

Designing of LDPE/fly ash/ expanded graphite sheet for electrostatic charge dissipation application

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ABSTRACT

The paper aims to explore the utilization of industrial waste fly ash as a filler material into low density polyethylene (LDPE) polymer matrix with / without expanded graphite to optimize the electrostatic charge dissipative (ESD) properties as injection molded sheets. Homogeneous mixing of composite mixture has been carried out in a single screw extruder via melt blending and designed to sheet form by using an injection molding machine. The mechanical and thermal properties of the composite sheet depend on the formulation of composite material. The presence of fly ash particles and fly ash/ expanded graphite in the polymer system allows the composite sheet to acquire good mechanical and electrostatic charge dissipative properties. Static voltage decay rate and decay value measurement were carried out for LDPE and LDPE/fly ash/expanded graphite composite sheet. LDPE/fly ash/expanded graphite composite sheet having high percentage of expanded graphite showed good electrostatic charge dissipative properties. Further, structural analysis, surface morphology, thermal stability and mechanical properties have been explored by XRD, SEM, TGA and tensile testing. Copyright © 2016 VBRI Press.

Keywords: Polymer; composite; tensile strength; electrostatic charge dissipation.

Introduction

Polymers make up a fundamental piece of our daily lives. On the basis of application, polymers are classified as commodity and engineering polymers. In fact, polymers can be molded to different forms very easily and cheaply as compared to metals. Low density polyethylene (LDPE) is a most generally utilized commodity polymers worldwide which is affordably available. This polymer is characterized with increased toughness at lower temperatures, easy processability and chemically inert at room temperature [1]. Strength of LDPE and high specific modulus can be utilized as a decent possibility for a strengthening component in composites [2]. LDPE is mainly utilized for packaging and non-packaging film and sheet applications. Non-packaging application includes various polymer products like carry-out bags, garbage bags, industrial sheeting, construction and agricultural films. This polymer can also be used for insulation and sheathing of wires and cables. Polymer product like film, sheets, filaments and cables have capability to retain static charges on their surface owing to large surface area. It is well known that static charge accumulation differs from material to material, but static charge build up leads to many undesirable consequences especially in food and electronic packaging industry. Failure to disseminate static charge results in damage and many other problems [3]. Issues postured by accumulation of static charges on the surface of the sheets are attraction of dirt and dust particles [4] in food packaging which is completely unhygienic and unwanted.

Electrostatic charge accumulation may also cause shock, damage, explosion and fire hazard when come in contact of flammable materials. In addition to this, adhesion of sheets stacked on top of each other which results in tearing on separation, accounting for a lot of material wastage. Researchers have attained attention for the development of anti-static agents, conducting fillers and additives [5] to immune polymers from static charge accumulation. Conduction of electricity on the surface or through volume refers to Electrostatic charge dissipation (ESD) property of a material [6]. Retention or enhancement of mechanical and thermal properties of LDPE and bestowing ESD property would solve the above negative implications. Thus the challenge is to change naturally insulating materials into a product with an effective antistatic performance. Several scientists have carried out their investigation on Polymer/graphite composites for this application. Study of the effect of graphite particle size on the conductivity of polymer/graphite composite was done by Nagata *et al.* [7]. Zheng *et al.* reported conversion of HDPE from insulator to conductor on addition of expanded graphite with a slight increase in mechanical properties [8]. Krupa *et al.* studied electrical and thermal conductivity of PE/graphite composite [9]. Taking into account the results, expanded graphite seems appropriate filler. An alternate issue being confronted in society is the dumping of a noxious waste known as flue ash [10, 11] or fly ash. Fly ash is a residue acquired from power plants due to burning of coal as a means to generate electricity. Fly ash consists of alkali

mixtures, metal oxides of iron, aluminum, silicon and small amount of potassium, sodium, calcium and magnesium oxides. The composition of fly ash depends on coal composition and its processing [12]. Besides being an air contaminant, it is associated with many health problems [13]. Its disposal by land filling has become an issue. Despite being a waste product, it exhibits some valuable characteristics like filling ability, low density, heat resistance, chemical inertness and large specific surface [15, 16]. One of the major aims of research is to exploit industrial waste like fly ash in useful applications [17]. Fly ash has been reviewed as a property enhancing substance by various researchers [18].

In the present research article, fly ash has been utilized as filler in the LDPE polymer matrix along with expanded graphite. The aim of using Fly ash is the waste material utilization, thus tackling the problem of land filling and also reducing the cost of commodities made up of LDPE. Since, ESD materials should have better mechanical properties, and moderate electrical conductivity so expanded graphite is used to make LDPE conductive with the retention or up gradation of other properties as aforementioned. The findings have been analyzed for morphological, mechanical, thermal and ESD properties.

Table 1. Composition and conductivity of LDPE composite samples.

S.no	Samples	LDPE (wt. %)	FA (wt. %)	EG (wt. %)	Room Temperature Conductivity (S/cm)
1.	LFE1	100	0	0	$>10^{-12}$
2.	LFE2	90	10	0	$>10^{-12}$
3.	LFE3	85	15	0	$>10^{-12}$
4.	LFE4	80	20	0	$>10^{-12}$
5.	LFE5	96	2	2	7.18×10^{-11}
6.	LFE6	93	2	5	9.94×10^{-10}
7.	LFE7	88	2	10	1.02×10^{-9}

Experimental

Material

LDPE granules (Grade: 16MA400) were purchased from Reliance Industries Ltd. Concentrated Sulphuric Acid (H_2SO_4), Nitric Acid (HNO_3) and Ethanol (C_2H_5OH) have been procured from Merck, India and natural graphite was purchased from Loba Chemie, India. Fly ash (FA) was obtained from Badarpur Thermal Power Plant located in NCT of Delhi, India.

Synthesis of expanded graphite

Expanded graphite has been synthesized from natural graphite which was mixed with a solution of concentrated H_2SO_4 and HNO_3 (4:1: vol: vol) [19, 20]. A thick slurry was formed which was continuously stirred for a few hours. The solution was filtered and washed thoroughly till the solution became neutral. The obtained product underwent drying in oven to allow the moisture to evaporate. Ultrasonication using probe was carried out with ethanol as a medium. The sample was again dried to get the desired product. The expansion of the graphite was confirmed by XRD pattern.

Synthesis of LDPE composites

Composites of LDPE/FA, LDPE/FA/EG were synthesized via melt blending process. The composite mixtures were

extruded in wire form using a single screw extruder with a temperature of 110 °C. Different samples of LDPE along with FA and EG have been formulated (by weight %) are listed in **Table 1**.

Wires were converted to granules with the help of the cutter. These composite granules were processed into square shaped sheets by a semi-automatic plunger type injection molding machine with a shot capacity of 60 gm. The temperature of the heaters and mold were set at 130 °C and 35 °C, respectively, with an injection pressure of 600 psi. Schematic representation of the formation of a LDPE/FA/EG sheet has been shown in **Fig. 1**.

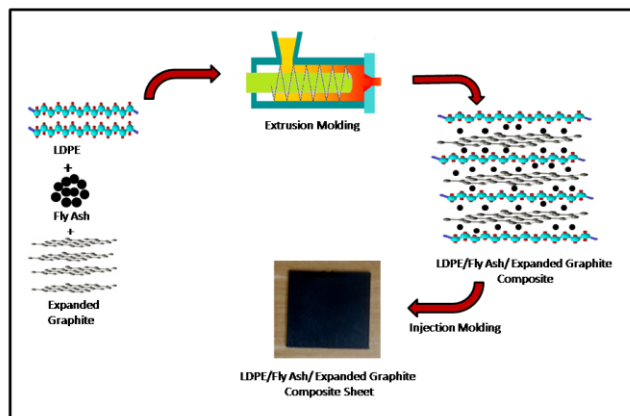


Fig. 1. Schematic representation of the formation of injection molded LDPE/FA/EG (LFE) composite sheet.



Fig. 2. (a) Injection Molding Machine (Thermo scientific Haake MiniJet) (b) Dumbbell shaped samples for tensile testing.

Characteristics

Instron Universal Testing Machine (UTM-5967) was used to carry out the tensile test. The cross-head speed kept at 5 mm/min according to ASTM D638 [21-24] standards. The gauge length was kept at 15mm with a width of 3.1 mm and thickness of 3.2 mm. The test was carried out at room temperature. Dumbbell shaped samples for tensile testing were made on an injection molding machine (Thermo scientific Haake Minijet II) with cylinder temperature and mold temperature at 170 °C and 40 °C, respectively. Injection pressure was maintained at 400 bars (Fig. 2(a, b)). The morphology of fractured LDPE composite sheets after tensile testing was examined on scanning electron microscope (Model LEO 440). Preparation of SEM samples was done by ultra-sonication of various samples with ethanol and placing one or two drops of suspension on silicon wafers. Thermo gravimetric analysis (TGA) had been performed on the Thermo gravimetric analyzer (Mettler Toledo TGA/SDTA 851e) to measure thermal stability of samples under inert N₂ atmosphere with temperature varying from 25-700 °C. Rigaku Powder X-ray Diffractometer (Model: XRG 2KW) was used to carry out X-ray diffraction studies (XRD) using CuK α radiation ($\lambda = 1.540598 \text{ \AA}$) in a scattering values ranging (2θ) 10°- 70°. This study was carried out to confirm the presence of FA and EG in polymer composites. John Chubb Instrument, UK (JCI 155 v5) charge decay test unit was used to measure static charge decay time of LDPE and polymer composite sheets (150 × 150 × 1 mm) at room temperature.

Results and discussion

X-ray diffraction studies

X-ray diffraction studies were carried out for FA, EG and LDPE composite samples, the results being shown in Fig. 3. The main peaks for fly ash (Fig. 3(a)) were observed at $2\theta = 26.6^\circ$ ($d = 3.34393 \text{ \AA}$), 33.26° ($d = 2.6912 \text{ \AA}$), 35.24° ($d = 2.540 \text{ \AA}$), 40.89° ($d = 2.20617 \text{ \AA}$), 42.607° ($d = 2.2063 \text{ \AA}$), 54.12° ($d = 1.6955 \text{ \AA}$), 60.69° ($d = 1.52469 \text{ \AA}$) and 64.58° ($d = 1.44169 \text{ \AA}$) [15]. Fig. 3(b) shows the XRD plot of expanded graphite and peaks were observed at 2θ values 26.34° ($d = 3.38346 \text{ \AA}$), 54.68° ($d = 1.68545 \text{ \AA}$) [19, 20]. Similar peaks of FA and EG have been observed in LFE composite samples which confirms the presence of these fillers into the polymer matrix. While the presence of LDPE has been confirmed by peaks at $2\theta = 21.2^\circ$ ($d = 4.18419 \text{ \AA}$), 23.44° ($d = 3.79124 \text{ \AA}$) and 36° ($d = 2.49392 \text{ \AA}$) [25]. It is also observed that the intensity of FA and EG peak has been suppressed in LFE2 and LFE7 composite, respectively, due to the high percentage of LDPE in composite samples. Peaks of FA in LFE2 composite have also been highlighted in the inset of Fig. 3(c).

Thermogravimetric analysis

Thermal gravimetric analysis of the LDPE composite sheets has been carried out to check the effect of FA and EG, respectively on the thermal stability of the composite sheets. Thermo grams for LDPE/FA composites have been shown in Fig. 4(a), while Fig. 4(b) shows the effect of EG loading on LDPE sheets. It is clearly shown from Fig. 4(a)

that LFE1 shows single step weight loss within temperature range 330° – 450 °C whereas LFE2 shows degradation around 400 °C. After 500 °C, only 18 % residue is left, which does not degrade further with increase in temperature. Further, it is observed that as the percentage of FA increase up to 20 %, an increase in thermal stability is seen with LFE4 being the most stable with a 25 % left over residue. Similarly, Fig. 4(b) shows the results for composites having 2 % FA each and expanded graphite with 2 %, 5 % and 10 % weight loading. It is further observed that the thermal stability again increases with increasing the EG percentage. LFE7 composite sample shows a high order of thermal stability as compared to other LDPE composite samples. It is concluded that the addition of both FA and EG to LDPE polymer matrix bind the system efficaciously with improved thermal stability of composite sheet. Conductivity details for various samples have also been summarized in Table 1.

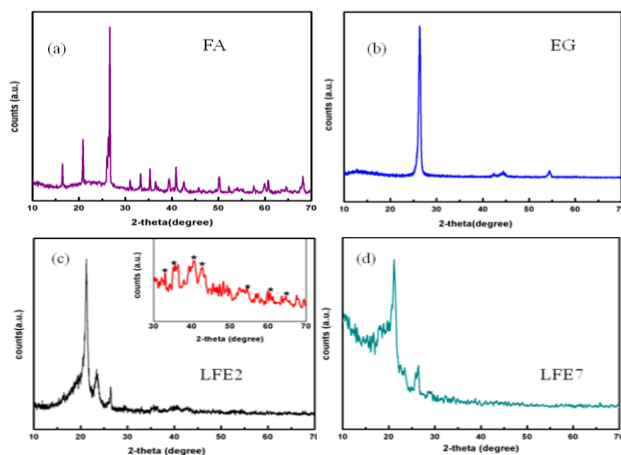


Fig. 3. XRD patterns of a) Fly Ash b) Expanded Graphite c) LFE2 sheet d) LFE7 sheet.

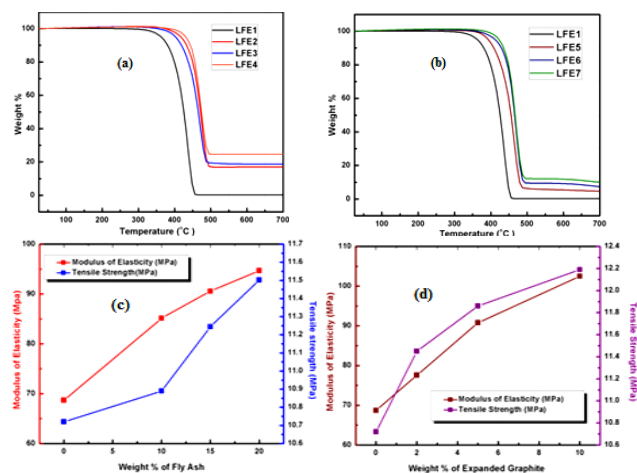


Fig. 4. Thermal gravimetric analysis of a) LDPE/fly ash samples b) LDPE/fly ash/expanded graphite samples, variation of tensile strength and elastic modulus c) LDPE/fly ash samples d) LDPE/fly ash/expanded graphite samples.

Mechanical analysis

Dependency of elastic modulus and tensile strength as a function of weight percentage of filler is shown in Fig. 4(c) and (d). Four specimens were tested for each set of sample

and the average values have been plotted. The variation of tensile strength and elastic modulus for LDPE composite sheets containing a range of FA as reinforcement has been shown in **Fig. 4(c)**. LFE1 i.e. blank LDPE shows an elastic modulus and tensile strength of 68.69 MPa and 10.72 MPa, respectively. Both, tensile strength and elastic modulus increase significantly with the increase of FA content in the LDPE polymer matrix. In the order (modulus; tensile strength), the respective average values for LFE2, LFE3 and LFE4 are (85.14; 10.89) MPa, (90.55; 11.24) MPa and (94.67; 11.50) MPa. Similarly, **Fig. 4(d)** shows the variations of tensile strength and elastic modulus for composite sheets containing a fixed amount of FA and varying percentages of EG to analyze the effect of EG in the presence of FA. Here also an increase in elastic modulus as well as tensile strength is noticed with increasing EG percentages. Tensile strength increases significantly from 11.45 to 12.189 MPa with the increasing percentage of EG in the LDPE matrix, but the increase in values does not show linear curve rather it is positively sloped. A similar style has also been observed for modulus with the increase of the EG content (2 to 10 %) from 77.57 to 102.49 MPa.

Morphological analysis

Morphology of FA particles has been demonstrated in **Fig. 5(a)**. From the micrographs, it has been observed that FA particles are spherical in shape, and particle size ranges from 50 nm to 1 μm as shown in **Fig. 5a**. **Fig. 5b** depicts the morphology of EG and it is observed that particle size of EG varies from of 2 μm to 20 μm . Various sheets of LDPE have been injection molded with/without FA or EG/FA particles to optimize the properties. To understand more about the morphology of LDPE/composite sheets, SEM images of a fractured surface after tensile testing of LDPE, LFE2 and LFE7 sheets has been demonstrated in **Fig. 5(c, d and e)**, respectively. Fractured surface of the blank LDPE sheet indicates very rough and deep flow patterns. When FA particles are added to the LDPE polymeric system, interstitial spaces have been occupied by smaller FA particles that result less flow patterns on the fractured surface of the LFE2 sheet. **Fig. 5(d)** clearly shows the complete encapsulation of FA particles within the LDPE polymer matrix, marked as red circles. No left out holes are visible, which confirms the good interaction between the polymer matrix and FA particles. When EG sheets introduced along with FA particles into the LDPE polymer matrix, further reduction in flow channels has been confirmed as shown in **Fig. 5(e)** and it has been observed that LDPE matrix is sufficient to encapsulate both reinforcement as again no left holes has been shown on the fractured surface of LFE7 sheet. From this morphology analysis, it can be assumed that the presence of the EG sheets into the LDPE polymer matrix along with FA particles not only introduces conducting channels within the sheet but also improves the mechanical properties of the composite sheet due to strong interactions.

Electrostatic dissipative behavior

LDPE and composite sheets were tested on John Chubb instrument to measure static voltage dissipation rate and final value after decay as shown in **Fig. 6**.

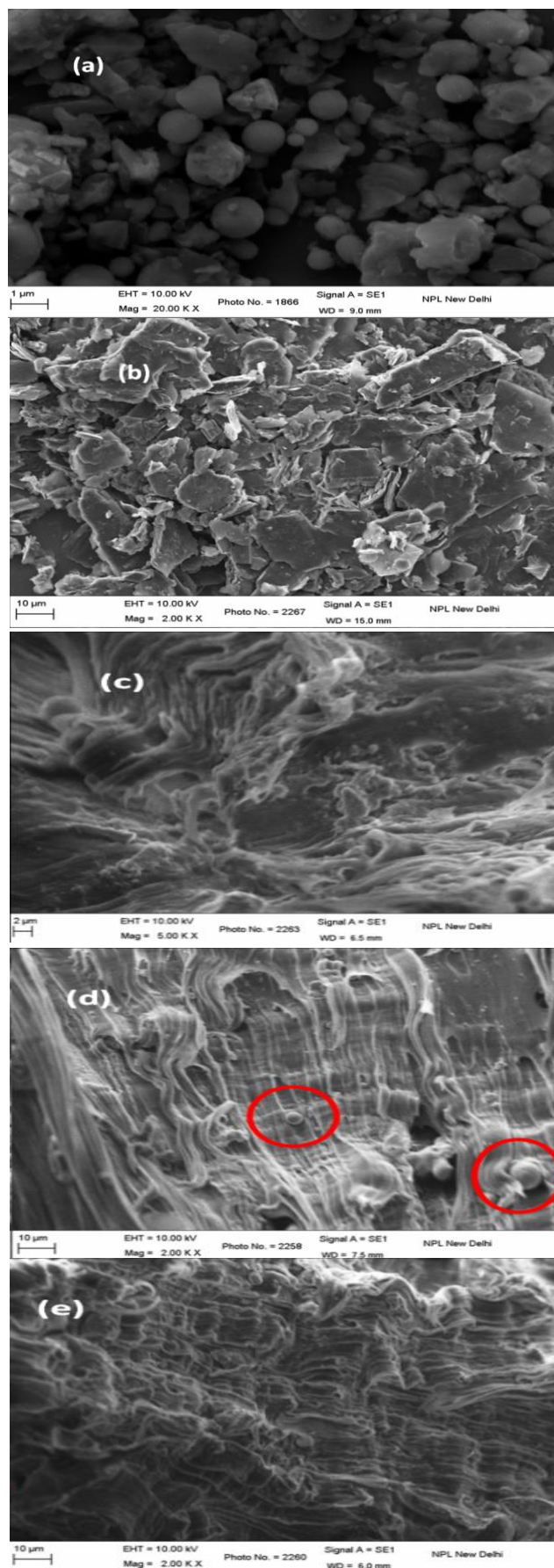


Fig. 5. Scanning electron micrographs of (a) Fly Ash, (b) Expanded Graphite, (c) Fractured surface of LDPE, (d) Fractured surface of LFE2 and (e) Fractured surface of LFE7.

A positive as well as a negative high corona voltage of 5000V was applied to the surface of the sheets. This measurement gives an idea as to how rapidly the voltage falls as the charge is desolated from the surface of the polymer sheet and how much quantity of voltage is decayed. Peaks at -1013.04V and +1015.77V as in **Fig. 6(a, b)**, respectively, show that this amount of voltage was received at the surface which is purely dependent upon properties of the material and did not dissipate at all.

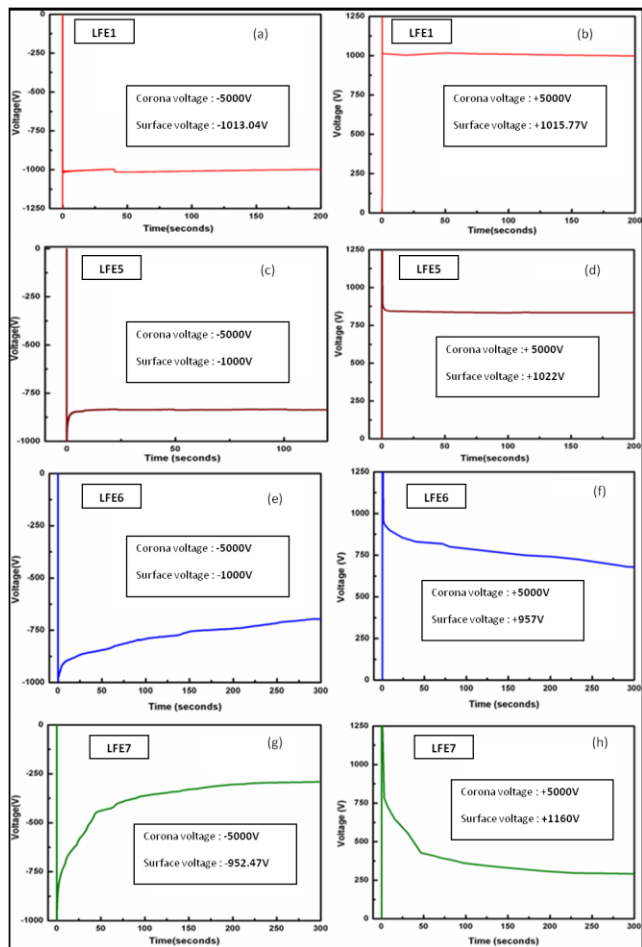


Fig. 6. Electrostatic Charge Dissipative graph of LDPE and LDPE/FA/EG composites showing static voltage dissipation.

This confirms the insulating nature of the LFE1 sheet as the total voltage received was completely retained on the sheet surface. LFE5 sheet showed peaks at -1000V and 1022V (**Fig. 6(c, d)**). The voltage decayed to a value of -844V in 3.7 seconds and 844V in 5.4 seconds in case of negative and positive voltage, respectively. Further, no dissipation was seen. LFE6 sheet showed further improvement than the previous samples and a much steeper curve was obtained as shown in **Fig. 6 (e, f)**. With a peak at -1000V the final voltage value reached a value of -696V. A similar moderate decay rate was seen for positive corona voltage as well. The best result out of all samples was obtained for LFE7 sheet. The voltage decayed down to a value of -290V as shown in **Fig. 6(g)** and +291V as shown in **Fig. 6(h)** which is about 5.8 % of the originally applied 5000V. Better electrostatic charge dissipation (ESD) properties of LFE7 sheet are due to the presence of the

highest percentage of EG filler among all the LDPE/FA composite samples. Based on the above observations, not only a marked increase in dissipation rate was seen for LFE7 composite, the obtained curve being the steepest but also the voltage dropped down to a much lower value with increasing percentage of EG.

Conclusion

Various combinations of LDPE/FA and LDPE/FA/EG composite with different loading levels of FA and EG were formulated via melt blending. The thermal, morphological and electrostatic charge dissipative properties were investigated. Thermal studies indicated that the presence of FA improves the thermal properties of LDPE and when EG introduced into the same system, it further shows positive impact on thermal properties of LDPE sheets. The same trend of result has been obtained for tensile strength and elastic modulus due to the excellent interaction between reinforcement phase and LDPE polymer matrix. The use of EG not only improves the electrical and mechanical properties, but also shows good electrostatic charge dissipative properties.

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