

Electrical Properties of Chemically Modified Polyethylenes

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ABSTRACT

This report describes electrical properties such as space charge distribution, water treeing characteristics and ac breakdown strength in PE (polyethylene) modified by grafting and blending techniques. A control PE shows heterocharge. In AA (acrylic acid)-grafted PE (LDPE-g-AA), the heterocharge observed in PE decreases at low AA contents and homocharge is observed at high AA contents. In NBA (n-butyl acrylate)-grafted PE (LDPE-g-NBA), on the other hand, this heterocharge gets larger at all graft ratios to 0.12%. However, these components decrease the water tree length measured under specific conditions and increase ac breakdown strength. Details of the results are described and their origins are discussed. The results obtained with the LDPE-g-NBA are compared with those of PE/ethylene NBA copolymer blends.

1. INTRODUCTION

THE development of PE (polyethylene) having a better resistance to degradation problems such as water treeing has been one of the major subjects for materials scientists working in the field of electrical insulation of power cables. This improvement can be achieved by a chemical modification of PE, which includes the use of additives, blending, copolymerization and grafting techniques.

The improvement of electrical properties can be achieved simply by mixing proper additives to the PE. However, this method has an inherent disadvantage in that the additives, mostly low molecular weight molecules, migrate through the material and disappear by the so-called blooming effect characterized by a removal of additives at the surface of the material [1, 2]. The blending technique has been employed successfully to improve the electrical properties of PE [3–6]. However, it can generate interfaces between the phases where charge accumulates and hence the local electric field is highly distorted. For example, a series of studies of charge distributions in PE/EVA (ethylene-vinyl-acetate) copolymer EVA laminates has revealed that the charge accumulates at the interfaces between PE and EVA phases, which distorts the electric field distribution in the direction of increasing the electric field in the PE phase [7–11]. Since the blends are a mixture of two or more components, there exist interfaces between the components in the blends, which creates a microscopically inhomogeneous morphology. Then it becomes complicated to interpret the results obtained with these samples. However, blending is still a widely used technique to improve electrical properties of PE. A homogeneous morphology and thus homogeneous

electrical properties can be achieved by the copolymerization of ethylene with comonomers such as styrene and halogen-element containing compounds [12, 13]. However, this technique is not readily accessible and is very complicated. The grafting technique is readily accessible by the so-called reaction extrusion technique [14–16]. This reaction can be completed during the extrusion process, provided that the comonomers are premixed with the PE pellets before the extrusion and that proper extrusion equipment such as a twin screw extruder is available.

In light of this background, in this study, LDPE (low-density polyethylene) was modified by grafting monomers such as AA (acrylic acid) and NBA (n-butyl acrylate), and their electrical properties such as space charge distributions, water treeing characteristics and dielectric breakdown phenomena were measured. For the comparison of results, electrical properties of PE/ENBA (ethylene-n-butyl acrylate) copolymer blends were also measured.

2. EXPERIMENTAL PROCEDURES

2.1. MATERIALS

LDPE without any additives was used as a control PE in the study. This LDPE, a product of Hanyang Chemical Co., Korea, has a density of 0.920 g/cm³, a melt index of 2.0 g/10 min, a weight average molecular weight of ~144000 g/mol and a MWD (molecular weight distribution) of 7 to 8. AA, NBA and DCP (dicumyl peroxide) were purchased from Junsei Chemical Co. and used as received. Their chemical structures are shown in Figure 1.

Three types of ENBA copolymers were used for the preparation of

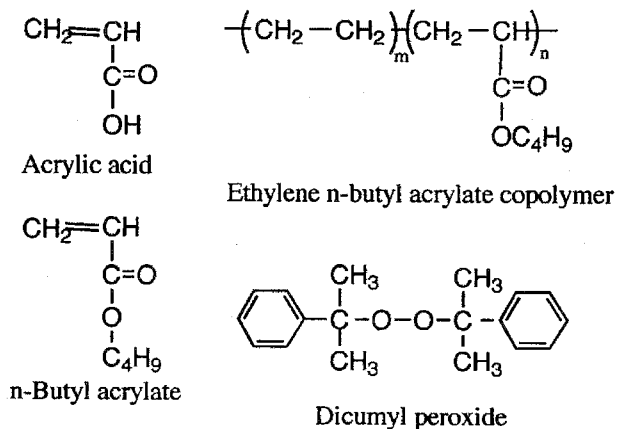


Figure 1. Chemical structures of materials used in this study.

Table 1. Specifications of polymers used in this study.

| material | density g/cm ³ | melt index g/10 min | NBA %wt |
|----------|------------------------------|------------------------|------------|
| LDPE | 0.920 | 2.0 | |
| ENBA 5 | | 3.0 | 5.0 |
| ENBA 19 | | 0.3 | 19.0 |
| ENBA 20 | | 6.0 | 20.0 |

PE/ENBA blends. These copolymers were provided by Quantum Chemical Co., USA, and have better hot adhesive characteristics than the EVA. These samples are different in their nominal weight percentages of NBA and melt indices. Their nominal weight percentages are 5, 19 and 20% and their melt indices are 3.0, 0.3 and 6.0 g/10 min. These samples are designated as ENBA 5, ENBA 19 and ENBA 20. Table 1 shows details of the polymers used in this study.

2.2. MODIFICATION REACTION AND ANALYSIS

Chemical modification of PE was carried out through grafting monomers such as AA and NBA onto the LDPE backbone by reactive processing. The grafting reaction, the schematic diagram of which is shown in Figure 2, was carried out in a twin screw extruder at a rotation speed of 15 rpm and a barrel temperature profile of 165 to 200°C [17]. DCP of 0.02 to 0.05 parts per hundred (phr) resins were used as the initiator for the grafting reaction. All components were premixed in a Henschel mixer before they were fed into the extruder. Grafted samples were washed by immersing in the proper solvent, for example methanol for AA-grafted PE (LDPE-g-AA) and butanol for NBA-grafted PE (LDPE-g-NBA), to remove unreacted monomers. Washed samples were then dried at 70°C for 24 h in a vacuum oven.

The graft ratio was determined by both elemental analysis and FTIR. First, a calibration curve was obtained from both elemental analysis and FTIR spectra for a series of grafted samples. Peak heights were read from appropriate peaks of the FTIR spectra and graft ratios were then determined by reading the value in a calibration curve. Microgel content which could be caused by the thermal decomposition of DCP leading to a crosslinking reaction was determined by a xylene extraction technique. The microgel content was found to be < 0.02% for all grafted samples.

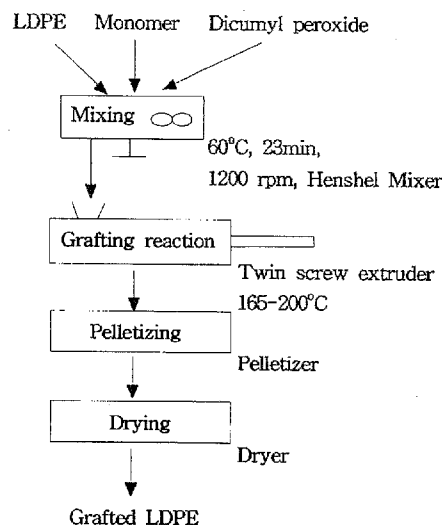


Figure 2. Preparation of grafted PE.

Blends were prepared using the same twin screw extruder set at 180 to 200°C and at a rotation speed of 15 rpm. The blended samples were kept in a dessicator until tested.

2.3. MEASUREMENTS OF ELECTRICAL PROPERTIES

Charge distributions were measured by a PEA (pulsed electroacoustic) method the measurement principles and techniques of which are described elsewhere [18, 19]. It consists of an electric pulse generator (pulse width 10 ns, pulse height -2 kV), HV source ($+50$ kV_{dc}), a test cell with a piezoelectric detector, and a data acquisition system. The piezoelectric detector was made of 9 μm thick PVDF (polyvinylidene fluoride) film. The data acquisition system consists of wide band preamplifier, digitizing oscilloscope and a personal computer. When an electric pulse is applied to the anode, the charges in the sample generate an acoustic pressure wave which obeys $f = qE$, where f is the electric force, q is the charge and E the electric field. The waves propagate through the sample and then reach the piezoelectric detector where the acoustic pressure waves are transformed into the electric signal, which represents the space charge distribution. The measured signal was calibrated with the area of a calibration peak obtained at 5 kV. Since the signal is attenuated while traveling through the lossy medium, the charge peak near the cathode where the piezoelectric detector is located was taken as a significant data.

Samples for space charge density measurements were prepared by compression molding at 130°C for 10 min. The thickness of the grafted PE samples was typically 0.7 mm and that of the semicon electrodes was typically 0.2 mm. The voltage was applied for 30 min and then the sample short circuited. Immediately after short circuiting, the charge distribution was measured, which means that the charge distributions shown in this report are the profiles of charge remaining after the discharge. The delay time was < 3 min.

Semiconductive electrodes were used for the measurements of charge distributions. EVA (ethylene-vinyl-acetate)-based semicon electrodes were prepared by curing at 180°C for 20 min using a hot press. These electrodes were vacuum degassed at 80°C for 100 h before being used

as electrodes. The diameter of the top electrode is 1.5 cm. The raw material is the commercial product used for the strand shield of medium voltage power cables in Korea, which is a mixture of an olefin polymer with a polar functional group, conductive carbon black, processing aids and antioxidants. Since it has been reported that the constituents of semiconductive electrodes change charge distributions in PE [19, 20], the same type of semiconductive electrodes were used throughout the study.

The water tree length was determined by measuring the length of water trees grown under specific conditions. A schematic diagram of the test cell for the measurement of water tree length is shown in Figure 3. Slab samples having 8 indentations were prepared by compression molding at 150°C for 10 min using a mold. The distance from the needle tip to the ground was 2.0 mm and needles with a tip radius of 5 μm were obtained from Ogura Jewel Co., Japan. The electrolyte was 0.1 M AgNO_3 aqueous solution. This solution is known to produce silver trees [21]. Water trees were grown at 10 kV for 96 h at room temperature. The samples having water trees were stained by methylene blue and the water tree length was determined by measuring the length of the longest branch of the tree. The value averaged from 8 indentations was taken as the water tree length.

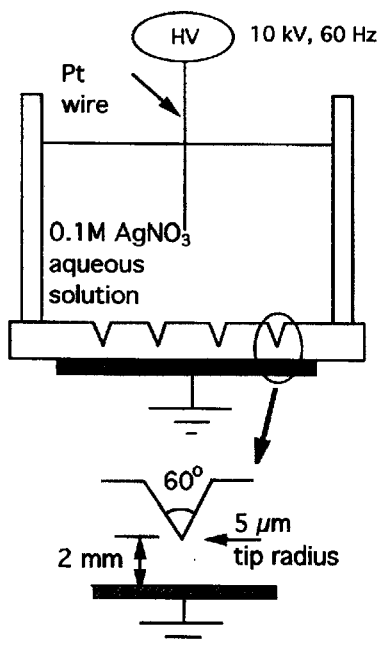


Figure 3. Schematic representation of test cell for water tree length measurement. Electrolyte: 0.1 M AgNO_3 aqueous solution, test conditions: 60 Hz, 10 kV, 96 h, room temperature.

The ac breakdown strength of samples was measured using a Hypotronics ac breakdown tester. The electrode edge was rounded in order to reduce the local enhancement of the electric field. Samples for breakdown tests were prepared by compression molding at 130°C for 10 min. The typical thickness of the samples was 70 to 75 μm . All tests were carried out at 1 kV/s in an insulating oil bath. Mean values from 8 to 10 samples were taken as the ac breakdown strength.

3. RESULTS AND DISCUSSION

3.1. CHARACTERIZATION OF GRAFTING REACTION

Figure 4 shows the FTIR spectra of grafted PE. FTIR spectra were taken after the as-grafted PE had been washed using fresh solvents. Each monomer has its own characteristic peaks which indicate the existence of the monomer. As shown in Figure 1, both AA and NBA contain carbonyl (C=O) groups, the environment of which varies depending on the sample. The 1714 cm^{-1} peak is for the C=O group in AA and the 1737 cm^{-1} peak is that in NBA. The presence of these peaks in the solvent-washed samples is an indication that the monomers were successfully grafted onto the PE chains.

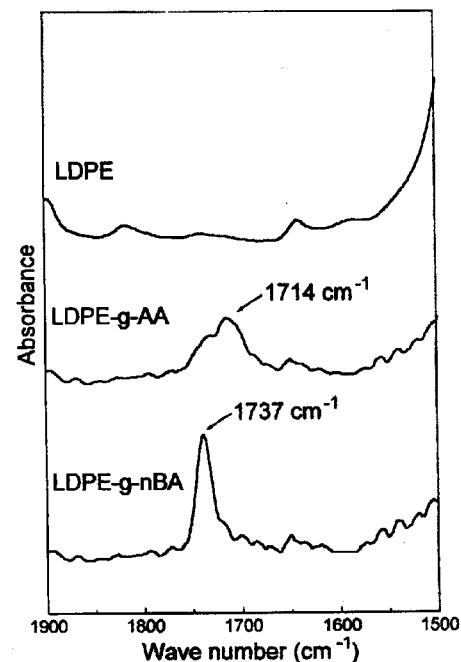


Figure 4. FTIR spectra of grafted PE.

3.2. CHARGE DISTRIBUTIONS IN GRAFTED SAMPLES

Figure 5 shows the charge distributions of LDPE-g-AA and LDPE-g-NBA samples. Figure 6 shows the charge near the cathode as a function of graft ratio for both as-grafted and solvent-washed samples.

The control PE used in this study shows heterocharge, *i.e.* positive charge at the cathode side and negative charge at the anode side. When AA is grafted on this PE, the heterocharge observed in the control PE sample decreases with the increase of AA graft ratio and homocharge develops at high graft ratios. The amount of charge in solvent-washed samples is smaller in the case of heterocharge at low graft ratios and larger in the case of homocharge at high graft ratios. The difference between solvent-washed and as-grafted samples is the existence of unreacted monomers. Therefore, the result in Figure 6 indicates that the unreacted AA molecules encourage the formation of heterocharge. The encouragement of heterocharge formation of unreacted molecules was also observed in MAH (maleic anhydride)-grafted PE [17]. Unreacted molecules are expected to remain as mobile molecules which can be oriented

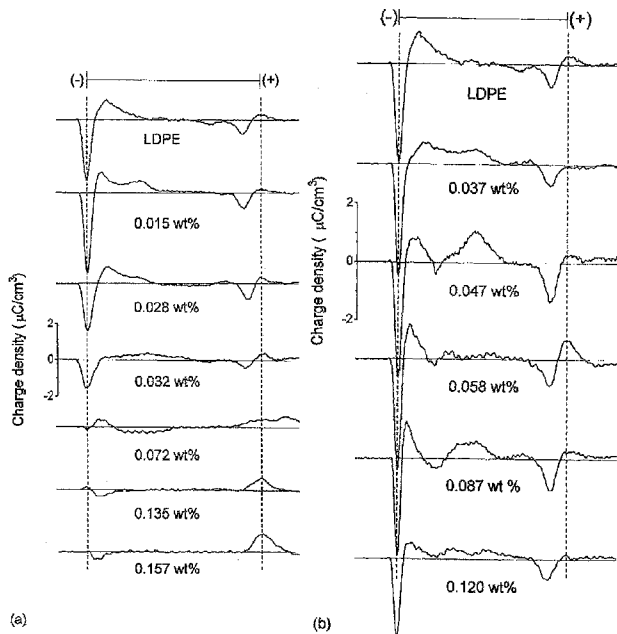


Figure 5. Charge distributions at 40 kV of (a) AA-grafted and (b) NBA-grafted PE.

or migrate under the electric field to form heterocharge. On the other hand, all LDPE-g-NBA samples show heterocharge, the amount of which is larger than that of the control PE sample. In this case, the solvent-washed grafted samples also show smaller charge than as-grafted samples.

A similar phenomenon was found in MAH-grafted PE (LDPE-g-MAH) [17]. When the MAH molecules were grafted on the PE chains, heterocharge developed in the control PE decreased with the increase of MAH graft ratio. The decrease of heterocharge as a function of MAH graft ratio was attributed to the enhancement of trapping of injected charge at MAH molecules. A similar explanation could be applied to the present observation with LDPE-g-AA. It seems that the charge injected from the electrodes during voltage application is trapped at AA sites, resulting in an increase of homocharge accumulation. As the concentration of these molecules increases, the density of homocharge trapped at these molecules will increase. This homocharge increase will result in an apparent decrease of heterocharge in PE. In this case, it seems that the carbonyl groups in AA act as trapping sites for the injected charge.

The present results, along with the results with LDPE-g-MAH, suggest that certain chemicals, for example MAH and AA, can change the type and amount of charge accumulated in PE when they are grafted on the PE chains. At this moment, however, the reason why NBA did not obey this trend is not known.

3.3. TREE LENGTH AND AC BREAKDOWN STRENGTH IN GRAFTED SAMPLES

Figure 7 shows the results of water treeing tests with grafted samples. This test was done with solvent-washed samples. As shown, the water tree length of AA-grafted samples decreases first at graft ratios to 0.05% and then remained unchanged at higher graft ratios. The water

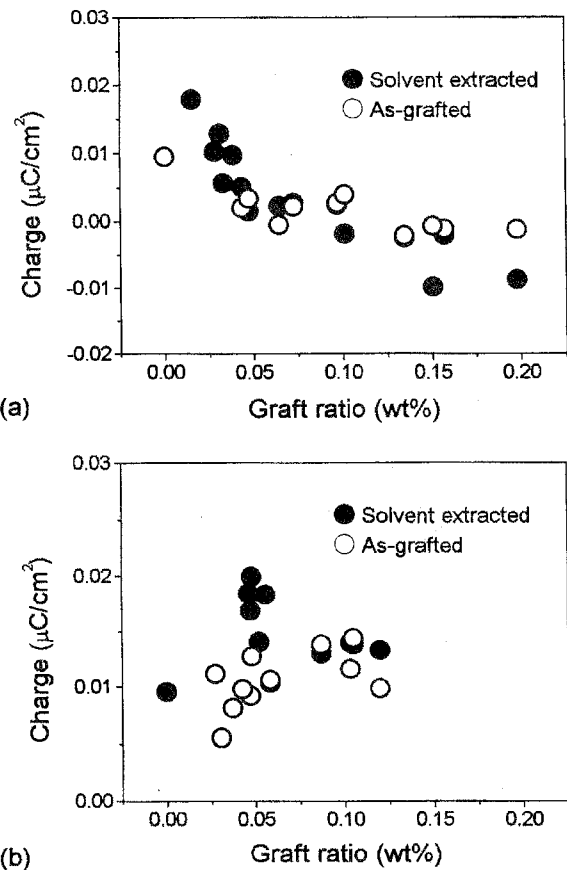


Figure 6. Charge near the cathode as a function of graft ratio for as-grafted (hollow circle) and solvent-extracted samples (solid circle): (a) LDPE-g-AA, (b) LDPE-g-NBA.

tree length of NBA-grafted samples is generally smaller than in the control PE.

Figure 8 shows the mean values of ac breakdown strength of LDPE-g-AA (Figure 8(a)) and LDPE-g-NBA (Figure 8(b)) for two concentrations of DCP, 0.02 and 0.05 parts per hundred resins (phr). A general trend is that mean ac breakdown strength of grafted samples is higher than that of the control PE, which holds true for LDPE-g-AA and some LDPE-g-NBA grafts. A maximum mean breakdown strength was observed at the graft ratio of 0.025 to 0.05% for LDPE-g-NBA, and all AA-grafted samples showed higher breakdown strength than the control LDPE samples.

The present results indicate that the electrical properties of the PE are improved when certain monomers are grafted on the chains. For example, the type and amount of space charge developed in the PE could be adjusted, while water treeing characteristics and ac breakdown strength could be improved. It is also found that the graft ratio where a maximum improvement is achieved is different depending on the type of monomer and the concentration of initiator.

Although electrical properties of PE are improved by grafting monomers on the chains, there are other influences to be evaluated before using this material as an insulating material. These are the effects of microgels and unreacted monomer. As mentioned in Section 2.2, the microgel content of grafted PE prepared by the procedure specified in the

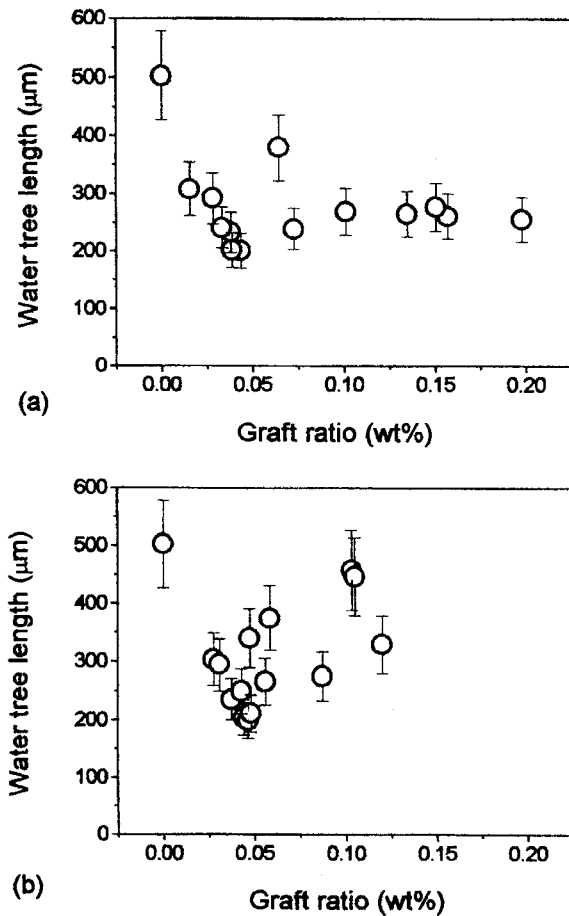


Figure 7. Water tree length as a function of graft ratio: (a) LDPE-g-AA, (b) LDPE-g-NBA.

present study is < 0.02%. This amount of microgel would not deteriorate their thermal and mechanical properties. However, although its concentration is relatively low, the microgel could accelerate the degradation of insulation, which would result in an instability of long-term electrical properties. In addition, there are unreacted monomers in the grafted PE. These unreacted monomers are free mobile molecules which could be oriented or migrate under the electric field, resulting in heterocharge formation, as confirmed with MAH-added PE [22]. This could also lead to unexpected electrical properties. Therefore, more work is needed to reduce the concentration of microgels and unreacted monomers in grafted PE.

3.4. CHARGE DISTRIBUTIONS IN BLENDED SAMPLES

Figure 9 shows charge distributions in control PE and the ENBA copolymers. The PE shows heterocharge while the ENBA copolymers show different types of charge depending on the sample. The ENBA 5 shows heterocharge while the ENBA 19 shows positive charge broadly distributed over the sample.

The charge distribution in ENBA 20 looks similar to that of ENBA 19 in that positive charge is distributed somewhat broadly over the sample at the anode side, but it has negative charge at the cathode side. The

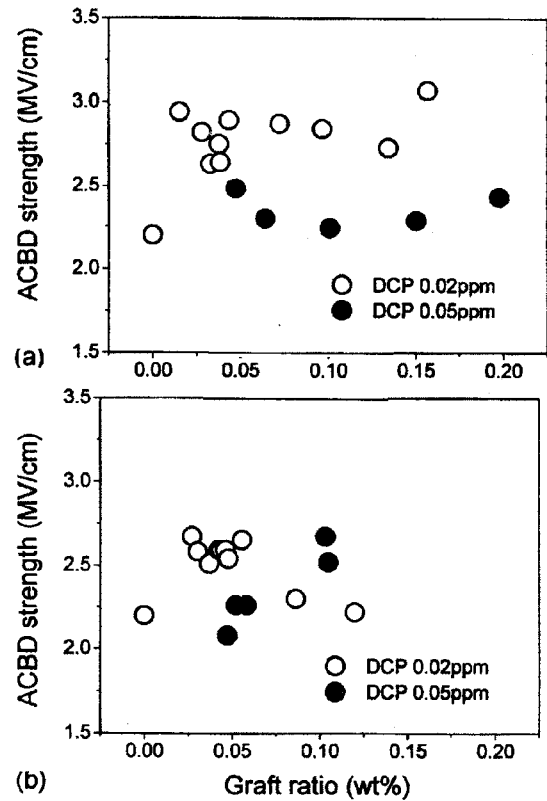


Figure 8. Mean ac breakdown strength of grafted PE. (a) LDPE-g-AA, (b) LDPE-g-NBA. DCP content 0.02 ppm (hollow circle), 0.05 ppm (solid circle).

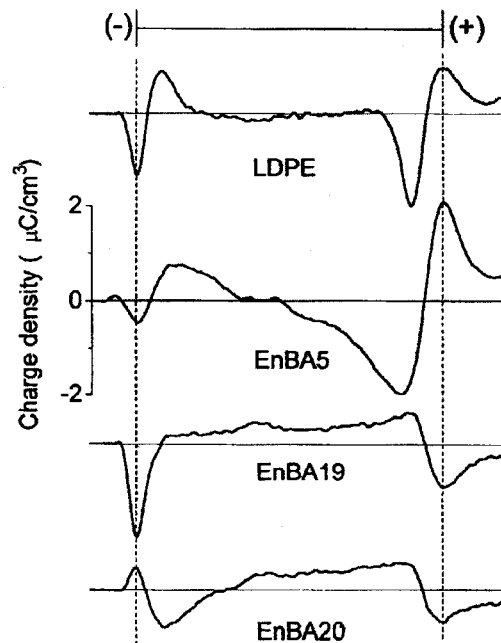


Figure 9. Charge distributions at 40 kV/mm in control PE and ENBA copolymers.

shapes of the charge distributions in both ENBA 19 and ENBA 20 are similar to those of EVA containing higher VA (vinyl-acetate) content [8].

It seems that the injection of positive charge dominates in both samples, as explained for EVA [8, 23–25].

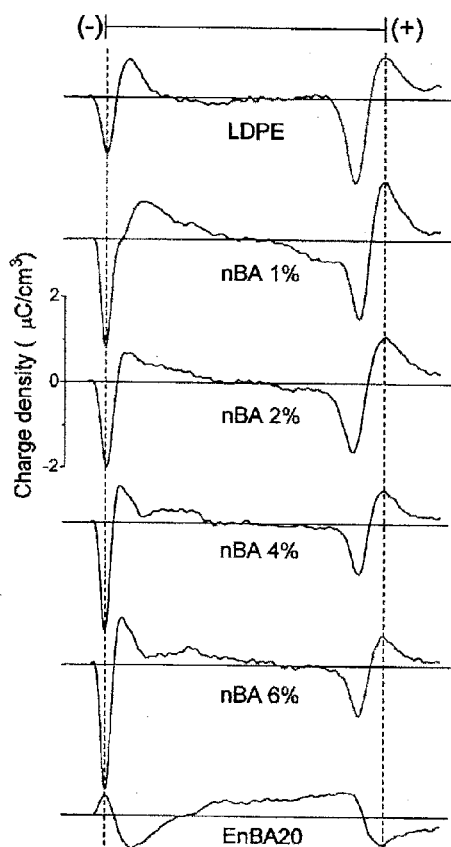


Figure 10. Charge distributions at 40 kV/mm in PE/ENBA 20 blends.

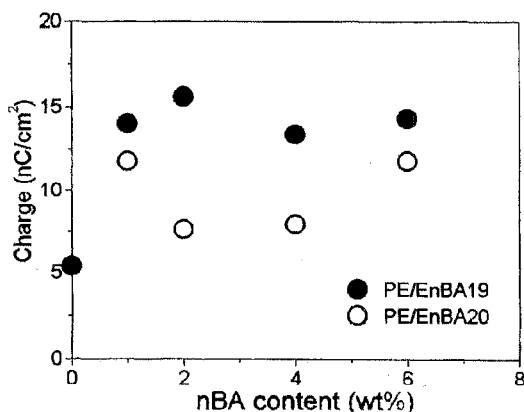


Figure 11. Charge near the cathode as a function of NBA content in the blends.

Figure 10 shows charge distributions in selected PE/ENBA blends, PE/ENBA 20 blends in this particular case. As shown, heterocharge develops in PE/ENBA 20 blends at the NBA content in the blends to 6%wt, although ENBA 20 itself develops homocharge. Figure 11 shows the change of charge near the cathode as a function of NBA content in PE/ENBA blends. The heterocharge in both PE/ENBA 19 and PE/ENBA

20 blends is larger than that in control PE, as shown in the present results, whereas the heterocharge in PE/EVA blends decreases with the increase of VA content, as shown in our previous reports [8, 9]. Both copolymers were expected to impose the same effects, because both contain carbonyls in their chain structures, although the environments around the carbonyl are slightly different. In particular, the charge distributions of the parent copolymers are very similar. Compare Figure 9 of the present report and Figure 1 of [9]. The fact that the charge distributions in EVA and ENBA copolymers are very similar suggests that the carbonyls in both copolymers ought to impose similar effects. However, the observation with these two blends is quite different. The exact reasons for the differences are not known. However, they could be related to the role of interfaces.

3.5. TREE LENGTH AND AC BREAKDOWN STRENGTH IN BLENDED SAMPLES

Figure 12 shows the water tree length of PE/ENBA blends as a function of NBA content. The water tree length of the blends increases first when NBA content in the blends reaches 3%wt and then decreases at higher NBA content. At the graft ratio of 4%wt, the water tree length of ENBA 5 blend is the largest while that of ENBA 19 blend is the smallest.

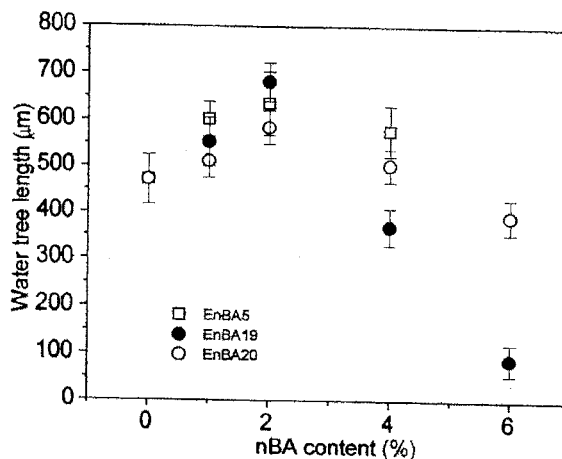


Figure 12. Water tree length of PE/ENBA blends as a function of NBA content.

The exact reasons why the tree length of ENBA 19 blend is smaller than that of ENBA 20 blend at high NBA content are not known. ENBA 19 and ENBA 20 are different in NBA content and melt indices, as listed in Table 1. The NBA contents of these copolymers differ by only 1%. Because the NBA content in the blends was set at the same concentration, the differences in water tree length should not originate from the differences in NBA content. A comparison of the extents of the difference and the NBA content of the parent copolymers suggests that the NBA content of the parent copolymers may not be a major factor in differentiating the water tree length of the blends. The differences could be related to the morphology of the blends. Since the melt indices of these two ENBA differ by a factor of 20, e.g. 0.3 g/10 min for ENBA 19 and 6.0 g/10 min, their flow properties and thus their dispersion characteristics for ENBA in PE could be different. The nature and the role of interfaces could consequently be different. The resulting differences in morphology of

the blends could make the water treeing characteristics different. More work is needed to confirm this speculation.

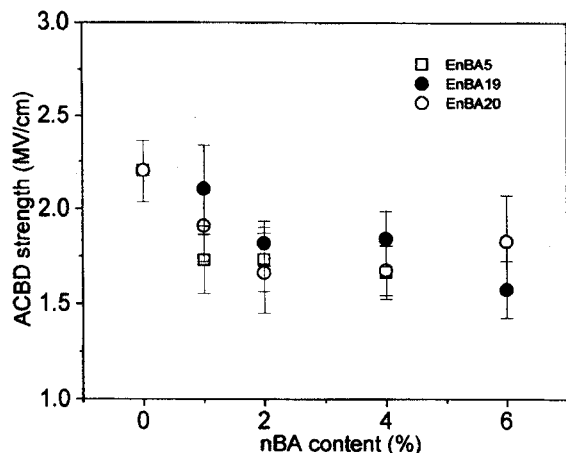


Figure 13. Mean ac breakdown strength of PE/ENBA blends as a function of NBA content.

Figure 13 shows average values of the ac breakdown strength of various PE/ENBA blends as a function of NBA content. As shown, the ac breakdown strength of the PE decreases when the ENBA is blended in. At the graft ratio of 4%wt, the ac breakdown strength is ENBA 19 > ENBA 20 ~ ENBA 5. These results indicate that the electrical properties could not be improved by blending ENBA with the PE in the range of < 3%wt of NBA.

4. CONCLUSIONS

THE control LDPE shows heterocharge. This heterocharge decreases with the increase of graft ratio of AA, which we attribute to the increase of homocharge trapped at carbonyl groups acting as trapping sites. At high graft ratio, homocharge is seen. No such decrease of heterocharge is found in NBA-grafted PE. Water treeing characteristics and ac breakdown strength are improved by grafting monomers such as AA and NBA on the PE chains. However, there seems to be an optimum graft ratio having a maximum improvement of electrical properties. On the other hand, the blending of ENBA with PE did not improve the electrical properties.

ACKNOWLEDGMENT

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