LDPE/EPDM Blends as Electrical Insulators with Unique Surface, Electrical and Mechanical Properties

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A B S T R A C T

The mechanical, thermal, morphological, dynamical mechanical and electrical properties of polymeric insulators are tested and reported. Low density polyethylene (LDPE), ethylene-propylene-diene monomer (EPDM) and blends of LDPE/EPDM are known polymers for use as low, medium and high voltage insulators. Several formulations containing EPDM and LDPE were prepared. EPDM was blended with LDPE in different weight ratios of LDPE/EPDM (100/0, 80/20, 60/40, 50/50, 40/60, 20/80, 0/100). The change in Haake-viscosity mixing torque with time for different blend compositions was obtained. The torque at any time is dependent on the blend composition. For EPDM the observed torque is directly influenced by its cross-linking density. However, for LDPE or blends of high percentage of LDPE the influence of cross-linking density on torque is relatively low. The mechanical properties of cross-linked EPDM are weaker than LDPE. Therefore changing the cross-link density of EPDM will not affect the blend properties significantly especially at high LDPE content. A drop shape analysis system G l0 (Kruss-USA) was used for hydrophobicity studies. Studies illustrate that in LDPE/EPDM blends, tensile strength of the blends are higher than either polymer component alone. But the synergism effect in LDPE/EPDM compound is stronger than HDPE/EPDM. Also the blends of LDPE/EPDM show good breakdown voltage strength and heat resistance comparing to LDPE. The result of mechanical measurement shows that mechanical properties such as tensile strength, modulus and elongation-at-break of blends can be enhanced with increase in LDPE in the formulation. Also tests were conducted to study various properties (mechanical, surface, electrical) under accelerated ageing condition. Because the life time of these compounds are generally too long to wait for a verdict on their behaviour in use. The ageing results show that mechanical properties follow the same trend as pre-ageing stage.

INTRODUCTION

Today, EPDM insulators are suitable materials as distribution and transmission class insulations. Their long term performance in clean environment has been successful and because of their combination of superior electrical properties, flexibility over a wide temperature range and their resistance to humidity and weather, EPDM is used in a broad range of electrical insulators (weather shed, power cable, appliance wire, moulded electrical accessories, etc.).

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However, EPDM insulators suffer from problems emerging from in service reduction of hydrophobicity and as a result poor resistance against tracking electrical discharge and sunlight may develop [1]. The principal value of polyethylene lies in its desirable balance of physical properties in the solid state and inertness. These qualities in combination with its low cost and easy processability make it the material of choice for a wide variety of applications. Also, polyethylene finds extensive use as an insulator, primarily in the wire and cable industry. Despite its intrinsically desirable electrical properties polyethylene is not totally immune to the effects of electrical fields and currents.

Nowadays, polymer blend is one of the major subjects in polymer industry. The purpose of alloying or blending one polymer system with another polymer is to gain synergistic improvement in properties at both the micro and macro levels [2-6]. Polymeric insulating materials such as EPDM (ethylene-propylene-diene monomer) and polyethylene are widely used in manufacturing of weather shed of outdoor insulators and cable terminators [7]. Batiuk, Herman and Healy reported that blends of EPDM especially with LDPE exhibit superior tensile strength, higher than predicted from their additive's individual effect [8-10]. A considerable amount of work has been presented on the blends of polyolefin and EPDM due to their commercial importance [10-13]. Among these efforts, some focused on the rheology [13-15], the degree of branching, density and molecular weight [16-17].

Bielinski et al. describe morphology, structure, and properties of polyolefin blends made of EPDM and low-density polyethylene (LDPE) [18]. The influence of the physical characteristics of both the EPDM matrix and LDPE dispersed phase was studied. Special attention was devoted to the surface layer of the blends and related physical properties of the system. They concluded that the extent of segregation depended on the molecular structure of the EPDM matrix, which determined the miscibility of the components on a segmental level. The higher the ethylene monomer content present in EPDM the lower was the PE content in the surface layer of the blends. The composition and structure of the surface layer was responsible for its lower hardness in comparison with the bulk of the blends studied. The surface gradient of the mechanical properties depended on the physico-chemical characteristics of the components and the blend composition, which created the possibility of tailoring the LDPE/EPDM blends according to the final applications. The viability of maleic anhydride grafted polyethylene modified jute fibre reinforcement in LDPE/EPDM and HDPE/EPDM blends was studied by Sarkhel et al. [19]. It was shown that the flexural strength, flexural modulus, impact strength, and hardness increased with increasing both the fibre loading and the compatibilizer dose. Another study shows that the structure exhibited by stress-strain curve of the polyethylene's blend is reduced with the addition of the elastomeric phase, and the ultimate properties improve because the amorphous segment becomes softer and reduces its capability to transmit the applied stress to crystalline particles [20].

There are many reports on the blends of ethylene-propylene terpolymer and isotactic polypropylene. However, the use of polypropylene requires special equipment ensuring high temperature (up to 220-240ºC), whereas rubber processing generally does not require such temperature range. Although thermoplastics based on polyethylene, in particular low-density polyethylene (LDPE), are of most interest, there are few papers published on the subject. In terms of total consumption of PE in Iran, 2,945,000 tons per year in 2002 rose to 3,745,000 tons per year by 2006 [21]. Using polymers such as EPDM and LDPE instead of porcelain and glass for outdoor insulation is expanding, and it is still under going development. There are variations in the material compositions of EPDM and LDPE that are presently available. These variations result in significant differences in the electrical and mechanical performance of the materials under different conditions. In this paper the use of LDPE/EPDM blends in different ratios for outdoor insulation was examined and compared to EPDM and LDPE alone.

EXPERIMENTAL

Materials
All materials used for this work were commercial products and they were used as received without further treatment. Table 1 shows the raw materials used in this investigation.

Table 1. Identification of materials.

Material	Grade	Supplier
EPDM	Vistalon 7500	Exxon Chemical
LDPE Industry	Poliran LF 0200	Iran Petrochemical
DCP	98% Purity	Hercules Inc. USA

Preparation of Compound

Several formulations containing EPDM and LDPE were prepared (Table 2). EPDM was blended with LDPE in different weight ratios of LDPE/EPDM (100/0, 80/20, 60/40, 50/50, 40/60, 20/80, 0/100). These compounds were mixed at 140ºC in Haake internal mixer for 10 min at a rotor speed of 60 rpm and then mixed with DCP on a two roll mill at room temperature. Vulcanization was performed with a hydraulically operated press at 170ºC and 15 bar.

Curing Characteristics

The cure characteristics of the compound were determined according to ASTM D2084 by using Zwick rheometer model ODR at 170ºC. The respective cure time (t_{95}), scorch time (t_5) and torque values were obtained from rheograph.

Mechanical Properties

Mechanical properties of the vulcanized rubbers, such as tensile strength, hardness, before and after ageing, were measured according to ASTM methods. Tensile strength, modulus and elongation-at-break of a specimen were measured in a universal testing machine (MTS). A dumb-bell specimen is required (die c), according to ASTM D 412-87. Hardness (shore A) testing was done according to ASTM D2240 using a Zwick load tester.

Accelerated ageing

Salt solution spray ageing was performed according to ASTM B117 at 95-98% (5% NaCl solution in 1000 h at 40ºC)

Morphology Studies

Morphology of the various compounds after tensile tests was studied by scanning electron microscope (SEM) technique using a Cambridge fractured under liquid nitrogen and the fractured surface of samples sputtered with gold in vacuum.

Electrical Properties

Dielectric Properties

Dielectric strength of the compression moulded samples was measured according to ASTM standard D 149-87 at 25ºC using a dielectric strength measuring instrument model CEAST 6136/000. The test voltage was applied on opposite faces of the samples of approximately 2 mm thickness. The cell was placed in an insulator oil (silicone) bath.

Volume Resistivity

A standard size specimen, according to ASTM D 257-99, was placed between two electrodes. A voltage resistance was measured in a high resistance meter. Surface or volume resistivity was then calculated. The volume resistivity of compression moulded sample was measured according to ASTM standard D 257-83 using Davenport Daventest SVR438134 model.

DMTA

Dynamic mechanical analysis was carried out by using the polymer lab instrument. Tests were performed from -60°C to 200°C at a frequency of 1 Hz and at a heating rate of 10°C/min (ASTM D945).

Wettability (Contact Angle)

Hydrophobicity of a surface is intimately related to the so-called contact angle. The contact angle is the angle at which a liquid/vapour interface meets the

Table 2. General description of materials evaluated.

Sample	1	2	3	4	5	6	7
EPDM	100	80	60	50	40	20	0
LDPE	0	20	40	50	60	80	100
DCP	3	3	3	3	3	3	3

solid surface. The most commonly used method for contact angle measurement of surface hydrophobicity is the sessile drop technique. A droplet of a purified liquid (distilled water) is placed on surface using a syringe. The resulting angle between the droplets is measured, generally using a geniometer or a charge coupled camera device (CCD) fitted onto a microscope. A drop shape analysis system G l0 (Kruss-USA) was used for hydrophobicity studies. The samples (1 to 7) have also been immersed in water (with 5% NaCl) for 500 h and 1000 h.

Figure 1. Haake torque of different LDPE/EPDM blends compositions plotted against time.

RESULTS AND DISCUSSION

Mixing Characteristics

The change in Haake-viscosity mixing torque with time for different blend compositions is presented in Figure 1. The torque at any time is dependent on the blend composition. This composition of rubber and plastic components determines the structure and its potential end-use properties. The rubber-rich blends can be used as thermoplastic elastomers and the plastic-rich blends can be used as rubber toughened plastics. Processing and final properties are dependent on the composition. The area under the torque-time curve represents the mechanical energy consumed during blending. Figure 1 shows that the rubber-rich blends require more energy than plastic-rich blends. A lower value for torque implies better processability [22].

Table 3 shows the effect of formulation on the scorch time and the optimum cure time of compounds, respectively. As expected, it can be seen that with increasing LDPE content scorch time and optimum cure time increase, but the max-min torque decreases. This is attributed to the polymer matrix which changes from rubber to plastic and the reduction of unsaturated bonds due to EPDM present.
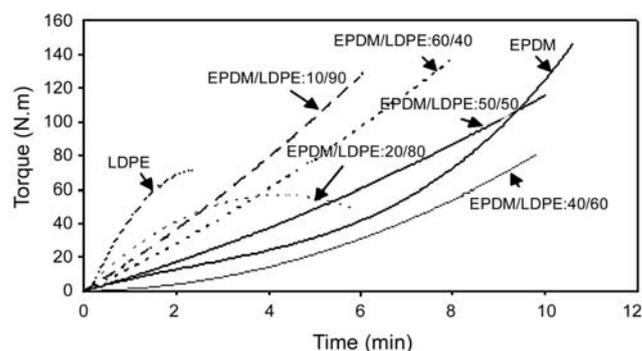
For EPDM the observed torque is influenced directly by the cross-linking density. But, for LDPE or blends of high percentage of LDPE the influence of cross-linking density on torque is relatively low. Apparently with increasing EPDM in the blend, torque difference will be a function of DCP concentration. If we increase the amount of DCP the torque for LDPE will not change. Due to unsaturated bonds the extent of cross-linking in EPDM is much higher than LDPE in the presence of DCP. The mechanical properties of cross-linked EPDM are weaker than LDPE. Therefore, changing the cross-link density of EPDM will not affect the blend properties significantly especially at high LDPE content.

At low peroxide concentration, the cross-linking mechanism in the pure low density polyethylene (LDPE) matrix is based, as expected, on the hydrogen abstraction from the chains and the coupling of the resulting macroalkyl radicals. The latter reaction takes place in competition with the disproportionation causing the formation of chain unsaturations that accumulate with the reaction time. At relatively large (>2%) peroxide concentrations, such unsaturations reach sufficiently high levels to scavenge all the initiating and macroalkyl radicals giving macroallyl

Table 3. Influence of LDPE content on cure characterisctics of samples (Δ_{Torque} = (max-min) torque).

Sample	1	2	3	4	5	6	7
t_5 (min)	0.82	0.86	0.93	0.91	0.90	1.01	125.0
t_{95} (min)	9.05	11.94	12.12	11.32	11.96	13.19	14.19
Δ_{Torque}	102.80	78.20	79.00	66.70	53.30	39.50	25.0

Table 4. Comparison of the value of hardness for virgin and salt fog aged samples.

Sample	1	2	3	4	5	6	7
H_1	55.3	69.2	81.1	85.5	88.5	91.4	94.2
H_2	53.9	69.2	80.8	85.4	88.5	91.4	89.4

H_1: hardness before ageing, H_2: hardness after salt fog ageing (1500 h).

Table 5. Comparison of the value of modulus 100% and 200% for virgin and salt fog aged samples.

Sample	1	2	3	4	5	6	7
SB_1	0.526	2.207	3.046	4.043	5.196	7.024	9.157
SA_1	1.176	2.684	3.902	4.727	5.286	7.209	10.264
SB_2	-	-	4.427	5.465	6.579	7.776	9.047
SA_2	-	-	5.164	6.488	6.658	7.979	10.18

(SB_1: stress-at-strain point 01 MPa for virgin samples, SA_1: stress-at-strain point 01 MPa after salt spray fog ageing, SB_2: stress-at-strain point 02 MPa before salt spray fog ageing, SA_2: stress-at-strain point 02 MPa after salt spray fog ageing).

radicals as the dominant intermediates and determining changes in the structure of the cross-links during the course of the reaction.

In general, the production of cross-linked polyethylene (by organic peroxides) in forms of cables is carried out in continuous operation and cross-linking reaction is completed during the different steps of processing. Cross-linking reaction takes place in melt condition and network develops along the production line. The rheological properties change with developing network and it is often vital to know the trends of these variations and the time for reaching the gel point.

Mechanical Testing

The mechanical properties such as tensile strength, elongation-at-break, modulus and hardness are presented in Tables 4 and 5 and Figures 2 and 3. It can be observed that mechanical properties such as tensile strength, modulus and elongation-at-break increase with increasing LDPE content in the samples, implying that the phase of matrix governs the properties.

These results were confirmed by SEM micrographs which will be described later. The increase in tensile properties and the reduction of elongation-at-break after ageing should be expected as a result of the plastic nature of LDPE. The elongation-at-break

of the blend at higher LDPE content was higher than those of the linear blends due to the chemical cross-linking of EPDM. Although EPDM content increases, the chemical cross-linking of EPDM restricts the mobility of polymer chain. It was also observed that mechanical properties after salt fog ageing, improved for most of the samples, which can be attributed to interaction and compatibility between the phases and in addition the reaction of residual DCP in compound during ageing [23].

The hardness of salt fog aged and virgin samples was measured. It can be observed from Table 4 that the hardness of samples decreased in effect of thermal ageing and it can be the result of thermal deterioration of samples.
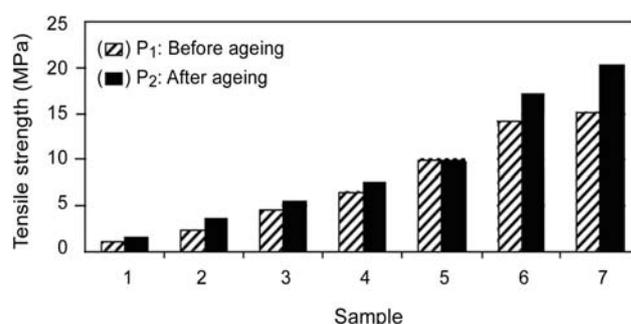
Figure 2. Comparison of the value of tensile strength for virgin and salt fog aged samples.
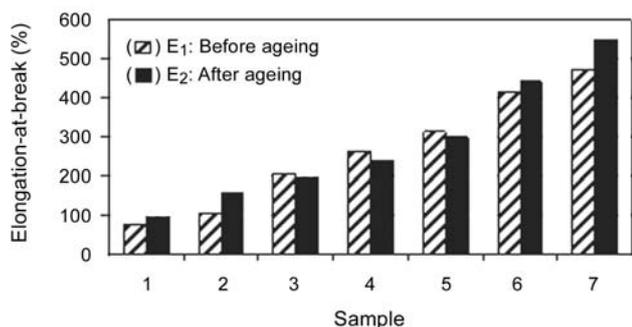
Figure 3. Comparison of the value of elongation-at-break for virgin and salt fog aged samples.

Dielectric Constant and Dielectric Strength

The dielectric constant and dielectric strength (breakdown voltage) of different EPDM/LDPE blends at ambient temperature are presented in Figures 4 and 5. The dielectric constant increases with increase in LDPE concentration in the blend. This is mainly due to propylene unit that exists in EPDM. The trend of dielectric loss factor with composition follows the same pattern as that of dielectric constant. The loss factor increases with an increase in LDPE concentration. Dielectric constant and loss factor, both of them show a slight reduction for sample 3 (EPDM = 60, LDPE = 40) as compared with sample 2 (EPDM = 80, LDPE = 20). This may be due to the increased contribution of Maxwell-Wagner type interfacial polarization in the blend [24]. The breakdown voltage decreases slightly with LDPE concentration in samples (Figure 5). This change can be correlated with the thickness of samples. Because the dielectric strength in volt per mm varies somewhat depending on the thickness of the sample tested. Ordinarily, thin test
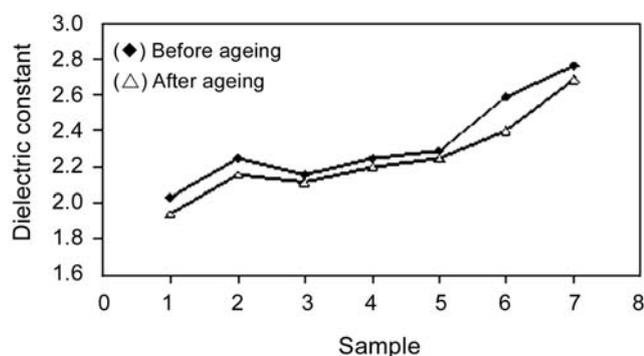

Figure 4. Comparison of the value of dielectric constant for virgin and salt fog aged samples.
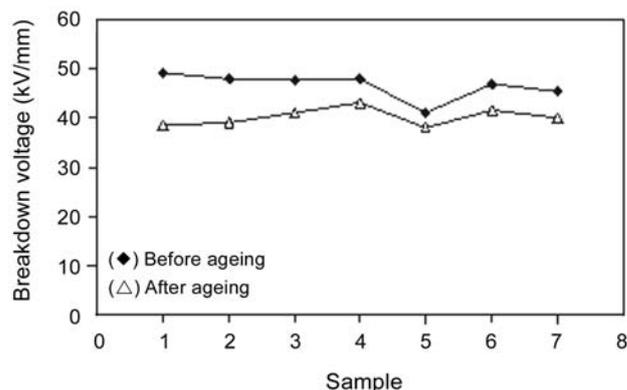

Figure 5. Comparison of the value of breakdown voltage for virgin and salt fog aged samples.

specimens show a higher dielectric strength than thick specimens. The reduction of breakdown strength in samples can be the result of chain scission during thermal ageing.
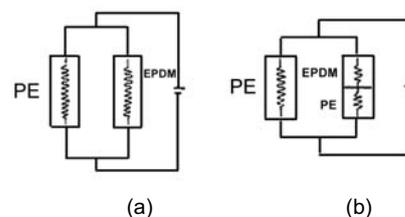
Volume Resistivity

Volume resistivity of the compounds at 28ºC as a function of blend composition is shown in Table 6. It should be noted that values obtained by this test are not absolute, because the dielectric strength in volts per mm varies somewhat depending on the thickness of the sample tested. Initial volume resistivity of the compounds and its change after ageing in salt fog chamber, are plotted in Table 6. There is a regular change in volume resistivity with increase in LDPE and relationship deviates from linearity. This blend system consists of two polymers, LDPE and EPDM having high resistivity in order of $10^{17}\Omega$cm. For describing the amount of theoretical conductivity of any blend composition, the two constituents can be considered as two separate resistances [25]. So it is found that the theoretically calculated resistivity is in close agreement with the experimentally observed data (Table 6).

$$\frac{1}{R_{blend}} = \frac{1}{R_{PE}} + \frac{1}{R_x}$$

where

$$R_x = R_{PE} + R_{EPDM}$$

(a)　　　　(b)

It seems that volume resistivity increases with

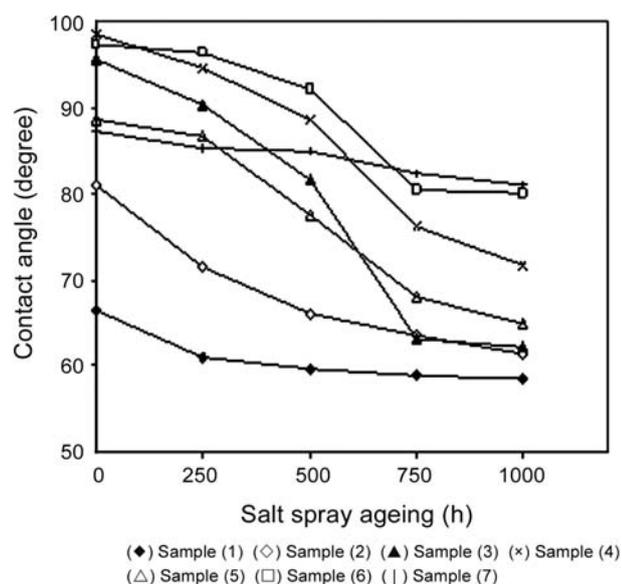**Table 6.** Comparison of the value of log (volume resistivity, ohm.cm) for virgin and salt fog aged samples.

Sample	1	2	3	4	5	6	7
Before ageing	17.21	17.20	17.19	17.07	17.00	16.95	16.95
After ageing	17.25	16.89	16.97	16.93	17.19	17.07	17.13

increase of LDPE content in the samples. It can be observed from Table 6 that the value of volume resistance of compounds increases after humidity ageing. This increment can be attributed to the removal of charge carried from vulcanized by the distilled water or with the rejection of the charge entered to the liquid medium. But some data of volume resistivity measured after immersion of the compound in water decreases with the time of immersion. This is possibly due to the diffusion of water into the system and the decreasing trend is not similar for all blends.
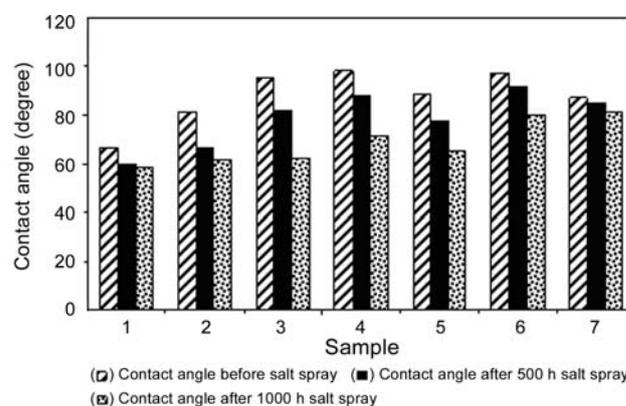
Wettability (Hydrophobicity)

The results in Figure 6 show the corresponding decrease in the hydrophobicity. All the samples have been picked up from saline water and measured after the surface had been dried at room temperature. In the present work, the reported measurement of the contact angle is averaged over 5 locations.

The thickness of samples was 2 mm for water salinity test. It can be seen from Figure 6, that

hydrophobicity of samples decrease after saline water ageing. It can be suggested that the presence of salt has a deleterious effect on the hydrophobicity of the surface as the result of oxidation reactions. Figure 7 shows a histogram of contact angle for different samples before and after ageing. The surface property not only depends on the contaminants, but also on the material properties which control the layer formation. The upper parts of samples are usually more severely affected by roughness. This roughness is due to some heterogeneity of the surface that can be attributed to scission or/and cross-linking of the surface polymer chains, oxidation or complete decomposition of the surface polymer. For the EPDM insulator, it was found that contact angle after 500 h salt spray fog ageing decreases dramatically and after that it remains constant. But for LDPE it was observed that the contact angles are much higher than those obtained with EPDM insulators and especially in the case of aged surfaces. For all samples, results show that salt polluted blends do not show any recovery of their contact angle, and wetting angles were reduced with the same trend which is shown in Figure 6. However, the differences in reduction patterns in contact angle are not similar in all blends, which are due to the following reasons. For example, wetting angle measurement shows that when a drop is deposited on

(◆) Sample (1) (◇) Sample (2) (▲) Sample (3) (×) Sample (4)
(△) Sample (5) (□) Sample (6) (|) Sample (7)

Figure 6. Comparison of the value of contact angle for samples.

(▨) Contact angle before salt spray (■) Contact angle after 500 h salt spray
(▨) Contact angle after 1000 h salt spray

Figure 7. Effect of ageing on contact angle.

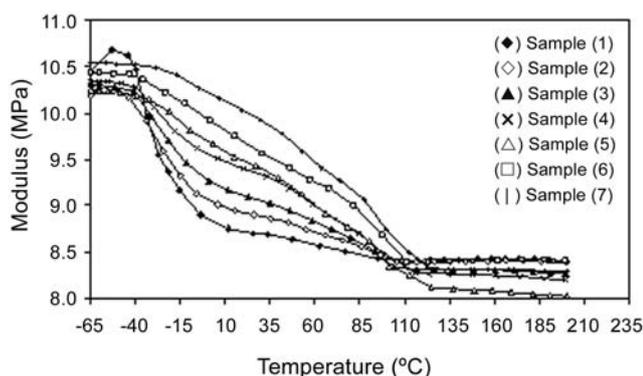**Figure 8.** The storage modulus of the samples.

Figure 9. Tan (δ) of samples versus temperature.

the aged EPDM surface, the insulator has, for a short time, some hydrophobicity. However, cracks spread over the surface seem to act as capillaries, spreading the water over the surface and making the insulator completely hydrophilic.

Dynamic Mechanical Thermal Analysis (DMTA)
The dynamic mechanical curves (E' and tan δ as a function of temperature) for samples are shown in Figures 8 and 9, respectively. The value of storage modulus for samples decreases at the glass transition (Figure 8). The decrease of storage modulus at the transition zone is significant. The decrease in the value of storage modulus for EPDM occurs only at the transition zone. It can be seen from Figure 8 that the value of storage modulus of EPDM (sample 1) improved with LDPE content and reaches its maximum with sample 7. Glass transition temperature and the loss tangent (tan δ) values of samples at 1 Hz as a function of temperature (-65 to 190ºC) are presented in Figure 9.

EPDM blends show two peaks, one is at a temperature around (-23 to -15ºC) due to glass transition temperature. At this curve PE exhibits two different relaxation peaks, termed α and β, where α peak occurs at the highest temperature around 56ºC, the β peak is for intermediate temperature (-5ºC). The mechanism of α-relaxation is attributed to vibrational or orientational motion within the crystals. The α-relaxation is due to relaxation of –CH$_2$– unit in the crystalline region. This relaxation depends on the amount of branching on both sides, and method of crystallization and heat treatment. The β-relaxation is associated with side branching of polyethylene. This
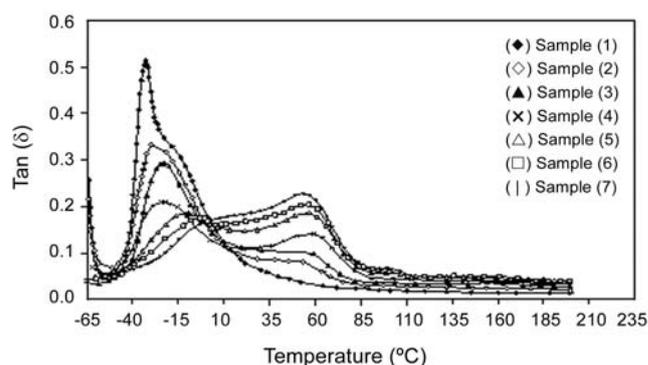
peak always occurs around the same temperature but the concentration of peak depends on the side group. Those side groups may affect the position of the β-peak. Onset of glass transition of EPDM is marked by a sharp decrease in storage modulus, as shown in Figure 8. Sample 1 shows a prominent peak at the temperature -27ºC and the tan δ value is 0.514. In blends (samples 2 to 7) the value of tan δ decreases with LDPE content (Figure 9). The other peak that occurs at a higher temperature and is more prominent is caused by glass transition of EPDM. Figure 9 shows that there is not an obvious T$_g$ peak for samples 4-7 compared to samples 1-3, but they show an α-transition damping peak (62, 58.2, 56.6, 53.7ºC) due to molecular motions in crystalline phase of the LDPE.

SEM Studies
Pure EPDM (Figure 10a) and pure LDPE (Figure 10g) show clearer dimple fracture topology, the black voids visible in these micrographs. However, the other compounds (Figures 10b, c, d, e and f) show similar trends for fracture surface topology. These micrographs represent initial crack tip, initial region and end band region, respectively. The cracks in sample 2 (Figure 10b), with 20 wt% LDPE, change to microcraters. These cracks become larger when the EPDM content increases to 40 wt% (Figure 10e). When the EPDM content increases to 50 wt%, the cracks and microcraters are elongated and semi-continuous phases become evident.

The change in the morphological structure after LDPE content increases to 60 wt% and it is a sign of phase inversion. SEM observations of ion-etched
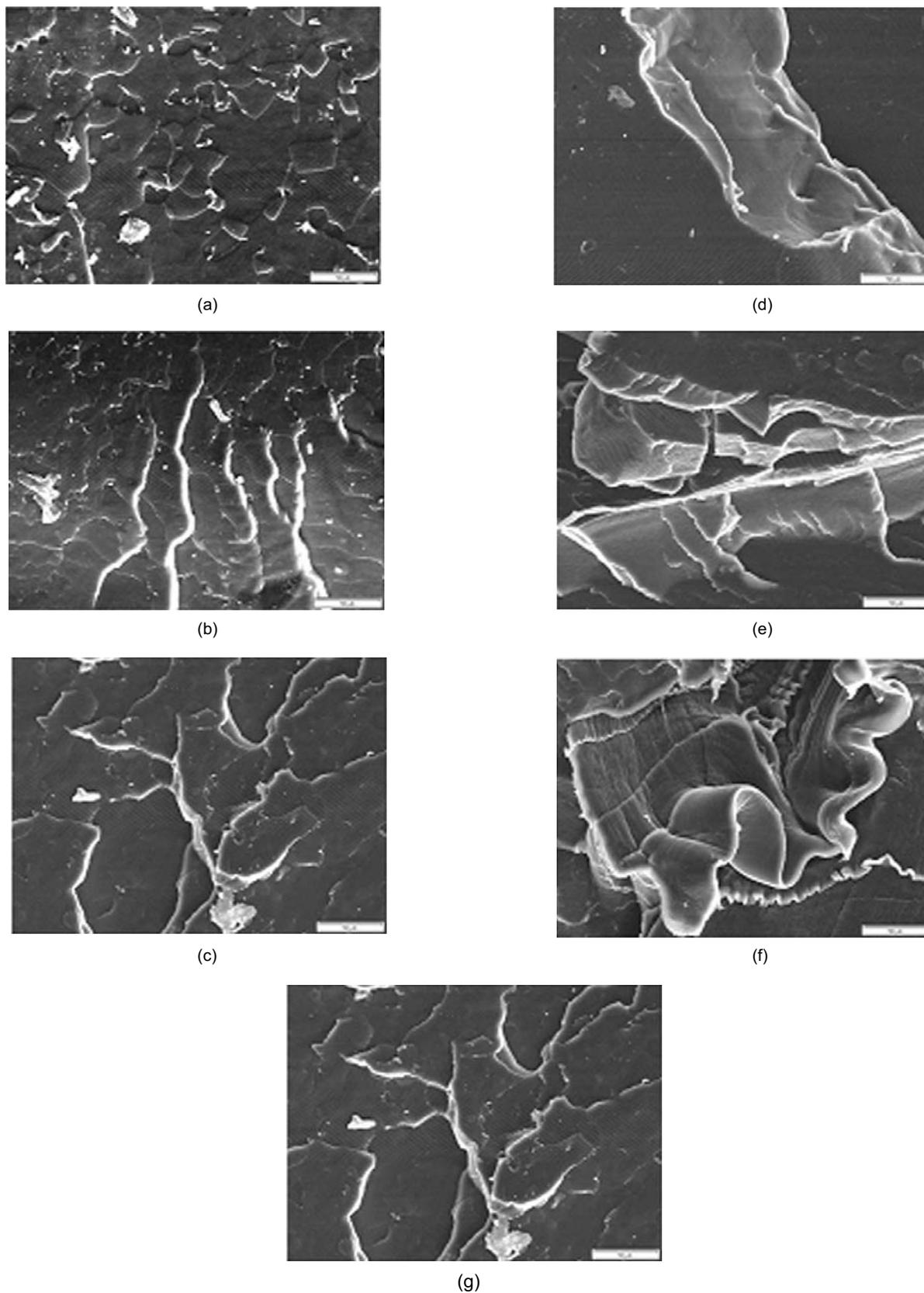
(a)

(b)

(c)

(d)

(e)

(f)

(g)

Figure 10. SEM micrographs of fractured surfaces: (a) EPDM, (b) EPDM/LDPE:80/20, (c) EPDM/LDPE:60/40, (d) EPDM/LDPE:50/50, (e) EPDM/LDPE:40/60, (f) EPDM/LDPE:20/80, and (g) LDPE (Magnification: 20000×).

surfaces of these blends (LDPE/EPDM) have shown that EPDM crystallizes on LDPE lamellae in the surface regions of LDPE crystallites with many tiny LDPE spherulites act as filler reinforced EPDM [30]. In all samples LDPE / EPDM ratio has a major effect on the blend morphology. The minor component (EPDM or LDPE) forms a distinct phase dispersed in the continuous phase (EPDM or LDPE). However, at the higher level of EPDM, LDPE shows rod-like structure as a dispersed phase in the EPDM. At 50:50 blend ratio (Figure 10d) both components seem to have distinct structure.

CONCLUSION

The objectives of this research were to study the compositional effect of LDPE/EPDM blends as insulation compounds. The vulcanized blends should have:
- High dielectric strength to withstand an electric field.
- High volume resistivity to prevent leakage of current.
- Good ageing resistance to prevent damage in environmental ageing.
- Good mechanical properties to resist mechanical forces, vibrations, shocks, etc.
- Good economical advantage to ultimately make the product cost effective.

Generally, one may conclude that the increase of LDPE content in the LDPE/EPDM blend leads to an increase in tensile strength, an increase in modulus and elongation-at-break. Therefore, the mechanical properties of the blend depend on the volume fraction of low density polyethylene. The best insulator should have the lowest dielectric constant, loss factor and highest volume resistivity and dielectric strength. Depending on specific requirements, any composition can be selected as a base polymer in designing insulating compounds.

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