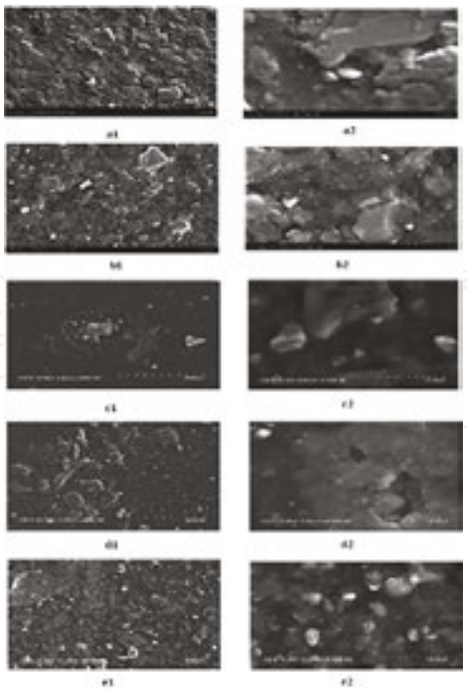

Figure 11. SEM micrographs of electron beam irradiated (15 Mrad) samples of SiR–EPDM blends. 11 (a1), 11 (b1), 11 (c1), 11 (d1) and 11 (e1) -500 magnification; 11 (a2), 11 (b2), 11 (c2), 11 (d2) and 11 (e2) -4000 magnification.

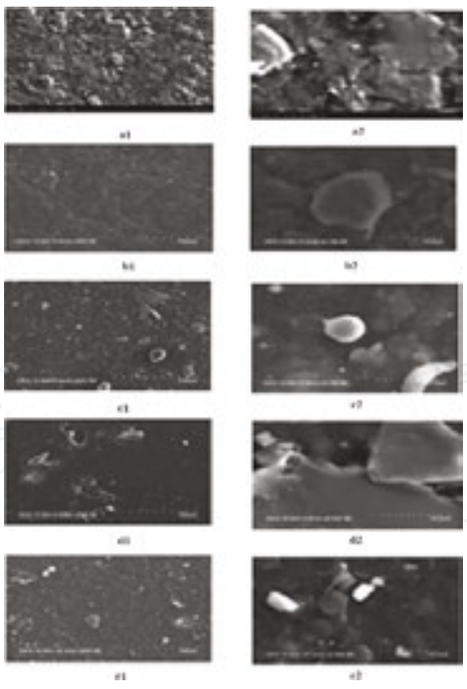

Figure 12. SEM micrographs of electron beam irradiated (25 Mrad) samples of SiR–EPDM blends. 12 (a1), 12 (b1), 12 (c1), 12 (d1) and 12 (e1) – 500 magnification; 12 (a2), 12 (b2), 12 (c2), 12 (d2) and 12 (e2) – 4000 magnification.

It is observed from **Figure 1**2(a1, b1) that the surface of SiR rich blend (A) has large number of cracks. This may be due to the decrease in silicon content for 25 Mrad dose of electron beam irradiation (inferred from EDXA analysis), but the surface of blend B and EPDM rich blends (D and E) has smaller number of cracks. This is validated through the increase in oxygen and silicon concentrations from EDXA analysis. The surface smoothness of blend C is moderate. This may be due to the reduction in oxygen and silicon concentrations.

6. Conclusion

The blend C is found to have the improved BDV and DS values for all doses of electron beam irradiation. Also a significant improvement in DC has been noticed at 5 Mrad in blends C, D, and at 5 and 25 Mrad in blend E respectively. A considerable improvement in DF has been observed at 15 and 25 Mrad in blend C and at 25 Mrad in blend D respectively. Hence, it is concluded that blend C and EPDM rich blends are found to have improved dielectric performance after the electron beam exposure.

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