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GAMMA-RAY IRRADIATION EFFECT ON MECHANICAL AND DIELECTRIC PROPERTIES OF VOLCANIC BASALT MINERAL REINFORCED LOW DENSITY POLYETHYLENE FILMS

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Abstract: The aim of the work is to study the mechanical and dielectric properties of low density polyethylene (LDPE) that can be modified by basalt mineral under low dose gamma irradiation. The composite films are prepared by a hot pressing method (15 MPa, 418 K, for about 10 min), and then irradiated by Co-60 gamma-rays in the dose range varied between 6 and 24 gray (Gy). Mechanical and dielectric properties of these samples are examined by means of stress-strain measurements and dielectric spectroscopy in the frequency range of 100 Hz –15 MHz at the room temperature. Experimental measurements evidence that the mechanical properties of LDPE were weakened by basalt additives and radiation. The tensile strength of 0.5 wt.% basalt additivity, which has an optimum value increased by 30 percent when compared to pure LDPE without radiation. When LDPE and LDPE/basalt composites were radiated between 6 to 24 Gy, the mechanical properties of both samples decreased at almost the same rate. The LDPE/ 0.5wt.% basalt composite sample without gamma-irradiation was the one with the highest mechanical strength. In the absence of radiation, the low (static) frequency real part of dielectric constant is increased with basalt additives. The dielectric strength reached a maximum value up to 20.0 wt.% basalt additive at 6 Gy dose. However, the polymeric structure of the sample was deteriorated.

Keywords: *basalt mineral, low density polyethylene, gamma irradiation, dielectric properties, mechanical properties*

Introduction

Polyethylene is an essential polymer commonly utilized in the fabrication of synthetic packaging materials. This polymer can be categorized as low-density and high density

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polyethylene (LDPE and HDPE), which are regularly characterized by their flexibility, clarity, toughness and the resistance to chemicals. They are plastics with excellent commercial and economic significance. Furthermore, LDPE shows some of attributes, such as fine dielectric properties, light weight thermal stability, and electrical insulation. It has a relatively low cost in comparison with other polymers that are used to for similar applications (Albano et al., 1999; Chibowski and Patkowski, 2007; Tuasikal et al., 2014; Moez et al., 2012).

Natural fillers, in the form particulate fillers and fiber fillers, have gained the attention of researchers for reinforcing materials in polymers, metals and ceramics. They are low cost, low density materials, and also renewable in a large amount when compared with the artificial fillers. Some previous studies about the use of natural fillers on the production of polymer composites have been studied for polypropylene composites (Toro et al., 2007), HDPE (Aigbodion et al., 2015), and recycled low density polyethylene (RLDPE) polymer composites (Olumuyiwa et al., 2012). Many researchers have developed blends and composites by using polymers (Sunilkumar et al., 2012; Prusty et al., 2002).

Basalt mineral has been a categorized material of for military research and defence applications (Colombo et al., 2012; Pavlovski et al., 2007). In recent years, there is a growing research interest in the usage of these minerals in industry out to their improved mechanical attributes. Basalt is chemically rich in oxides of iron, silicon, sodium, potassium, calcium and, magnesium along with traces of alumina. The basalt chemical content may differ based on the geographical distribution. There is a large amount of this mineral which takes up to 33% of the earth's crust (Dhand et al., 2014). Basalt has good mechanical wear resistance and abrasion properties, it shows good response to fire, heat and noise insulation, moreover, it is a resistant material from the chemical point of view. Basalt is an appropriate material for polymer composite, with regards to synthetic materials, such as silica, zircon, alumina etc. The basalt contains very hard phases like augite and has a fine grain structure (Akinci et al., 2012; Znidarsic-Pongrac et al., 1991).

Nowadays, radiation methods are widely used in improving the physical and chemical properties of the polymeric material for high technology applications (Ferreira et al., 2005; Radwan, 2009). Ionizing radiation lead to irreversible changes at the macromolecular scale in the structure of the target material. (Abdul-Kader et al., 2012; Bielinski et al., 2004). Moreover, the gamma irradiations of polymer may lead to irreversible modifications in their physical properties such as chain scission, radical composition, bond breaking, creation of unsaturated bonds, intermolecular cross-linking, free radical formation, hydrogen release and some oxidation reactions. Some covalent bonds of polymers due to irradiation are broken and free radicals are formed which, in the next step, react to new chemical bonds (Belkahla et al., 2013; El-Ashhab et al., 2013; Labouriau et al., 2015; Raghu et al., 2015; Tayel et al., 2015).

This work has been devoted to develop the mechanical and dielectric properties of LDPE by both doping different rates of basalt and low-dose gamma irradiation. From this viewpoint, the mechanical and dielectric properties of the LDPE/basalt composites have been investigated by means of rate of doping concentration and gamma irradiation.

Experimental

Materials

Low density polyethylene (density is from 0.91 to 0.94 g/cm^3, melting temperature 98–115 °C, melt flow index 0.26, and processing temperature at 134–210 °C) was obtained from Petkim Industry (in Turkey). They are obtained from Van in East of Turkey. The chemical analyses of the basalt powders were obtained via X-ray fluorescence (XRF) system. Also, operating conditions of the Philips PW-2400 XRF instrument were fixed at 60 kV and 50 mA. Volcanology and petrology of the volcanic areas are analyzed in detail by Karabul et al. (2015). The chemical distributions of the basalt sample are shown in Table 1.

Table 1. Chemical composition of basalt sample

Comp.	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5	Total
wt.%	41.668	2.080	13.106	13.823	0.192	9.754	10.602	5.261	1.737	1.777	100.0

The samples were ground with IKA A11 basic analytical mill and were milled in rotational speed of 20,000 rpm. Milling was performed with a 250 cm^3 chamber size using 30 mm steel balls, grinding media filling ratio of 25%. The grinding time for ball milling were used: 15 min. The particle size distribution of basalt has been determined by Mastersizer, Malvern Hydro 2000MU model as previous studies (Cinar et al., 2016). As seen from Fig. 1, 90% of the particle size is smaller than 58.438 μm, 50% of the particle size is smaller than 17.648 μm, and 10% of the particle size is smaller than 2.013 μm.

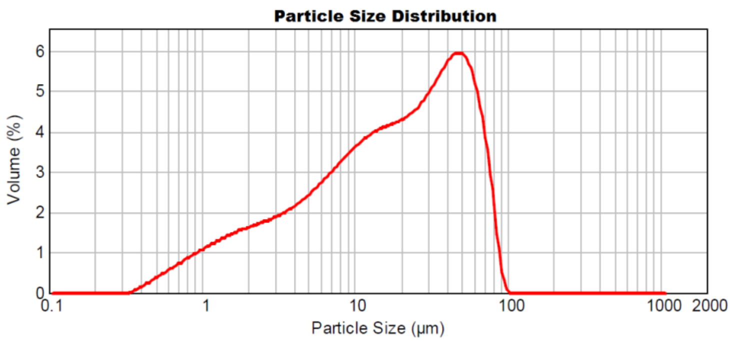

Fig. 1. Particle size distribution of basalt

To examine effects of basalt additive percentages (0.5, 1.0, 5.0, 10.0, 20.0 wt.%) on the mechanical and electrical properties of LDPE and the composites, basalt was added LDPE powder. The prepared composites were compression molded in an electrically heated press. The hot press procedures included pre-heating at 418 K for 20 min, flowed by compressing for 10 min at the same temperatures with a thickness of 51–100 µm. The films were cooled in a flow of cold water immediately after removal from the hot press. The samples were inserted into firmly capped flasks. Afterwards, these films were exposed to gamma rays produced by a ^{60}Co source at room temperature. The dose rate was 6 Gy/h detected by dosimetry with a radiochromic thin film dosimeter. Equal doses between 6 and 24 Gy were applied to the films. The error in dose can be estimated at 1%.

Fourier Transform Infrared Spectroscopy Analysis (FTIR)

Spectral analysis was performed on a Perkin Elmer Spectrum 400 FTIR spectrometer. All spectra of LDPE/basalt composite films were taken in the 4000-400 cm^{-1} range, with a resolution of 2 cm^{-1} after 4 scans. The measurements were performed in the transmittance mode. The variation in transmittance before and after exposure was compared, and the peaks were analyzed to determine the chemical change.

Mechanical Measurements

The mechanical properties of LDPE/Basalt composites were evaluated using the Lloyd Instruments LF Plus Single Column Universal Materials Testing Machine according to ASTM D638-10 standard with a crosshead speed of 50 mm/min at 23±3 °C to determine tensile strength, Young's modulus, percentage strain and energy at the break etc. The thickness of LDPE/basalt composite films was measured by using a Mitutoyo micrometer.

Dielectric Measurements

The dielectric properties of the basalts were carried out by using a two-point probe arrangement. These experiments with a high precision (approximately 0.17% typ.) of the samples were taken by an HP 4194A Impedance Analyzer at room temperature. The dielectric parameters were measured in the frequency range of 100 Hz–15 MHz.

Result and discussion

FT-IR Analysis

The Fourier Transform Infrared (FTIR) spectra of the non-irradiated and irradiated of LDPE and LDPE/basalt composites are given in Fig. 2. It is well-known that polymer such as polyethylene or polypropylene occurs cross-linking of polymeric chains by irradiation (Croonenborghs et al., 2007; Sohn et al., 2008; Fintzou et al., 2007; Kim et al., 2010). The spectrum of the pure LDPE shows the main characteristic peaks: C–H stretching vibrations at near 2950 cm^{-1}, C–H scissoring vibration at near 1460 cm^{-1} and CH_2 rocking vibration mode at 720 cm^{-1} (Alkan et al., 2013; Suljovrujic, 2010). In Fig. 2a, the spectrum for LDPE is similar to those reported in this literature (Alkan et al., 2013; Suljovrujic, 2010; Suljovrujic et al., 1999; Alberola et al., 1992a,b; Boydag et al., 2005).

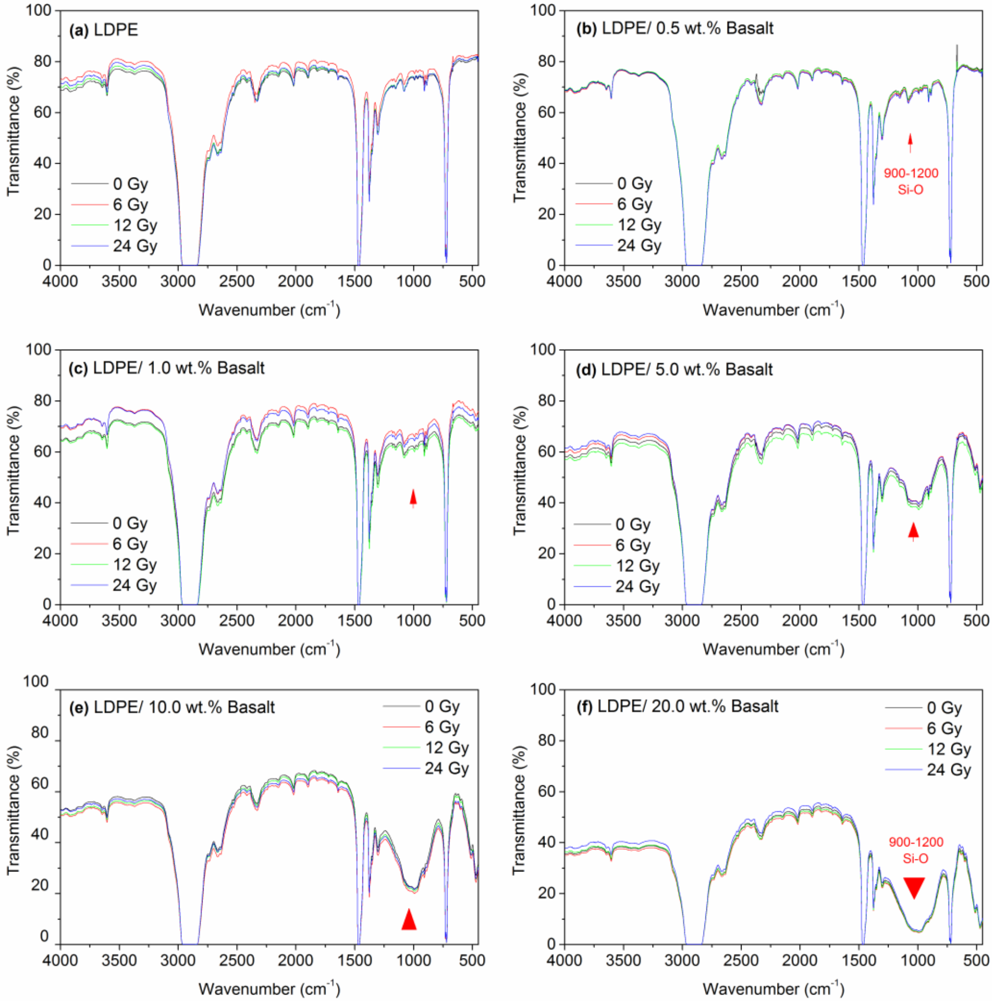

Fig. 2. FT-IR spectra of LDPE and LDPE/basalt composite samples irradiated with different radiation doses

The LDPE and LDPE/basalt spectra are displayed in Fig. 2b–f for different absorbed doses. Comparing LDPE/basalt FT-IR spectra with the example from previous literature (Alkan et al., 2013; Alberola et al., 1992a,b; Boydag et al., 2005; Suljovrujic, 2010; Suljovrujic et al., 1999), one might say that the same functional groups are present, but their respective peaks are of slightly different proportions. After irradiation, the methylene peak at 720 cm^{-1} of pure LDPE and LDPE/basalt composites has been changed. The irradiated samples by using Perkin Elmer FTIR Spectrum Analyser 5.0.1 software show that area of the carbonyl band increased between 1600 and 1800 cm^{-1}. Also, the new peaks (due to basalt additive) observed at asymmetric stretching of Si–O occurs between 900 and 1200 cm^{-1} (Jaret et al., 2015) in the spectra of basalt additive from 5.0 wt.% to 20.0 wt.% (Fig. 2d-f). Also, the transmittance percent is decreased with the basalt additives. These rates are 70% (Fig. 2a) and 40% (Fig. 2f) for pure LDPE and 20.0 wt.% basalt additivity, respectively.

Mechanical Analysis

The mechanical properties of basalt filled LDPE composites are dependent on numerous factors amongst which includes secondary particulate loading, size, size distribution, shape, irradiation, and aspect ratio (Mayo et al., 2004; Boydag et al., 2005). Table 2 shows the mechanical parameters of non-irradiated LDPE-based composites reinforced with basalt. Figure 3 shows, the variation of tensile strength and stress-percentage strain (non-irradiated) curve of LDPE and LDPE/basalt composites.

Table 2. Mechanical characteristic of non-irradiated LDPE and LDPE/basalt composites

Samples	Tensile Strength (MPa)	Young's Modulus (MPa)	Percentage Strain at Break	Energy to Break (mJ)
LDPE	8.69±0.38	9.06±1.40	736.66±44.42	12.57±0.52
LDPE/0.5 wt.% basalt	11.38±0.29	9.87±2.33	1001.5±23.63	14.97±0.43
LDPE/1.0 wt.% basalt	9.68±0.44	16.94±4.35	744.35±32.21	10.18±0.37
LDPE/5.0 wt.% basalt	6.07±0.30	22.44±3.72	266.99±46.84	2.46±0.36
LDPE/10.0 wt.% basalt	4.67±0.35	32.58±3.81	189.71±25.14	0.96±0.09
LDPE/20.0 wt.% basalt	5.63±0.42	33.62±4.67	214.79±36.80	1.83±0.16

As is seen from Fig. 3a, the tensile strength is increased from 8.69 to 11.38 MPa, when increasing basalt content from 0 to 0.5 wt.%. Although the basalt content is increased from 0.5 wt.% to 20.0 wt.% values in composite, the tensile strength is greatly decreased as compared to pure LDPE. Furthermore, the modified LDPE's, with 0.5 wt.% basalt, increased the percentage strain by approximately 31%, compared to value of pure LDPE (Fig. 3b). It is possible that the basalt content of 0.5 wt.% as well as the homogeneous distribution contributes to the observation of reinforcement effects. The mechanical durability improvement of LDPE/ 0.5 wt.% basalt composites can be attained, when the basalt in LDPE/basalt composite can withstand the propagation of cracks with increasing basalt content at mechanical stress as indicated by the increase in the tensile strength.

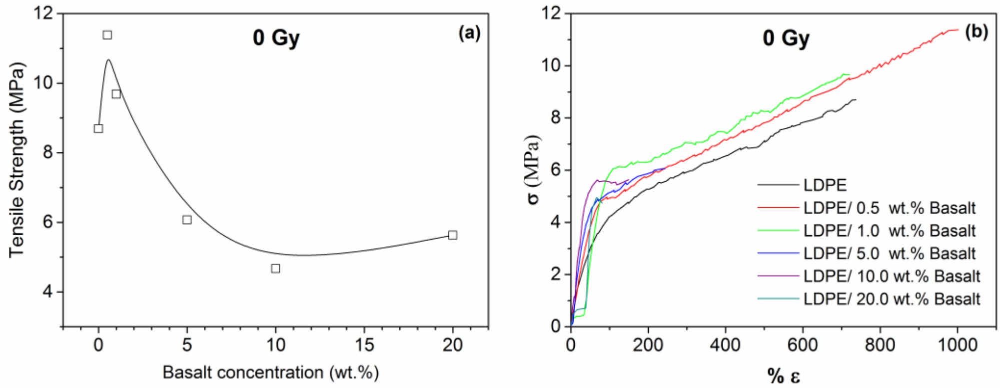

Fig. 3. The variation of (a) tensile strength and (b) stress-strain curve of non-irradiated LDPE and LDPE/basalt composites

Figures 4 to 6 show that tensile strength, Young's modulus, percentage strain and energy at the break parameter values from mechanical tests for different gamma irradiation doses. In the analysis of tensile strength and Young's modulus from mechanical tests (Fig. 4), most of the measured data are observed that reduced the measured value when applying gamma radiation dose from 6 to 24 Gy.

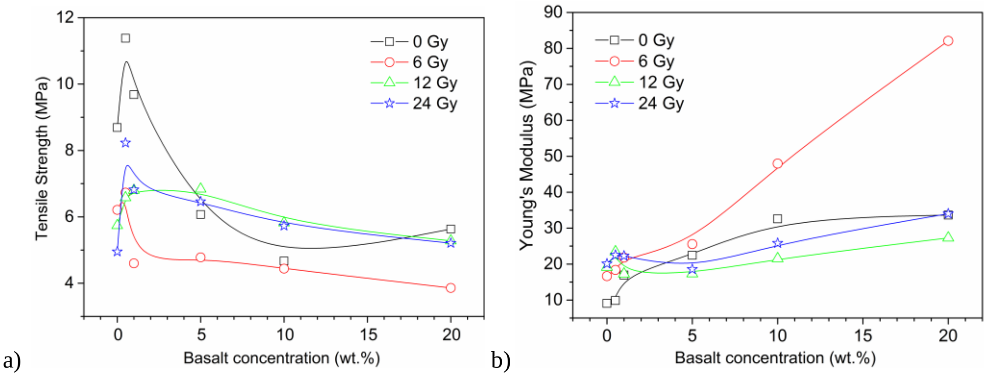

Fig. 4. Variation of (a) tensile strength and (b) Young's modulus of LDPE and LDPE/basalt composites under different gamma irradiation doses

When gamma irradiation caused a decrease in the mechanical strength of sample, a minimum value of the strength occurs at the 6 Gy dose. Figure 4a shows that the tensile strength decreases for 6 Gy. Also, under 6 Gy dose, Young's modulus is significantly increased in the irradiated samples with increasing basalt additives (Fig. 4b). Especially, for 20.0 wt.%, this value reaches about 80 MPa (for pure LDPE about 10-20 MPa) which corresponds to a maximum rigidity of the sample. The increasing of rigidity leads to a minimum percentage strain at break for materials, as seen in Fig.

5a. This is out to the variation of the molecular structure in the sample which also shows less energy needed for breaking (Fig. 5b).

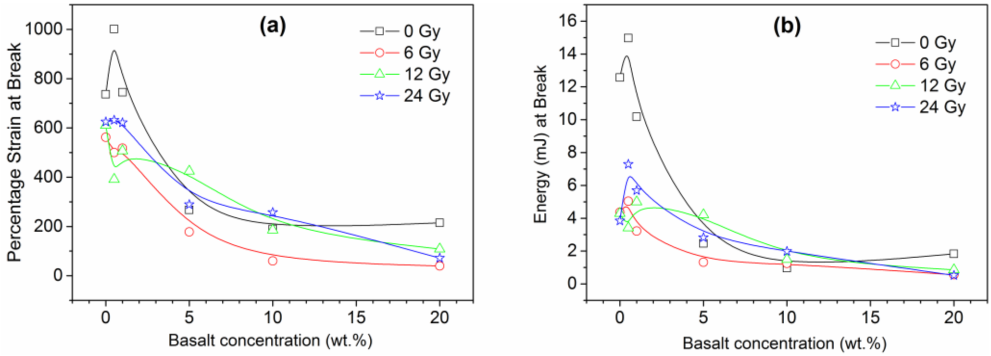

Fig. 5. Variation of (a) percentage strain at break and (b) energy at break of LDPE and LDPE/basalt composites under different gamma irradiation doses

Also, there is a limiting effect on the cross-linked band due to gamma irradiation. In the work by Suarez et al., 2002, a research about pure LDPE showed that crosslinking occurs for lower doses and molecular chain scission for higher dose. The effects in molecular structure of mechanical degradation and the degree of breaking covalent bonds in molecular structure increased with the increment of absorbed dose.

Dielectric Analysis

Dielectric spectroscopy is a non-destructive and powerful experimental technique for the characterization of electrical properties of materials. This technique allows to measure under a wide frequency and temperature range. The dielectric constant and energy loss are essential parameters for dielectric material science. The frequency dependent dielectric properties of materials can be described with regards to complex dielectric constant (ε). The complex dielectric constant is given by

$$\varepsilon^*(\omega) = \varepsilon'(\omega) + i\varepsilon''(\omega) \,. \tag{1}$$

Here, ω, $\varepsilon'(\omega)$, and $\varepsilon''(\omega)$ are the angular frequency, the real part of complex dielectric constant, and the imaginary part of the complex dielectric constant, respectively. Therefore the complex conjugates of dielectric can be written as (Cole and Cole, 1941),

$$\varepsilon'(\omega) - i\varepsilon''(\omega) = \varepsilon'_\infty + \frac{\varepsilon'_s - \varepsilon'_\infty}{1 + (i\ \omega\ \tau)^{(1-\alpha)}} \,. \tag{2}$$

In this expression, ε'_s and ε'_∞ are low (static) and high frequency dielectric constants. The ε'_s and ε'_∞ are shown in Fig. 6.

When LDPE is doped by different weight percentage of basalt, the real part of the dielectric constant (ε'_s) increases at low frequency. This increment reaches the maximum point at 20.0 wt.% without gamma-irradiation, as seen in Fig. 6. For pure LDPE, the ε'_s increases with radiation dose. But, the effect of radiation is not observed on dielectric constant for the sample with basalt additives.

The dielectric strength $\Delta\varepsilon'$ is the difference between the dielectric values at low and high frequencies. The dielectric strength $\Delta\varepsilon'$ is given by

$$\Delta\varepsilon' = \varepsilon'_s - \varepsilon'_\infty \tag{3}$$

Equation (3) is been used to calculate dielectric strength ($\Delta\varepsilon'$) and given in Table 3. Under gamma irradiation between 6 and 24 Gy doses, the dielectric strength shows different behaviors for 20 wt.% basalt samples. There is a sharp increase in 6 Gy (See Fig. 8). On the other hand, it is observed that the $\Delta\varepsilon'$ does not change significantly with other additives as well as dose conditions.

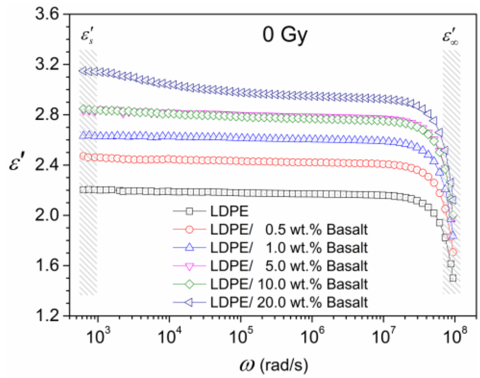

Fig. 6. Frequency dependency of the real part of dielectric constant of LDPE and LDPE/basalt composites without gamma irradiation

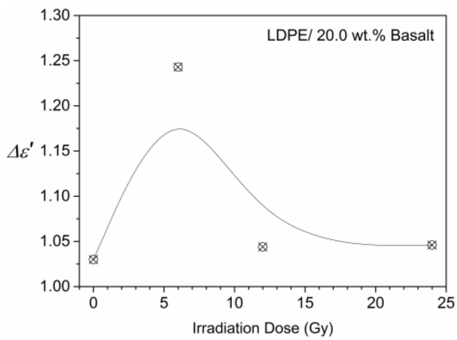

Fig. 7. Variation of dielectric strength in LDPE with 20.0 wt.% basalt under different gamma irradiation doses

Table 3. Dielectric parameters (ε'_s , ε'_∞ and $\Delta\varepsilon'$) of LDPE and LDPE/basalt composites under different gamma irradiation

Applied gamma dose	Parameter	LDPE	LDPE/ 0.5 wt.% basalt	LDPE/ 1.0 wt.% basalt	LDPE/ 5.0 wt.% basalt	LDPE/ 10.0 wt.% basalt	LDPE/ 20.0 wt.% basalt
0 Gy	ε'_s	2.203	2.468	2.644	2.85	2.855	3.152
	ε'_∞	1.500	1.706	1.836	1.988	1.994	2.122
	$\Delta\varepsilon'$	0.703	0.762	0.808	0.862	0.861	1.030
6 Gy	ε'_s	2.551	2.491	2.752	2.641	2.683	3.112
	ε'_∞	1.751	1.696	1.942	1.836	1.836	1.869
	$\Delta\varepsilon'$	0.800	0.795	0.810	0.805	0.847	1.243
12 Gy	ε'_s	2.749	2.802	2.779	2.698	2.750	2.912
	ε'_∞	1.960	1.968	1.905	1.861	1.911	1.868
	$\Delta\varepsilon'$	0.789	0.834	0.874	0.837	0.839	1.044
24 Gy	ε'_s	2.653	2.767	2.696	2.791	2.660	3.138
	ε'_∞	1.854	1.924	1.881	1.856	1.711	2.092
	$\Delta\varepsilon'$	0.799	0.843	0.815	0.935	0.949	1.046

Conclusion

In this paper, the effects of gamma irradiation on the dielectric and mechanical properties of LDPE and LDPE/basalt composites have been studied. The mechanical properties of LDPE film degraded while increasing the basalt additive, but the tensile strength improved significantly when increasing basalt content from 0 to 0.5 wt.%. Similar behavior was observed in the other mechanical properties of these composites. The mechanical properties of pure LDPE and LDPE/basalt composites were significantly influenced by the effect of gamma radiation (6, 12 and 24 Gy). Additionally, the results indicate that gamma irradiation causes deformation of molecular structure on the composites such as crosslinking for low doses. This modifies the molecular structure of the composites and changed their mechanical properties. Especially, the rigidity obtained a maximum when the effect of gamma irradiation was 6 Gy doses obtained the maximum rigidity. When the sample is exposed to 6 Gy dose, the polymeric structure of LDPE/20.0 wt.% basalt sample is deteriorated. The addition of 0.5 wt.% basalt content into non-irradiated LDPE and gamma radiated LDPE can effectively enhance the mechanical strength of these composites under the effect of gamma radiation.

The real part of the dielectric constant (ε'_s) of non-irradiated LDPE/basalt composites is improved by increasing basalt content. However, the real part of the dielectric constant (ε'_s) of irradiated LDPE/basalt composites is not improved. On the other hand, it is observed that the dielectric strength $\Delta\varepsilon'$ does change significantly under 6 Gy dose for 20.0 wt.% basalt additive. If LDPE/basalt composites are produced under 6 Gy dose for 20.0 wt.% basalt additive, a good insulating material can be obtained. Furthermore, foods and pharmaceutical products could be preserved and sterilized by using this irradiation. These composites may also be used for specific packaging applications.

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