Polym. Bull. (2012) 68:2323–2339
DOI 10.1007/s00289-012-0741-y

ORIGINAL PAPER

Mechanical, electrical, and thermal properties of irradiated low-density polyethylene by electron beam

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Received: 17 July 2011 / Revised: 18 January 2012 / Accepted: 15 March 2012 /
Published online: 23 March 2012

Abstract The effect of electron-beam (EB) irradiation on the mechanical, electrical, and thermal properties of low-density polyethylene (LDPE) was studied The LDPE was irradiated by using 3 MeV EB machine at doses ranging from 25 to 250 kGy in air at room temperature and analyzed for mechanical, thermal, and electrical properties. It was revealed by differential scanning calorimetry analysis that the crystallinity of the EB-radiated LDPE decreased slightly as verified by a marginal reduction in the densities, enthalpy, and melting points. Thermogravimetric analysis test showed that the thermal degradation of LDPE improved by increasing irradiation. The results obtained from both gel content and hot set tests, indicating whether the applicable LDPE has been properly cross-linked or not, showed that under the EB irradiation conditions employed, the cross-linking of the LDPE samples occur mainly in the amorphous region, and the cross-linking density at each irradiation dose depends almost on the amorphous portions of the LDPE. A significant improvement in the tensile strength of the neat LDPE samples was obtained upon EB up to 250 kGy with a concomitant decline in elongation at break. The results on the electrical properties revealed that the surface resistance, volume resistivity, and dielectric strength of the LDPE increase with irradiation dose and reaches a maximum at a 250 kGy irradiation dose. No considerable change of

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breakdown voltage, dielectric constant, and dielectric loss factor were observed with increasing irradiation dose. The enhancement in the heat deformation, hardness, and thermal aging properties of LDPE upon EB irradiation, suggests that irradiated LDPE is more thermally and mechanically stable than virgin LDPE.

Keywords Electron beam · Cross-linking · LDPE properties · Irradiation

Introduction

Low-density polyethylene (LDPE) has the largest annual production in the world today. The reasons for its popularity are its low cost and its unique physical properties. As a result, the low cost of LDPE, along with its excellent mechanical, processing, and electrical properties, makes it a good choice for many applications, such as wire and cable insulation [1]. However, unmodified LDPE has some shortcomings, such as low softening temperature, cold flow, and a tendency for stress cracking on contact with chemical reagents. These shortcomings can be overcome by the formation of intermolecular covalent bonds between the polyethylene chains [2]. Formation of a network structure changes thermoplastics into materials that no longer melt and will resist temperature-induced deformation or flow. Electron-beam (EB) irradiation of polyethylene improves the maximum permissible conductor temperature and maximum allowable temperature in the case of "over-current" and soldering [3]. Irradiated LDPE is also used at temperatures higher than their inherent softening point for a long period by choosing an adequate antioxidant [4]. This paper reports on investigation into the effects of absorption dose rate by EBs on physical, mechanical, electrical, and thermal properties of irradiated LDPE. These data will be useful for the application of LDPE in wire and cable insulation.

Experimental

Material

LDPE in the form of granules with grade LH0075, density of 0.921 g cm^{-3}, and a melt flow index (MFI) of 0.89 g $(10\ min)^{-1}$, was obtained from the Bandar Imam Petrochemical Company (Iran).

Sample preparation

LDPE granules were pre-heated for 5 min, and then compression molded according to ASTM D-1928 into sheets (with dimensions of 150 × 150 × 2 mm) at 150 °C under a pressure of 10 MPa for 3 min.

Irradiation

Irradiation of the LDPE sheets was carried out using an electron accelerator (Rhodotron TT200, Belgium) with energy of 3 MeV and beam current of 5 mA. The dose rate was 50 kGy/pass. The samples were irradiated ranging from 50 to 250 kGy.

Density measurement

The density of samples was determined according to ASTM D-1505 and measured using a Toyoseiki automatic densitometer model DH-100.

Gel content

The gel content of the EB-treated samples was determined using the solvent-extraction method according to the ASTM D-2765 method. The samples were refluxed with hot xylene for 20 h then the remaining insoluble sample dried in a vacuum oven to a constant weight.

Hot set test

The hot set test was carried out in a hot set oven Heraes UT 6050 HS. The samples in the form of dumbbells were placed in the hot set oven under a definite static load at 200 °C and the elongation between two marks was measured after 15 min.

Thermal analysis

Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) of the samples were carried out using Shimadzu Analyzer TGA-50 and DSC-50 from Kyoto Japan, respectively. The DSC and TGA tests were performed under nitrogen environment and the rate of heating and scanning were 10 and 20 °C/min.

Tensile strength and elongation at break

The tensile strength and elongation at break were determined according to ASTM D-638 on an Instron model 4411 testing machine (UK). The test procedure was carried out at a cross-head speed of 50 mm/min and room temperature conditions.

Hardness test

The Rockwell hardness test was carried out according to ASTM D785 using HA-101 Rockwell Automatic Digital Hardness Tester. The Rockwell hardness scale used was *L* (minor load 10 kg, major load 60 kg, and Indentor diameter 6.350 mm).

Heat deformation test

This test was carried out using a parallel plate plastometer. Sample measured 3.0–1.5 cm and thickness about 2.0 mm together with the parallel plates were heated at 120 °C for 30 min. Immediately after heating the sample was placed on a semicircular bar between the parallel plates of the measuring apparatus and 1 kg weight was applied on top of the sample. Heating was continued for another 30 min. Upon completion the thickness of the sample as shown at the dial gauge was recorded. Heat deformation was calculated from the different thicknesses before and after heating.

Dielectric properties

The dielectric constant, dielectric strength, dielectric dissipation factor, and dielectric loss tangent measurement were determined by model TRS-10T and 6303 (ANDO Electric Co., Japan and FRIBORG Co. Sweden) apparatus according to ASTM D-149 and 150 using an Ando insulation resistance measuring set at temperature of 23 °C and frequency of 1 MHz.

Resistivity properties

Surface and volume resistance were done according to ASTM D-257 and measured at room temperature by a CEAST model teraohmmeter.

Breakdown voltage

The breakdown voltage of the samples was done according to ASTM D-495 and measured by a CEAST model dielectric rigidity system.

Measurements of combustion characteristics

The limiting oxygen index (LOI) measurements were carried out in accordance with ASTM D 2863. The specimens used for the test are of dimensions $100 \times 6.5 \times 3$ mm. The cone calorimeter based on the oxygen consumption principle has been used to evaluate the combustion behavior of LDPE. The cone calorimeter test was performed according to ISO 5660 standard procedures. Each specimen, with dimensions of $100 \times 100 \times 3$ mm, was wrapped in aluminum foil and exposed horizontally to an external heat flux of 35 kW/m^2.

Thermal properties

To study the thermal aging property, irradiated LDPE were placed in a hot water bath at 95 °C for 1,500 h. The loss of tensile strength and elongation at break were measured at different irradiation doses (80–150 kGy).

Results and discussion

The gel content of irradiated LDPE was determined to evaluate the content of cross-linking produced by irradiation. Figure 1 shows the gel content values of irradiated LDPE at different irradiation doses of 120–250 kGy. It was concluded that the gel content increased with the augment of irradiation dose from 51 to 84 %. This study has also found that unirradiated LDPE to be completely soluble in hot xylene. However, by increasing the irradiation dose, the solubility was reduced significantly due to the formation of three-dimensional networks in the irradiated LDPE [5, 6]. The greater cross-linking clearly shows that irradiation-induced cross-linking reactions which occur mainly in the amorphous phase of the polymers [7] and some of the radicals are likely to be trapped in the crystalline phases [8]. The radical species formed within the amorphous regions can be utilized readily in cross-linking reactions, consequently those radicals trapped in the crystalline regions have a much lower mobility and a longer lifetime; such radicals can migrate slowly to the amorphous region, where they subsequently react [9].

Figure 2 illustrates the variations of the tensile strength of LDPE at various irradiation doses. The tensile strength of LDPE increased from 12 to 18.8 MPa. Cross-linking and chain scission took place simultaneously over the whole range of irradiation doses (0–250 kGy) and it seems that the rate of cross-linking was much more dominated [4]. The tighter network in the LDPE accounts for the high tensile strengths of irradiated LDPE due to the restricted movement of the molecular chains [5]. Consequently, the tensile strength values of LDPE increased after irradiation.

Figure 3 shows the relationship between elongation at break and irradiation dose of LDPE at different irradiation doses from 0 to 250 kGy. Unirradiated LDPE shows the highest value of elongation at break (561 %) whereas for all irradiated LDPE due to the formation of three-dimensional network structures and restricted movement of the molecular chain elongation at break decreased sharply to 382 % at 250 kGy [10, 11]. In general, increasing irradiation dose resulted in reduction in

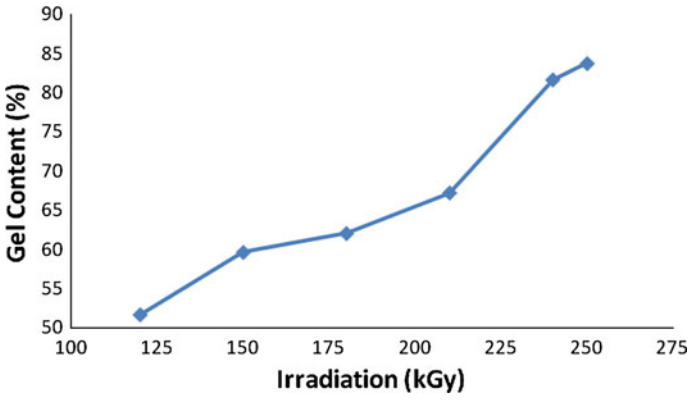

Fig. 1 Gel content of LDPE as a function of irradiation

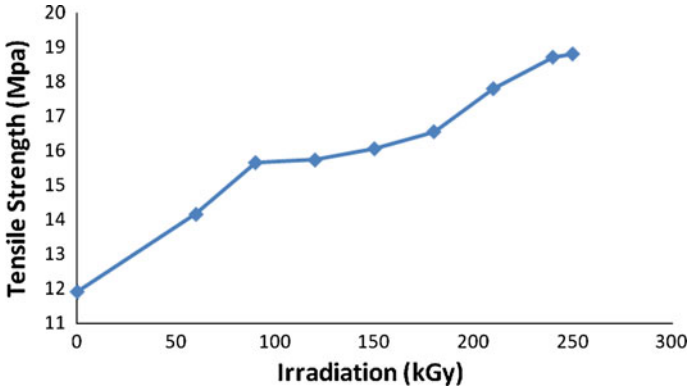

Fig. 2 Effect of irradiation on the tensile strength of LDPE

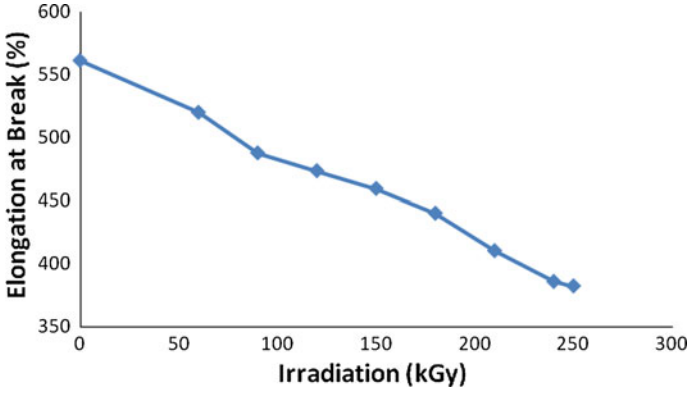

Fig. 3 Effect of irradiation on the elongation at break of LDPE

elongation at break of the LDPE [8]. As the dose increased more, cross-links were produced in the sample matrix which prevented the structural reorganization during drawing. [12].

Hot set test provides a quick check on cross-linking of samples and can be correlated with gel content. Table 1 shows that the hot set test values continuously decreased while irradiation dose values increased from 0 to 250 kGy. It was found that unirradiated samples failed the test immediately under elevated temperature 200 °C and load 20 N/cm^2 for 15 min. Irradiated LDPE at 150 kGy still failed in hot set test but at longer time. However, performance of the samples at high temperature improved as the dose increased. This is due to the increase of irradiation-induced cross-links as evident by the gel content [12]. It is remarkable that different polymer structures require different specific numbers of cross-linking for certain reduction of deformation caused by the load in the hot set test [9]. Thus, formation of adequate radiation cross-linking network in the polymer rendered the polymer to have better elongation at high temperature [7].

Table 1 Hot set test results at 200 °C under 20 N/cm^2 load for 15 min

Irradiation dose (kGy)	HS
0	Fails immediately
25	Fails immediately
50	Fails (30 s)
75	Fails (45 s)
100	Fails (2 min)
125	Fails (4 min)
150	Fails (4.5 min)
175	196 (%)
200	156 (%)
225	149 (%)
250	140 (%)

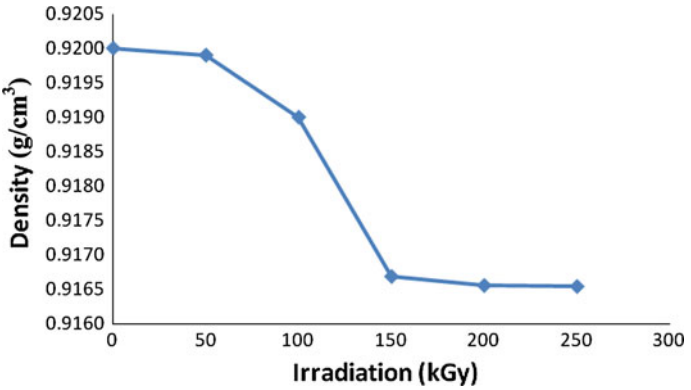

Fig. 4 Density of LDPE as a function of irradiation

Figure 4 shows the densities of the LDPE at various irradiation doses. The density of the unirradiated LDPE showed the highest value but increasing irradiation dose decreased the density values from 0.92 to 0.9165 g/cm^3 slightly. This reduction in density is attributed to reduction in crystallinity of LDPE upon irradiation [5, 9]. It was reported that increasing irradiation dose made the crystalline size smaller and consequently extended the amorphous region [10].

Figure 5 shows the hardness values of the LDPE with increasing irradiation dose. It was found that the hardness of LDPE increased gradually with increase the irradiation dose up to 250 kGy. The hardness value of unirradiated LDPE was measured 32 Rockwell L whereas increasing irradiation dose to 250 kGy increased the hardness value to 37.8 Rockwell L. Thus, it is evident that the enhancement in hardness of LDPE is due to the formation of radiation-induced cross-links as confirmed by the gel content and hot set tests [13]. In definition, hardness generally refers to the resistance of the material to local deformation [13]. The results content

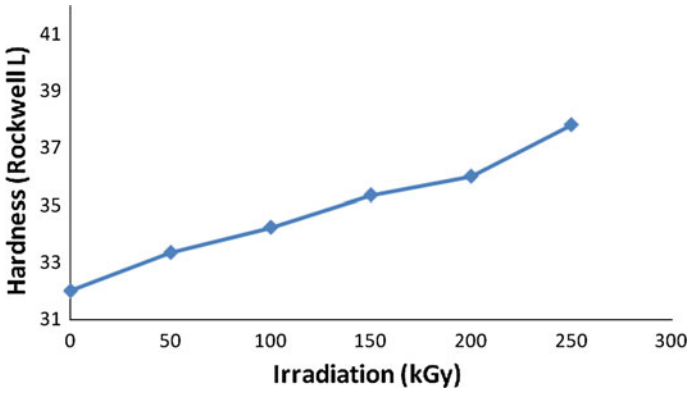

Fig. 5 Effect of irradiation on the hardness of LDPE

in Fig. 5 proved that the irradiated LDPE was more resistance toward local deformation and consequently increased the hardness [12].

Table 2 shows the tensile strength and elongation at break of irradiated LDPE after 1,500 h thermal aging in hot water. The thermal aging properties were determined by heating the irradiated samples in hot water at 95 °C for 1,500 h. The loss of elongation at break and tensile strength as a result of thermal aging were measured. Although the amounts of oxygen in water is low, but heat aging at 95 °C, caused thermo-oxidative degradation of samples during long-time immersion in hot water [8]. Table 2 shows the tensile strength and elongation at break of irradiated LDPE decreased significantly after 1,500 h in hot water. Whereas increasing irradiation dose from 80 to 150 kGy improved the thermal aging of LDPE.

Figure 6 illustrates the effect of irradiation dose on heat deformation of LDPE. It was shown that unirradiated LDPE underwent more than 98 % deformation after heating at 120 °C for 1 h. This observation attributed by the flowing of the uncross-linked LDPE which undergo melting at 120 °C [12]. It is also clear that increasing of irradiation dose from 0 to 250 kGy made the heat deformation percent decrease from 98 % at 0 kGy to 16.2 % at 250 kGy. Such enhancement of this heat deformation property was due to the formation of a cross-linking network, which restricted the flow of the molten sample to deform the LDPE [12].

The melting temperatures against irradiation doses are in Table 3. It is revealed that melting points of the unirradiated LDPE showed the highest value but increasing irradiation dose decreased the melting point values from 111 to 109.6 °C. Such reductions with increasing dose suggest the decrease in the crystalline regions of LDPE upon irradiation [4, 14]. In view of this, it has been reported that the crystalline regions in LDPE become impaired due to branching and cross-linking in the interfaces between the amorphous and crystalline regions [9]. As the cross-linking increased, the melting temperature decreased due to reduction of crystalline region of LDPE. Therefore, the melting temperature of LDPE decreases with irradiation dose.

Table 4 and Fig. 7 show the enthalpy of melting, crystallization, and crystallization temperature of LDPE at various irradiation doses. It is clear from Table 4

Table 2 Elongation at break and tensile strength of the irradiated LDPE after 1,500 h thermal aging in hot water

Dose	80 kGy			100 kGy			120 kGy			150 kGy		
Tests	0 h	1,500 h	Loss %	0 h	1,500 h	Loss %	0 h	1,500 h	Loss %	0 h	1,500 h	Loss %
Elongation at Break (%)	500	245	51	482	193	60	473	189	60	460	179	61
Tensile Strength (N/mm^2)	15.10	8.3	45	15.7	8.32	47	15.75	8.50	46	16.06	8.83	45

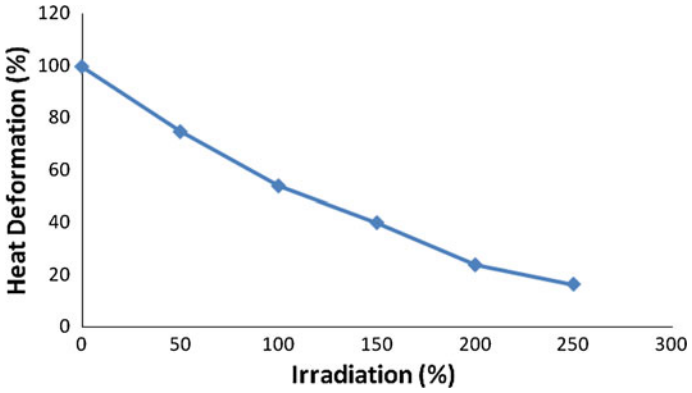

Fig. 6 Effects of irradiation on the heat deformation (%) of LDPE

Table 3 Melting point data for LDPE under various irradiation doses

Irradiation dose (kGy)	Melting point (°C)
0	111.0
50	110.8
100	110.2
150	110.1
200	110.0
250	109.6

Table 4 Enthalpy of melting and crystallization of the LDPE under various irradiation doses

Irradiation dose (kGy)	Enthalpy of melting (J/g)	Enthalpy of crystallization (J/g)
0	104.0	65.5
50	103.6	65.3
100	103.2	62.9
150	102.9	62.0
200	102.7	62.8
250	102.2	59.6

and Fig. 7 that the enthalpy of melting, crystallization, and crystallization temperature of LDPE reduced with increasing irradiation dose to 250 kGy. It was reported that irradiation below the melting point allows for cross-linking reactions which occur within the amorphous part of the polymer, whereas more radicals are likely to be trapped in the crystalline phases [6]. The radical species formed within the amorphous regions can be utilized readily in cross-linking reactions, whereas those radicals trapped in the crystalline regions have a much lower mobility and a longer lifetime; such radicals can migrate slowly to the amorphous region, where

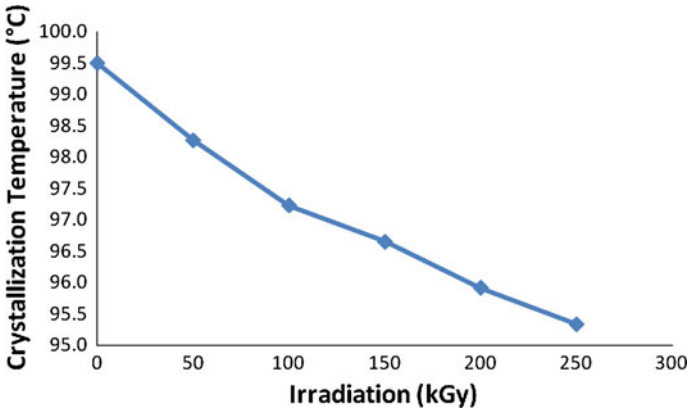

Fig. 7 Effect of irradiation dose on crystallization temperature of LDPE

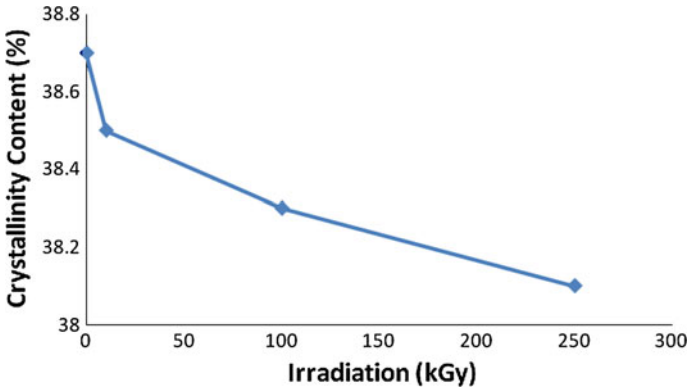

Fig. 8 Crystallinity content in the EB-exposed samples at different irradiation doses

they subsequently react [9]. The branching and cross-linking reactions of the polymer chains in the amorphous region may interfere with the crystalline region at the interface of these two regions [12]. Such interference caused slight reduction in crystallinity rate upon irradiation [4]. Thus, the enthalpy of melting and enthalpy of crystallization for LDPE decreased from 104 to 102.7 and 65.5 to 62 J/g, respectively.

Figure 8 shows the crystallinity content of LDPE at different irradiation doses. This figure revealed that increasing irradiation dose from 0 to 250 kGy made the crystallinity content of LDPE decreased slightly from 38.7 to 38.1 %. This decreasing in crystallinity content was due to increasing the amorphous region by increasing the irradiation dose and consequently it provides reduction in free volume of the amorphous regions [15]. These results also suggest that the EB irradiation causes only marginal reduction in crystallinity content of LDPE. On the other hand, the minor changes in melting point of the polymer could be the result of minor defects created with crystals by the irradiation [15].

Table 5 Thermogravimetry of LDPE as a function of irradiation

Polymer	Irradiation dose (kGy)	Weight loss (%) at different temperatures (°C)				T_{max} (°C)
		300	350	400	500	
LDPE	0.0	7.5	12.8	34.4	89.3	421.1
	50.0	7.0	8.8	21.1	84.2	429.3
	100.0	5.5	6.9	10.6	81.3	437.4
	150	5.5	6.6	10.1	78.5	440.8
	200	4.9	6.1	9.5	76.3	445.5
	250	4.4	5.7	8.7	74.2	451.4

Table 6 The properties of LDPE flammability

LOI	18
Heat flux (kW/m^2)	35
Time to ignition (s)	108
Maximum HRR (kW/m^2)	2,649
Total heat release (MU/m^2)	227
Average heat of combustion (MJ/kg)	38.281
Average smoke extinction area (m^2/kg)	801.42
Average CO (kg/kg)	0.004
Average CO_2 (kg/kg)	1.360
Average HRR 60 s (kW/m^2)	1935.583
TGA data (starting decomposition, DTG temperatures, and weight loss)	430 °C, 633 °C, and 98%, respectively
Results of horizontal burning test (burning time and rate of burning)	126 (s) and 35.71 (mm/min)

Table 5 reveals the TGA of LDPE at different irradiation doses. It shows that the irradiation increased the degradation temperature and had a positive effect on thermal stability of the LDPE. The temperature, at which degradation apparently started, increased with increasing the irradiation dose, indicating the thermal stability of LDPE increases upon EB irradiation. This is attributed to the higher degree of mobility and freedom of polymer chains in a linear uncross-linked structure than that in a cross-linked network structure of LDPE [5]. The radiation cross-linking and degradation reactions of LDPE mainly occur in the amorphous regions of semicrystalline LDPE during the irradiation [14]. It was reported that the higher amorphous content inside the LDPE is favorable to the radiation cross-linking of LDPE, especially for high dose rate irradiation [15, 16].

Measurements with a cone calorimeter test were done to check for changes in flammability properties. This method is the most advanced method for assessing materials reaction to fire. Table 6 shows the results of cone calorimeter and LOI experiments. LOI test is so simple and can be defined as the minimum concentration of oxygen in an oxygen–nitrogen mixture, required to just support downward

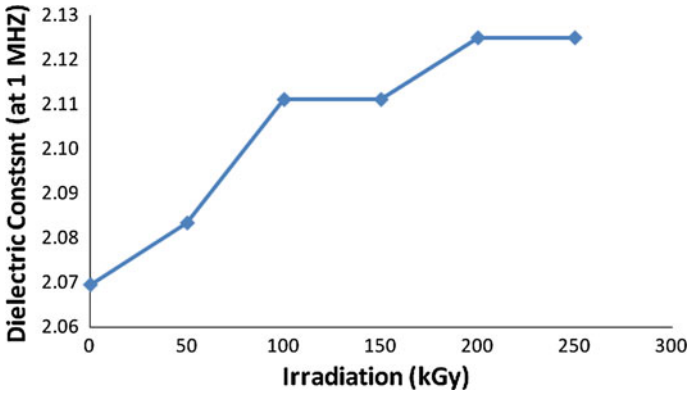

Fig. 9 Dielectric constant of LDPE at different irradiation doses

burning of a vertically mounted test specimen. The LOI of pristine LDPE is 18 (<21) so if LDPE is exposed to flame it easily inflames. Table 6 also shows values for time-to-sustained-ignition, peak heat release, time for peak HRR, and the total heat release. It summarizes effective heat of combustion, average HRR at 60 s, and average HRR at 120 s, smoke extinction areas (SEA), CO, and CO_2 yields. These results are consistent with previous studies [16, 17].

The results of dielectric constant test are shown in Fig. 9. It reveals that there is a slight increase in dielectric constant (from 2.070 to 2.125) with increasing irradiation dose to 250 kGy. Irradiation-induced reactions in polymers can result in formation of smaller molecules and dipoles which will lead to increase in the permittivity [5]. The permittivity of the polymer is representative of the various polarization phenomena that come into play when the polymer is subjected to an electric field. The overall polarization of LDPE is the sum of four terms: electronic, atomic, orientation, and space charge polarization, among them the first two are intrinsic in nature and for non-polar polymer are important [9].

Figure 10 illustrates the variations of volume resistivity at different irradiation doses. It shows the volume resistivity of LDPE increased from 83.6 to 85.7×10^{13} Ω cm with increasing irradiation dose to 250 kGy. It was shown that irradiation dose increased the cross-linking bonds and provided barrel points inside LDPE. These barrel points hint the charges to move between LDPE chains [18]. Therefore, increasing irradiation dose lead to grow the number of traps and cross-linking bonds inside the LDPE and it prevented the charges to move inside the LDPE and increased the electrical resistance [18]. Figure 10 clears that the value of volume resistivity increased with increasing irradiation dose.

Figure 11 shows the dielectric strength of LDPE at different radiation doses. It is observed that the dielectric strength did not change significantly with increasing irradiation dose. Dielectric strength of LDPE increased from 22.8 to 27.6 kV/mm with absorbing dose to 250 kGy. It was reported that an important factor which affect the electrical properties is the morphological state of polymers [18]. The irradiation changed the polymer network and the morphological state of the

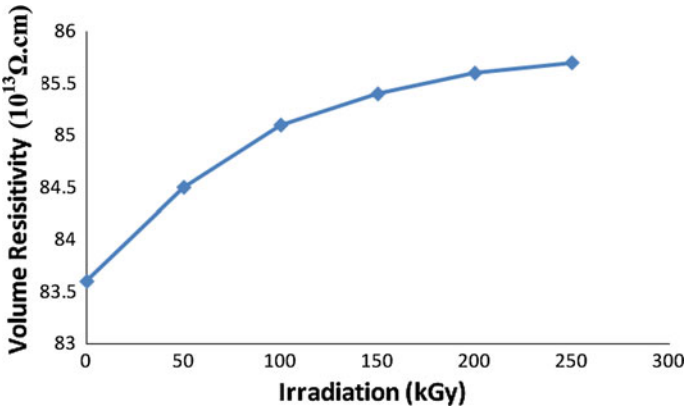

Fig. 10 Effect of irradiation on the volume resistivity of LDPE

polymer. It was mentioned earlier that increasing irradiation dose leads to increase amorphous region of irradiated LDPE. Therefore, an increase in the radiation dose caused an increase in the degree of cross-linking in amorphous area which barreled to prevent the electric breakdown path and hence increased dielectric strength values [19].

Figure 12 illustrates the variations of the surface resistance of LDPE with increasing radiation dose. It reveals that the surface resistance of LDPE changed at different irradiation doses. With increasing irradiation dose from 0 to 250 kGy, the surface resistance increased from 134×10^{12} to 138.1×10^{12} Ω. The presence of cross-linking points can be considered as barrels to prevent the charge movements between polymer chains and hence increased the electrical resistance of cross-linked sample [19]. It was also shown the growth of the number of traps in LDPE prevented the charge movement inside the LDPE and hence increased the electrical resistance [18]. In fact, cross-linking is a process occurring in the amorphous region [7]. Increasing irradiation dose provided chain scission and cross-linking inside

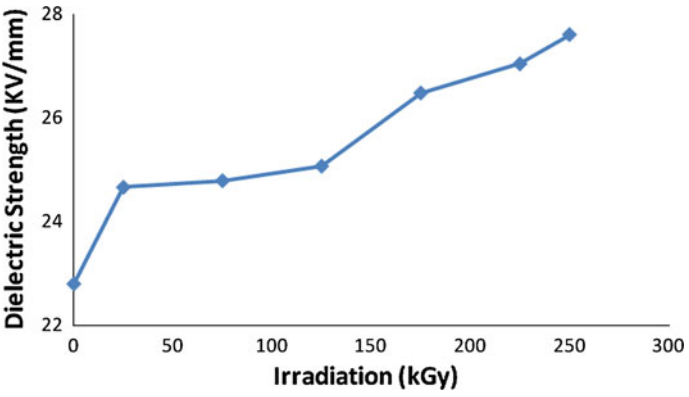

Fig. 11 Effect of irradiation on the dielectric strength of LDPE

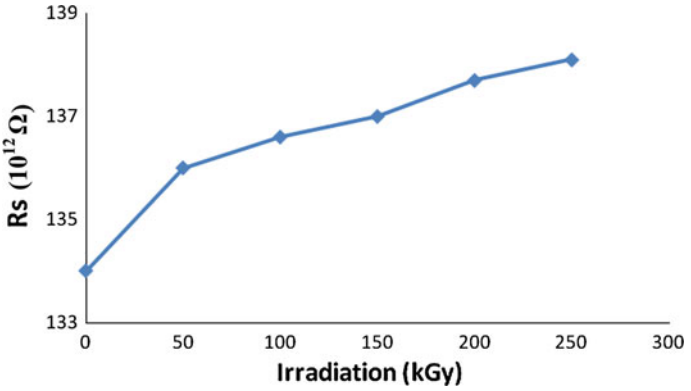

Fig. 12 Effect of irradiation on the surface resistance of LDPE

polymer matrix but issued results from gel content test showed the rate of cross-linking was more dominant than chain scission inside irradiated LDPE. Therefore, the higher the amorphous ratio, the more the cross-linking percentage. Consequently, it led to an increase in number of LDPE linked one another and which lead to increase in the surface resistance [19].

Figure 13 reveals the variations dielectric loss factor of LDPE at different irradiation doses. Increasing irradiation dose from 0 to 250 kGy changed the dissipation factor of LDPE from 0.00086 to 0.00190 at 1 MHz. It showed that the dielectric loss factor of all irradiated and unirradiated LDPE remained without significant change with increasing radiation dose. This property related to the inability of molecules in the insulating fluid to reorient themselves with an alternating electric field [18]. This ability was dependent on the temperature of the sample, the size of the molecules involved, and their polarity. It was also dependent on the frequency of the alternating field [18]. This suggests that incorporation of small amount of polar polymer affect the electrical properties of the non-polar polymer, whereas the radiation dose cannot create a considerable effect on the

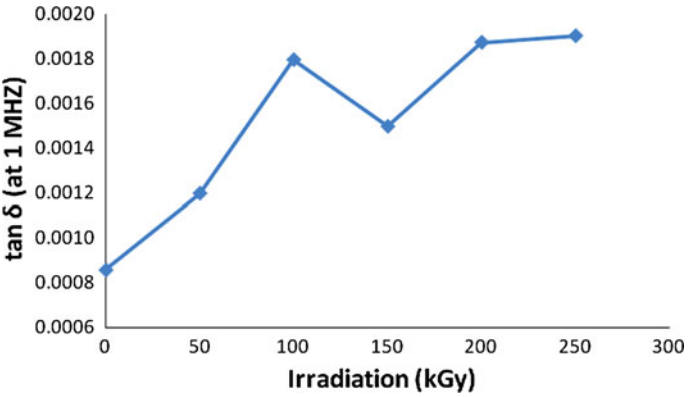

Fig. 13 Effect of irradiation on the dielectric loss factor of LDPE

electrical properties of such polymers [18]. Consequently, these minor changes might be related to the absence of polar groups and the homogeneous nature of the LDPE.

Conclusion

The EB irradiation found to cause considerable effect of the mechanical, electrical, and thermal properties of LDPE. The reduction in the densities, enthalpies of melting, enthalpies of crystallization, and melting points of LDPE with the increase in irradiation dose clearly indicated that the crystallinity of LDPE reduced upon irradiation. TGA test showed that the thermal degradation of LDPE improved with the increase in irradiation dose. The results obtained from both gel content and hot set tests showed that under the irradiation conditions employed, the LDPE sample cross-linked by the EB irradiation. A significant improvement in the tensile strength of the neat LDPE samples was obtained upon EB radiation up to 250 kGy with a concomitant decline in elongation at break. Results on that the surface resistance, volume resistivity, and dielectric strength revealed that the above electrical properties of the LDPE reaches a maximum at a 250 kGy irradiation dose. However, no considerable changes of breakdown voltage, dielectric constant, and dielectric loss factor were observed with increasing irradiation dose. The enhancement in the heat deformation, hardness, and thermal aging properties of LDPE upon EB irradiation suggests that irradiated LDPE is more thermally and mechanically stable than virgin LDPE.

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