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ORIGINAL PAPER

Simultaneous effect of organoclay and controlled peroxide curing on dielectric properties and flammability of LLDPE/EMA blend

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Abstract The effect of organoclay Cloisite®20A and controlled peroxide curing on the dielectric properties and flammability of LLDPE/EMA blend is explored in this article. The organoclay Cloisite®20A were melt blended with the LLDPE/EMA system at 1, 3, 5, and 7 wt% loading through the variation of the sequence of addition. The dielectric properties of the blend composites are evaluated at different temperature and frequency ranges. Results obtained reveal that the dielectric properties of LLDPE/EMA blend is influenced remarkably by extent of clay loading, variation of sequence of addition of organoclay, and controlled peroxide curing. Both the frequency and temperature dependency of dielectric constant is higher for filled samples than for control blend. On peroxide curing, dielectric constant decreases for all samples and is more significant in case of filled samples. The volume resistivity and breakdown voltage of the samples have also been evaluated. Limiting oxygen index of LLDPE/EMA blends is moderately improved on addition of organoclay.

Keywords Nanocomposites · Electrical properties · Curing · LLDPE/EMA blend

Introduction

Polymeric materials are mostly dielectrics or electrical insulators and resist flow of current. Polymers, being low dielectric constant material, they have been widely

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used in wire coatings, switches, and other electrical and electronic products. The interface properties also strongly affect the electrical properties of polymeric material. The polymeric interfaces act as a charge carrier-trapping sites [1]. Therefore, it is essential to study the effect of interfaces on the charge carrier generation, transport and storage in polymeric systems. In general, the electrical response of a material is characterized by its dielectric constant, loss factor, and conductance [2]. So, it is very important to study the dielectric properties of polymer as function of frequency or temperature for their proper selection as an insulating materials or capacitors.

Electrical properties of polymers and polymer blends have widely been studied. Saxena et al. [3] studied the dielectric properties of polyvinylidenefluoride (PVDF)/polysulfone (PSF) blend and reported that dielectric constant and dielectric loss of the blend increases with increase in PSF content in the blend. Shukla and Gaur [4] studied the electrical conduction of solution grown PMMA, PVDF, and PMMA/PVDF double-layered samples. Mongal et al. [5] studied the mechanical, thermal, and electrical properties of ethylene methyl acrylate copolymer (EMA). Piah et al. [6] studied the electrical tracking and erosion properties of aged natural rubber (NR)/linear low density polyethylene (LLDPE) blend and observed that aging period had significant influence on the electrical tracking of NR/LLDPE blend. Ray and Khastgir [7, 8] developed cable insulating material from low density polyethylene (LDPE)/ethylene vinyl acetate (EVA) and EVA/ethylene propylene diene monomer (EPDM) blends.

In recent years, polymeric materials filled with nanoscale inorganic fillers are gradually receiving more attention for dielectric applications. Polymer nanocomposites are second generation material in insulation engineering. The effective utilization of filled polymers are depends strongly on the ability to disperse the fillers homogeneously throughout the matrix [9]. However, the dielectric responses of polymer–clay nanocomposites have seldom been studied. Wang et al. [10] studied the dielectric properties of polystyrene/clay nanocomposites. They observed that dielectric properties of the composites decrease with increase in extent of exfoliation of clay particles. The dielectric properties of polyimide/clay nanocomposites have been studied by Zhang et al. and Wang et al. They reported that nanocomposites have relatively low dielectric constant than pure polyimide [11, 12]. Tanaka et al. [13, 14] reviewed the manufacturing process, characterization, and applications of polymer nanocomposites as dielectric and electrical insulating material. Yi et al. [15] proposed a simple model to predict the influence of aspect ratio, orientation, distribution, and interface of layered silicate on the dielectric properties of polymer/clay nanocomposites. Hernandez et al. [16] studied the effect of nanoscale dispersion on the dielectric properties of polyvinyl alcohol (PVA)/bentonite nanocomposites.

However, no investigation has been found in the literature that deals in the studies of dielectric properties of organoclay-filled LLDPE/EMA TPE system. The literature is also scanty in the area that involves dielectric studies of peroxide-cured nanocomposites.

The authors have recently studied and reported the effect of effect of organoclay and controlled peroxide curing on structure and properties of LLDPE/EMA-based

thermoplastic elastomer [17]. In this communication, we have studied the effect of organoclay and controlled peroxide curing (low level) on the dielectric and flammability properties of LLDPE/EMA blend. The effect of sequence of addition of clay (while preparing the nanocomposites) on the dielectric and flammability properties has also been evaluated.

Experimental

Materials

LLDPE (LLT12) having a density of 0.926 g/cm^3 and melt flow index (MFI) 3.7 g/10 min were obtained from Haldia Petrochemicals Ltd., India. Commercial grade of EMA, Elvaloy 1330 with 30 wt% of methyl acrylate and a MFI of 3.0 g/10 min of DuPont, Belgium was supplied by NICCO Corporation Ltd., India. Dicumyl peroxide (DCP) (Perkadox-BC-40B-PD) having an active peroxide content of 40% was purchased from Akzo Nobel Chemical Company, The Netherlands.

The compatibilizer used for this study is maleicanhydride-grafted LLDPE (LLDPE-*g*-MA). LLDPE-*g*-MA was prepared by melt blending LLDPE (100 g) with maleic anhydride (5 g) and DCP (40% activity; 0.5 g). The melt mixing was carried out in an internal mixer at 180 °C and 60 rpm for 8 min.

The organoclay Cloisite®20A was purchased from Southern Clay Products Inc. Cloisite®20A is a natural montmorillonite that has been ion exchanged with dimethyl, two hydrogenated tallow moieties and quaternary ammonium chloride to form an organoclay. The weight loss on ignition of Cloisite®20A was 38 wt%.

Preparation of nanocomposites and their vulcanizates

Prior to mixing, LLDPE, EMA, and clay were dried at 80, 50, and 70 °C, respectively, for 12 h in a vacuum oven. The melt blending was carried out in a HAAKE Rheomix OS (Germany) at 140 °C and a rotor speed of 60 rpm by the variation of two different mixing sequences.

In sequence M1, LLDPE and LLDPE-*g*-MA was first allowed to melt for 2 min; it was followed by EMA (4 min) and nanoclay (4 min). The total mixing time was 10 min. In sequence M2, first LLDPE and LLDPE-*g*-MA was allowed to melt for 2 min, and it was followed by nano clay for 4 min. Then, EMA was added to this mixture and mixed for another 4 min. For DCP-treated sample, DCP was added to the prepared nanocomposites and mixed for further 3 min. The mixes so obtained were sheeted out in a two roll mill set at 2 mm nip gap.

The sheeted material was then cured in an electrically heated hydraulic press (Moore Presses, George E. Moore & Sons Birmingham Ltd., UK) at 170 °C for 12 min under a pressure of 5 MPa. The mold was allowed to cool under pressure till ambient temperature is attained before removing the rectangular sheet from the mold. The details of the sample and their appropriate designations are given in Table 1.

Table 1 Sample designations

Sample code	LLDPE (wt%)	EMA (wt%)	LLDPE-*g*-MA (wt%)	Closite®20A (wt%)	DCP (wt%)	Sequence of clay addition
LLDPE	100	–	–	–		–
EMA	–	100	–	–		–
20AE	–	100	–	5		–
E40	60	40	–	–	–	–
3CE40	60	40	3	–	–	–
20AM1/5	60	40	3	5	–	M1
20AM2/5	60	40	3	5	–	M2
20AM2/1	60	40	3	1	–	M2
20AM2/3	60	40	3	3	–	M2
20AM2/7	60	40	3	7	–	M2
3CE40/0.3	60	40	3	–	0.3	–
20AM1/5/0.3	60	40	3	5	0.3	M1
20AM2/5/0.3	60	40	3	5	0.3	M2

Dielectric study

The dielectric and electrical properties of the nanocomposites were measured using a computer-controlled impedance analyzer (Novocontrol Technologies GmbH & Co. KG) on the application of an alternating electric field across the sample cell with a blocking electrode (aluminum foil). The measurements were made in the frequency range of 10^2 to 10^6 Hz at different temperatures ranging from 30 to 120 °C. Samples thickness of 0.6–0.8 mm was used to measure the dielectric properties. Before measuring the dielectric properties, all the samples were vacuum dried for 1 h. The parameters like dielectric constant (ε') and dielectric loss tangent (tan δ) were obtained as a function of frequency and temperature, respectively. Percentage error in the measurements was found to be $\pm 2\%$.

Volume resistivity measurement

The volume resistivity of the nanocomposites was measured at room temperature by Hewlett-Packard high resistance meter (Model 4339B) coupled with Hewlett-Packard (Model 16008B) a resistivity cell. The results reported here are the averages of three experiments. Percentage error in the measurement of volume resistivity was found to be $\pm 1\%$.

Breakdown voltage measurement

The breakdown strength of the samples was measured at room temperature by keeping the film samples in between two copper electrodes. The rate of increase of voltage was 2 kV/s. The dielectric strength was determined by dividing the

breakdown voltage by the thickness (mm) of the films. Three measurements were performed for each sample. Percentage error in the measurement of volume resistivity was found to be ±2%.

Determination of flammability of nanocomposites

The flammability of neat polymers and nanocomposites was evaluated by determining their limiting oxygen index (LOI) according to ASTM D2863-77. The specimens used for the test were in dimension of 100 mm × 10 mm × 2 mm. The determination of LOI of the samples was performed using flammability tester supplied by S. C. Dey Co. Kolkata. Percentage error in the measurement was found to be ±2%.

Results and discussion

Dielectric properties

The frequency dependency of permittivity of pure LLDPE, pure EMA, control LLDPE/EMA blend, and binary nanocomposites of EMA and Cloisite®20A at room temperature are presented in Fig. 1.

The dielectric constant or permittivity of a material is a measure of the extent to which the electric charge distribution in the material can be distorted or "polarized" by the application of an electric field. The material experienced different polarization process when subjected to an oscillating electric field. However, for polar polymers like EMA, the orientation polarization contributes a major part of

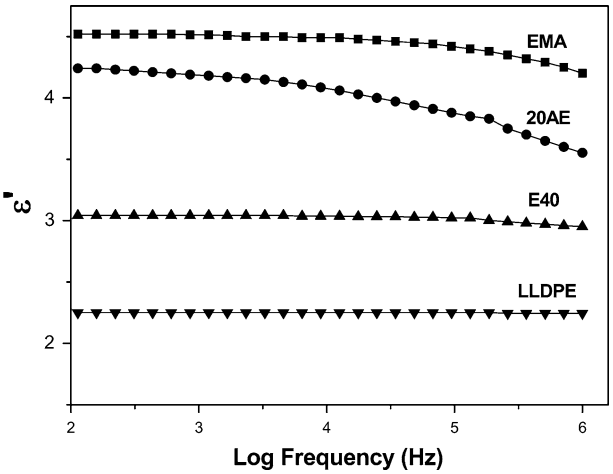

Fig. 1 Permitivity (ε′) as a function of log frequency for pure LLDPE, pure EMA, control LLDPE/EMA (60/40) blend, and binary nanocomposite of EMA and Cloisite®20A

the total polarization. It arises due to the presence of dipoles connected to the chains, chemical construction of the material, or other effects such as oxidation or diffusion of polar molecules from the environment [18]. Thus, EMA has higher ε' than LLDPE (Fig. 1). It can be seen that LLDPE shows almost a constant permittivity in the whole frequency range, which is a typical characteristics of non-polar polymer. However, in the case of control blend, the ε' register intermediate values in between that of LLDPE and EMA over the frequency range studied here. The morphology of control LLDPE/EMA blend consists of continuous LLDPE matrix with dispersed EMA domains, which was demonstrated via SEM analysis in our earlier communication [19, 20]. Here, the nature of variation in ε' with frequency is similar to that of pure LLDPE. It further supports the fact that in LLDPE/EMA 60/40 blend, LLDPE forms the continuous matrix and EMA forms the dispersed phase. From Fig. 1, it can also be seen that the ε' of binary nanocomposites of EMA/Cloisite®20A is lower than that of pure EMA. The frequency dependency of ε' is higher for nanocomposites than that of pristine EMA, probably due to the occurrence of additional dielectric relaxation that arises from anomalous dispersion of clay particles in the EMA matrix. The random distribution of clay particles (exfoliated-intercalated morphology) in EMA matrix has already been proven by WAXD and TEM analysis as reported in recent publications [17]. At sufficiently low frequencies, all three types (electronic, atomic, and orientation) of polarizations contribute to the total polarization. However, the orientation polarization requires much more time when compared with the electronic and atomic polarization to reach the "static field value." When the frequency of the applied field is increased, a situation arises when dipoles present in the system cannot reorient them fast enough to respond to applied field. In EMA/Cloisite®20A nanocomposites, the orientations of dipoles are further restricted by exfoliated and intercalated layered silicates. As a result the orientation polarization is remarkably reduced, which ultimately leads to the reduction of total polarization. This fall in polarizability leads to dielectric relaxation, which in turn, leads to a decrease in dielectric constant. In other words, at higher frequencies due to the rotational displacement of polar groups under the influence of the electric field, frictional loss increases and it reduces dielectric constant. Wang et al. [10] also observed similar kind of nanoscopic-confinement effect for polystyrene/clay nanocomposites.

Figure 2 shows the ε' as a function of test frequency for LLDPE/EMA blend and their nanocomposites at room temperature. It can be seen that ε' decreases with increase in frequency for LLDPE/EMA/Cloisite®20A nanocomposites. The frequency dependency of ε' for LLDPE/EMA/Cloisite®20A can be explained on the basis of nanoscopic-confinement effect of Cloisite®20A, as described earlier. There is also the possibility for interfacial polarization that arises due to the differences in conductivity between the two polymeric phases (LLDPE and EMA) as well as polymers and filler (Maxwell–Wagner effect) [21, 22]. However, the decrease of ε' is more in sequence M2 (20AM2) than in M1. It has been explained in our earlier communication that in mixing sequence M1, Cloisite®20A mostly located in the EMA phase, however, in sequence M2, clay particles located in the LLDPE phase along with the preferential location of the EMA phases [17]. As a result, probability of existence of both EMA–clay and LLDPE–clay interface is more in M2 than M1

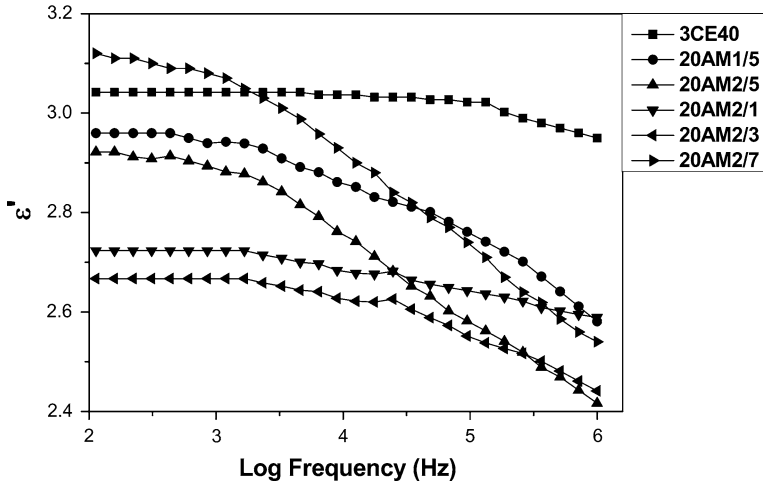

Fig. 2 Permittivity (ε') as a function of log frequency for control LLDPE/EMA (60/40) blend and their nanocomposites

and due to the presence of various interfaces, interfacial polarization plays a major role and frequency dependency of ε' is higher in M2.

In sequence M2, with the extent of organoclay loading, the ε' value follows the order: 20AM2/7 > 20AM2/5 > 20AM2/1 > 20AM2/3. This trend may be due to a balance between interfacial polarization, contribution from polar clay particles as well as restriction of orientation polarization in EMA domains [18].

Figure 3 shows the plot of tan δ versus log frequency for LLDPE/EMA blend and their nanocomposites. It can be seen that tan δ decreases with increase in frequency. Fothergill et al. [23] explained this type of behavior in light of "nanostructuration"

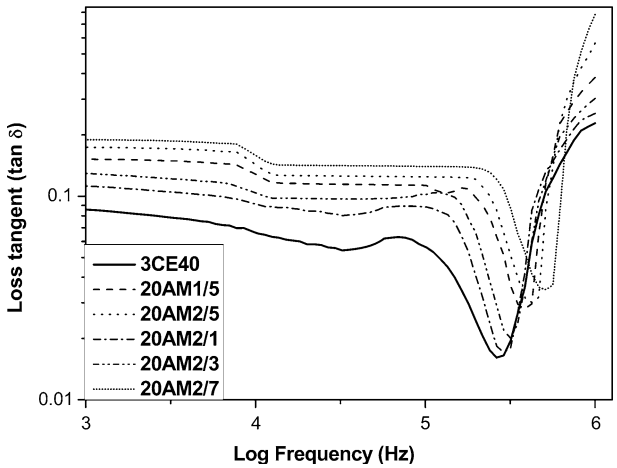

Fig. 3 Loss tangent (tan δ) as a function of log frequency for control LLDPE/EMA (60/40) blend and their nanocomposites

[22]. The broad relaxation appears at around 100 kHz, for control blend, may be associated with the movement of dipoles present in the EMA chain. The broad relaxation of control blend becomes sharper for all filled samples (nanocomposites) due to the presence of different interfaces [18]. The peak minima for tan δ which represent the cessation of the major mechanism of dielectric relaxation (interfacial and orientation polarization) shifted to higher frequencies. The Maxwell–Wagner effect is more prominent in nanocomposites, whereas it is less significant in case of control blend.

Figure 4 shows the variation of dielectric constant with temperature for LLDPE/EMA blend and their nanocomposites at 10^3 Hz. It is seen that for control blend, ε' does not change significantly with temperature. However, for nanocomposites, initially ε' increases with temperature, then it decreases marginally (above 60–70 °C), and finally shows an increasing trend above $\sim$110 °C. The initial increase in dielectric constant with temperature is due to the increase in molecular mobility. The marginal decrease of ε' above $\sim$60 °C may be due to the decrease of effective dipole moments per unit volume caused by thermal expansion as opposed to the increasing segmental mobility. Above 110 °C, the polymer starts softening and hence ε' shows an increasing trend due to the free motion of the dipoles. In addition to free dipole motion, interfacial polarization also contributes significantly at very high temperatures like 110 °C.

Effect of controlled peroxide curing

Figure 5 shows the plot of dielectric constant versus log frequency for LLDPE/EMA/clay nanocomposites cured with 0.3 wt% DCP. It can be seen that the permittivity decreases for all samples when cured with DCP. The decrease of permittivity is more significant for filled samples probably due to the better

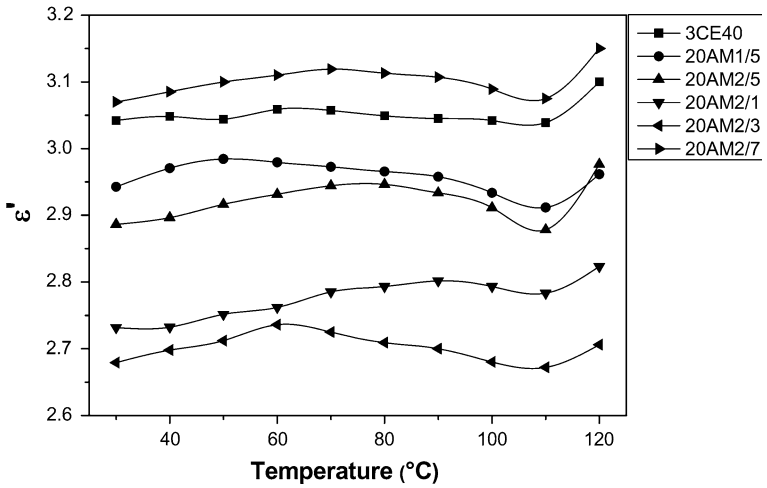

Fig. 4 Variations of permittivity (ε') as a function of temperature for control LLDPE/EMA (60/40) blend and their nanocomposites at 1000 Hz

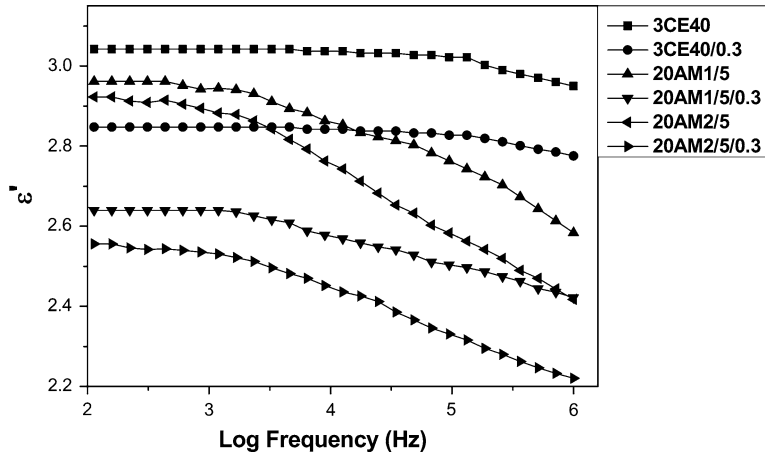

Fig. 5 Permittivity (ε') as a function of log frequency for peroxide-cured LLDPE/EMA/Cloisite®20A nanocomposites

dispersion (exfoliation) of clay particles as well as polymer cross-linking in DCP-cured samples. The better dispersion (exfoliation) of clay particles in DCP-treated samples has been observed from TEM micrographs [Fig. 6]. After peroxide curing, the average decrease of ε' is 6.5% for control blend, whereas the decrease is 10 and 12% for 20AM1 and 20AM2, respectively. When comparing between mixing sequences M1 and M2, the decrease of ε' is more significant in sequence M2 (20AM2/5/0.3). One important observation, the dielectric constant is more or less frequency independent in DCP-cured samples. This is due to the diminished of interfacial polarization due to cross-linking. In other words, controlled DCP curing improves the interfaces of LLDPE/EMA blend nanocomposites.

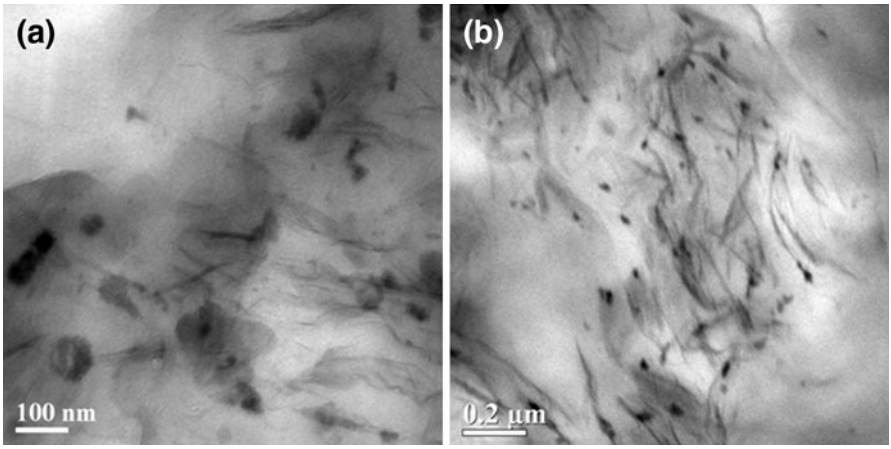

Fig. 6 TEM micrographs of **a** uncured nanocomposites (20AM2/5), **b** DCP-cured nanocomposites (20AM2/5/0.3)

Figure 7 shows the variation of dielectric constant with temperature at 10^3 Hz for LLDPE/EMA/Cloisite®20A nanocomposites cured with 0.3 wt% DCP. It can be seen that ε' decreases at higher temperature. In general, at higher temperature the dielectric properties are mostly controlled by interfacial polarization and ionic conduction. When cured with 0.3 wt% DCP, the EMA phase and interfaces of LLDPE/EMA blend nanocomposites are strengthened due to cross-linking. As a result, interfacial polarization diminishes in cured samples. Moreover, due to cross-linking, chain mobility at the melting and softening temperatures is restricted leading to decrease in dielectric permittivity with temperature.

Volume resistivity and electrical break down strength

The study of electrical resistivity is very important for insulating materials, because the most desirable characteristic of an insulator is its ability to resist the leakage of electrical current. Table 2 shows the volume resistivity of pure LLDPE, pure EMA, control LLDPE/EMA blend (60/40 w/w), and their clay nanocomposites. LLDPE, with a volume resistivity in the order of 10^{16} Ω cm is an excellent electrical insulator, while the volume resistivity of EMA is in the order of 10^{13} Ω cm. The volume resistivity of control blend lies between that of LLDPE and EMA (in the order of 10^{13} Ω cm range). From Table 1, it can be seen that the volume resistivity of nanocomposites are higher than that control blend. However, the volume resistivity of nanocomposites prepared by sequence M1 (20AM1/5) is slightly higher than that of prepared by sequence M2 (20AM2/5). This is as per the dielectric results mentioned earlier. From Table 2, it can also be seen that the volume resistivity of DCP-cured samples are higher than that of uncured ones. This is due to the presence of cross-linking point in cured samples which act as barriers to prevent the charge movement between polymer chains and thus increase the

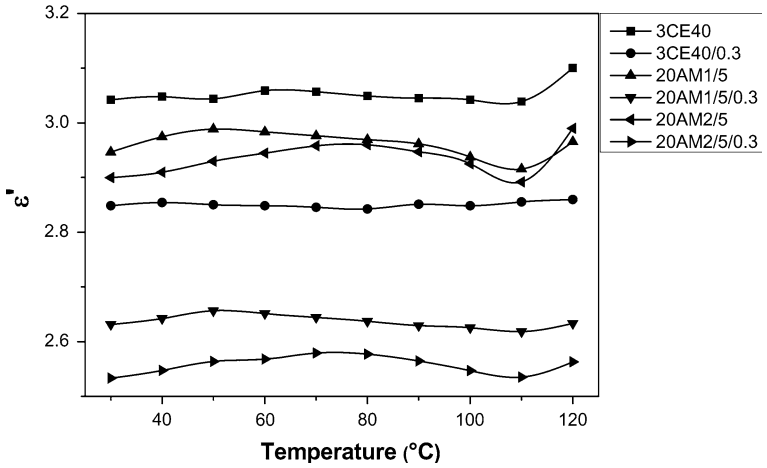

Fig. 7 Variations of permittivity (ε') as a function of temperature for peroxide-cured LLDPE/EMA/Cloisite®20A nanocomposites at 1,000 Hz

Table 2 Volume resistivity and voltage breakdown strength of LLDPE/EMA blend and their nanocomposites

Sample code	Volume resistivity (Ω cm)	Voltage breakdown (kV/mm)
EMA	2.4×10^{13}	–
LLDPE	1.2×10^{16}	–
3CE40	1.8×10^{14}	32.6
20AM1/5	5.5×10^{14}	38.1
20AM2/5	4.9×10^{14}	37.2
20AM2/1	6.4×10^{14}	38.9
20AM2/3	6.6×10^{14}	39.7
20AM2/7	1.1×10^{14}	30.5
20AM1/5/0.3	8.6×10^{14}	42.1
20AM2/5/0.3	8.8×10^{14}	42.8

electrical resistance of cured samples [24]. Overall, the resistivity results reflect that all the LLDPE/EMA/Cloisite®20A nanocomposites are good electrical insulator.

The dielectric strength or breakdown strength of an insulating material is the maximum electric field strength; it can withstand without experiencing failure of its insulating properties. The breakdown strength of LLDPE/EMA blend and their clay nanocomposites are presented in Table 2. It can be seen that all the nanocomposites undergo dielectric breakdown at higher voltages than that of control blend. There exist large interfacial area between clay particles and polymer matrix. This interfacial area plays an important role on dielectric breakdown of insulating material. However, the cured samples exhibit higher breakdown strength than those of uncured samples possibly due to the cross-linking of polymer material. The cross-linked domains act as barriers to prevent the electrical breakdown path inside the polymer matrix, as explained earlier.

Measurement of LOI as a measure for the flammability of the nanocomposites

To measure the effect of addition of organoclay on the flame retardancy of LLLDPE/EMA blend, their LOI was evaluated. The LOI of a material is determining by measuring the minimum volume ratio of oxygen in oxygen–nitrogen mixture that just support flaming combustion, i.e.,

$$\%\mathrm{LOI} = \frac{\mathrm{O_2}}{(\mathrm{O_2} + \mathrm{N_2})}\%$$

The greater the LOI of a given material, the lower is its flammability. The LOI values of the control blend and its nanocomposites, prepared by different mixing sequence, are presented in Table 3. It is seen that addition of organoclay moderately increases the LOI of control LLDPE/EMA blend, i.e., the presence of the organoclay reduced the flammability of the control blend. This is due to the accumulation of silicate layers on the surface of the burning specimen which creates a protective barrier to heat and mass transfer [25, 26]. The LOI of LLDPE/EMA/clay nanocomposites increases with increase in clay loading up to 5 wt% beyond that level (7 wt%) it levels off. This may be ascribed to the aggregation of the clay

Table 3 LOI of LLDPE/EMA blend and their nanocomposites

Sample code	%LOI
3CE40	22.2
20AM1/5	26.3
20AM2/5	26.7
20AM2/1	23.7
20AM2/3	25.0
20AM2/7	23.4
20AM1/5/0.3	27.7
20AM2/5/0.3	27.8

particles at higher loading level, which provided smaller surface area. However, DCP-cured LLDPE/EMA/clay nanocomposites have LOI higher than uncured one probably due to the better dispersion and higher extent of exfoliation of the clay nanocomposites in DCP-cured samples; which has been demonstrated via WAXD and TEM analysis in our earlier communication.

Conclusion

The dielectric properties and flammability of LLDPE/EM/Cloisite20A nanocomposites were investigated by dielectric analyses and LOI measurement from which following conclusion can be drawn:

(1) The dielectric properties of LLDPE/EMA/Cloisite20A nanocomposites are influenced remarkably by extent of clay loading, variation of sequence of addition of clay as well as peroxide curing.
(2) The addition of organolay results in the lowering of dielectric constant of control LLDPE/EMA blend. The ε' of the nanocomposites decreases with increase in applied frequency over the frequency range studied here. The frequency dependency of ε' is more in sequence M2 (20AM2/5) when compared with that of sequence M1 (20AM1/5), probably due to the increased contribution of interfacial polarizations in the earlier one
(3) Upon addition of DCP, the ε' of the nanocomposites further decreases. This is due to the better dispersion of clay particles as well as formation of cross-linked network structure with improved interfacial bonding in DCP-cured samples.
(4) The volume resistivity and breakdown strength of the LLDPE/EMA blend increases on addition of organoclay and peroxide curing.
(5) Addition of organoclay and DCP moderately improved the LOI of LLDPE/EMA blend.

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