



## Effect of Sample Preparation on Water Tree Resistance of XLPE/SEBS Blends

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### ABSTRACT

The effects of an aromatic polymer, such as poly[styrene-*b*-(ethylene-co-butylene)-*b*-styrene] (SEBS), and its preparation processes on water tree resistance, morphology, crystallinity, dielectric constant and dielectric loss tangent of cross-linked polyethylene (XLPE)/SEBS and XLPE/SEBS/ethylene vinyl acetate (EVA) blends were investigated. The XLPE/SEBS samples were characterized by means of differential scanning calorimetry, scanning electron microscopy, dielectric spectral analysis and the accelerated water treeing experiment. The results clearly showed that although SEBS was partially compatible with LDPE and it was dispersed evenly in XLPE matrix, the blends demonstrated excellent dielectric performances, such as dielectric constant and dissipation factor. The crystallinity of XLPE/SEBS blends was decreased with increase in SEBS content and it was further decreased with the addition of EVA. SEBS could effectively improve the water-tree resistance of XLPE and both SEBS and EVA were found to improve the water tree resistance of XLPE synergistically as expected. The differences in sample preparation processes could change the phase morphology and dielectric performance of the blends, and could also influence the dispersion of SEBS in XLPE matrix as well as the crystallinity of the blends. Different preparation processes led to different water tree resistance behaviours of the samples. The improvement of SEBS dispersion in XLPE matrix helped to improve the water tree resistance of XLPE.

### Key Words:

poly[styrene-*b*-(ethylene-co-butylene)-*b*-styrene] (SEBS);  
water tree resistant;  
cable insulation;  
dielectric constant;  
synergetic effect.

### INTRODUCTION

Polyethylene is mainly used as thermoplastic insulating material for medium and high voltage power cables because of its characteristics of easy maintenance and low transmission loss. Nowadays, cross-linked polyethylene (XLPE) power cables are widely used in underground transmission lines. Owing to the combination of electrical stress and water penetration, XLPE power cables are susceptible to a form of

degradation known as "water treeing" [1].

Since the discovery of water trees in underground cables in 1968, much work has been devoted to understanding the initiation and growth mechanism of water treeing and to development of water tree-resistant polyethylene compounds [2]. It was shown in previous works that the water tree resistance of XLPE could be improved by chemical and physical modifications,

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such as blending, additives, copolymerization and other techniques [3-7]. Our research group has improved water tree resistance of polyethylene through the grafted modification of sodium acrylate or the blending of ethylene ionomers with XLPE [8-10]. Besides polar modification or blending of polar copolymers, our research group has investigated the effect of non-polar polymers, e.g. polyolefin elastomer (POE), on the performance of water tree resistance of XLPE insulation, and investigated the synergistic effect of POE and EVA on water tree resistance of XLPE [11]. Meanwhile, our group also studied the influence of aromatic polymer on water tree growth of XLPE, e.g. styrene butadiene rubber (SBR), which was also confirmed by Nagasaki et al. to resist the growth of bow-tie tree [12].

Recently, in a report authors demonstrated that styrene-ethylene/butadiene-styrene copolymer shows clear resistance to water treeing in XLPE [13]. In this work, the effects of sample preparation on water tree resistance and physical properties of XLPE/SEBS and XLPE/SEBS/EVA blends have been investigated.

## EXPERIMENTAL

### Materials

Polyethylene of LDPE (100 BW) grade was obtained

from ExxonMobil in Saudi Arabia, which has a melt flow index (MFI) of 2.0 g/10 min and a density of 0.9225 g/cm<sup>3</sup>. Styrene-ethylene-butadiene-styrene (SEBS Kraton G1651H) was purchased from Shell Chemical Co. in America, had a density of 0.91 g/cm<sup>3</sup> and contained 31 wt% styrene. Ethylene vinyl acetate (EVA VA600) was supplied from Hyundai Petrochemical Corp. in South Korea, which has MFI of 6.0 g/10 min and a density of 0.95 g/cm<sup>3</sup>. Dicumyl peroxide (DCP) (Chemical Reagent) was supplied from Shanghai Gaoqiao Petroleum Co., Ltd., China, with purity higher than 99.5%. Sodium hydroxide (NaOH) (AR) and sodium chloride (NaCl) (AR) were obtained from Shanghai Medicine Chemical Co., Ltd., China.

### Sample Preparation

The samples were prepared by two processes:

Process 1: LDPE, SEBS, EVA and DCP were directly blended in a Haake Rheometer RC90 at a temperature of 115°C and a rotor speed of 60 rpm for 10 min.

Process 2: LDPE and SEBS were first blended in the Haake Rheometer at a temperature of 170°C and a rotor speed of 60 rpm to obtain LDPE/SEBS blends, and then the prepared blends, EVA and DCP were blended again in a rheometer at a temperature of 115°C and a rotor speed of 60 rpm for 10 min. All the blends were mould pressed at 175°C and 10 MPa for

**Table 1.** Composition of the XLPE blends in this study.

Sample	Notation	LDPE (phr)	SEBS (phr)	SEB (phr)	DCP (phr)
XLPE	XLPE	100	0	0	2
LDPE/SEBS	LDPE/S15/P1	100	15	0	0
LDPE/SEBS/EVA	LDPE/S15/E1/P1	100	15	1	0
LDPE/SEBS	LDPE/S15/P2	100	15	0	0
LDPE/SEBS/EVA	LDPE/S15/E1/P2	100	15	1	0
XLPE/SEBS	XLPE/S5/(P1/P2)	100	10	0	2
XLPE/SEBS	XLPE/S10/(P1/P2)	100	10	0	2
XLPE/SEBS	XLPE/S15/(P1/P2)	100	10	0	2
XLPE/SEBS/EVA	XLPE/S5/E1/(P1/P2)	100	5	1	2
XLPE/SEBS/EVA	XLPE/S10/E1/(P1/P2)	100	10	1	2
XLPE/SEBS/EVA	XLPE/S15/E1/(P1/P2)	100	15	1	2

XLPE/S15/E1/(P1/P2) stands for XLPE/S15/E1/P1 and XLPE/S15/E1/P2, the sample contains 15.0 phr SEBS, 1.0 phr EVA in XLPE matrix, and were prepared by processes 1 and 2, respectively.

15 min to obtain samples in several series. The composition of the XLPE blends is listed in Table 1.

### Characterization

#### *Differential Scanning Calorimetry*

The DSC measurements of samples were performed by using the Netzsch DSC 200F3. The samples were heated from 20°C to 150°C at a heating rate of 20°C/min, the samples were kept at 150°C for 3 min to eliminate the heat history and were then cooled to 20°C at a rate of 20°C/min, and both exothermic and endothermic curves were recorded as a function of temperature. A value of 290 J/g was set as the melting enthalpy of 100% crystalline to calculate the crystallinity of the samples from the melting enthalpies [14]. All the DSC measurements were performed under N<sub>2</sub> atmosphere. The instrument was calibrated with an Indium standard.

#### *Scanning Electron Microscopy*

The morphologies of the fractured surfaces of samples were characterized by a JEM-7401F scanning electron microscope, Japan. Cylindrical samples were broken in liquid nitrogen and the fractured surfaces of the samples were etched in toluene for 48 h. The etched surfaces were sputtered with thin layers of gold to avoid the accumulation of charge.

#### *Dielectric Properties*

Dielectric constant and dielectric loss measurements in the frequency range of 40 Hz-1 MHz were performed using an impedance analyzer (Agilent 4294A) with 16451B Dielectric Test Fixture at room temperature, applying 1.0 V AC voltage across two opposite sides of the plane samples, and only one sample of each blend was measured for dielectric properties evaluation. The data of impedance and phase angle measured were converted into the relative dielectric constant and dielectric loss, considering the appropriate geometric coefficient. Thin films with thickness of around 0.45 mm were used for dielectric measurements.

#### *Water Treeing Test*

The circular specimens for water treeing test were prepared based on sandblasted test. The test cell was

similar to the reference testing cell, and the thickness of specimens was 2.0 mm [15]. The experiment of water treeing was carried out under the action of an applied voltage of 5 kV at the frequency of 8 kHz at room temperature for 15 days, and only one sample of each blend was analyzed. After a pre-set time, all samples were stained with methylene blue and blend specimens were each sliced up at least to 25 slices respectively with 0.2 mm thickness. The water tree length of each slice was determined by measuring the length of the longest branch of the tree. The water tree length (WTL) distributions were obtained by also using the two-parameter Weibull distribution analysis, and the characteristic water tree length that the cumulative probability of water tree length of 69.03% was used to evaluate the water treeing results [16-19].

The probability of finding a value of water tree length less than or equal to a length  $L_t$  at a defined time is given by:

$$F(L) = 1 - \exp \left[ - \left( \frac{L_t}{L_{ts}} \right)^\beta \right] \quad (1)$$

where  $L_{ts}$  is the characteristic residual insulation thickness (the relative value) and the function of time;  $L_t$  is the length of water tree grown for a defined time,  $\beta$  is the shape parameter for the Weibull distribution, which is a measure of the spread of water tree length. Higher  $\beta$  values indicate a narrower data distribution. Eqn (1) can also be impressed as that:

$$\ln \left( \ln \left( \frac{1}{1 - F(L)} \right) \right) = \beta \ln(L_t) - \beta \ln(L_{ts}) \quad (2)$$

and experimental results were used to fit the function curves.

The cumulative probability,  $F$ , was calculated using the equation:

$$F = \frac{i - 0.3}{N + 0.4} \quad (3)$$

where  $i$  is the sample number having a cumulative probability of  $F$ , and  $N$  is the number of the trees, and in our paper,  $N$  is the slice number of each sample.

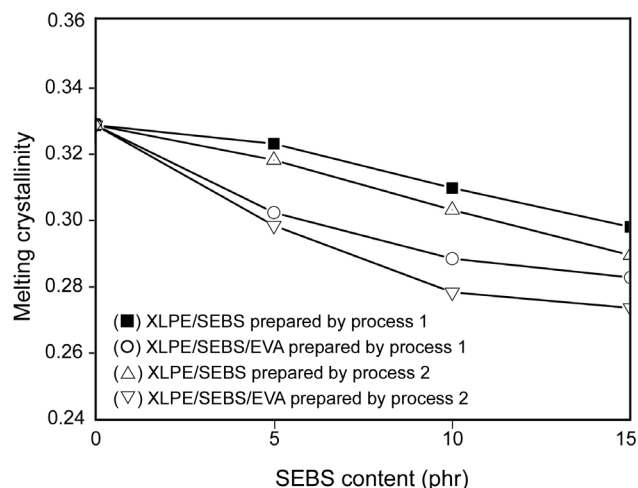
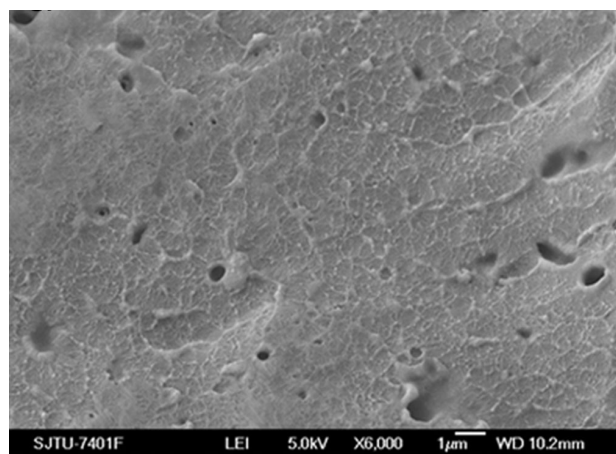


Figure 1. Crystallinity of the blend samples.

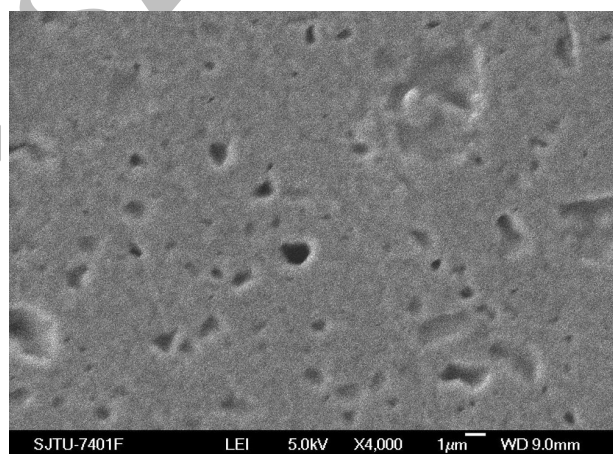
## RESULTS AND DISCUSSION

### DSC Characterization

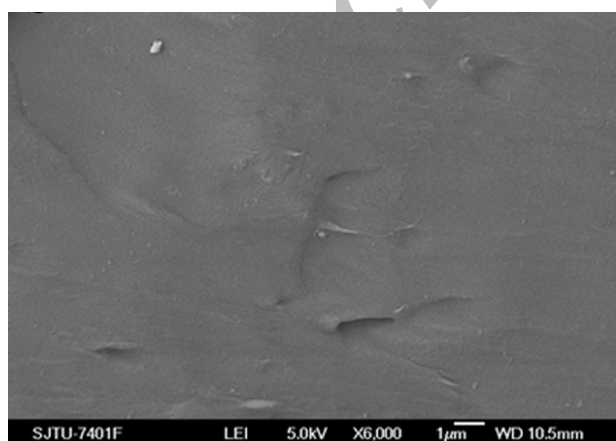
It was shown in the previous work that the water treeing behaviours are associated with the crystallinity of polyethylene [11]. The crystallinity of our blend samples was measured by DSC and the data are presented in Figure 1. It can be seen that: (i) the crystallinity of samples decreases with the increasing content of SEBS; (ii) the blends prepared by process 1 shows higher values of crystallinity when compared with those prepared by process 2. These results indicate that the introduction of SEBS shows a negative effect on the crystallization of XLPE and that the well dispersion of SEBS phase could significantly hinder the crystallization. The



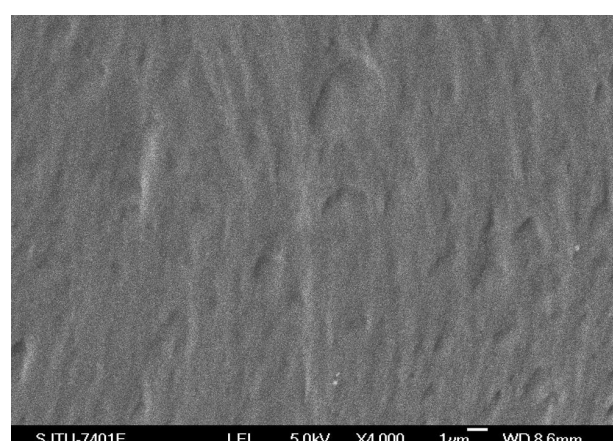
(a)



(c)



(b)



(d)

Figure 2. SEM images of the etched blends: (a) LDPE/S15/P1, (b) XLPE/S15/P1, (c) LDPE/S15/P2 and (d) XLPE/S15/P2.

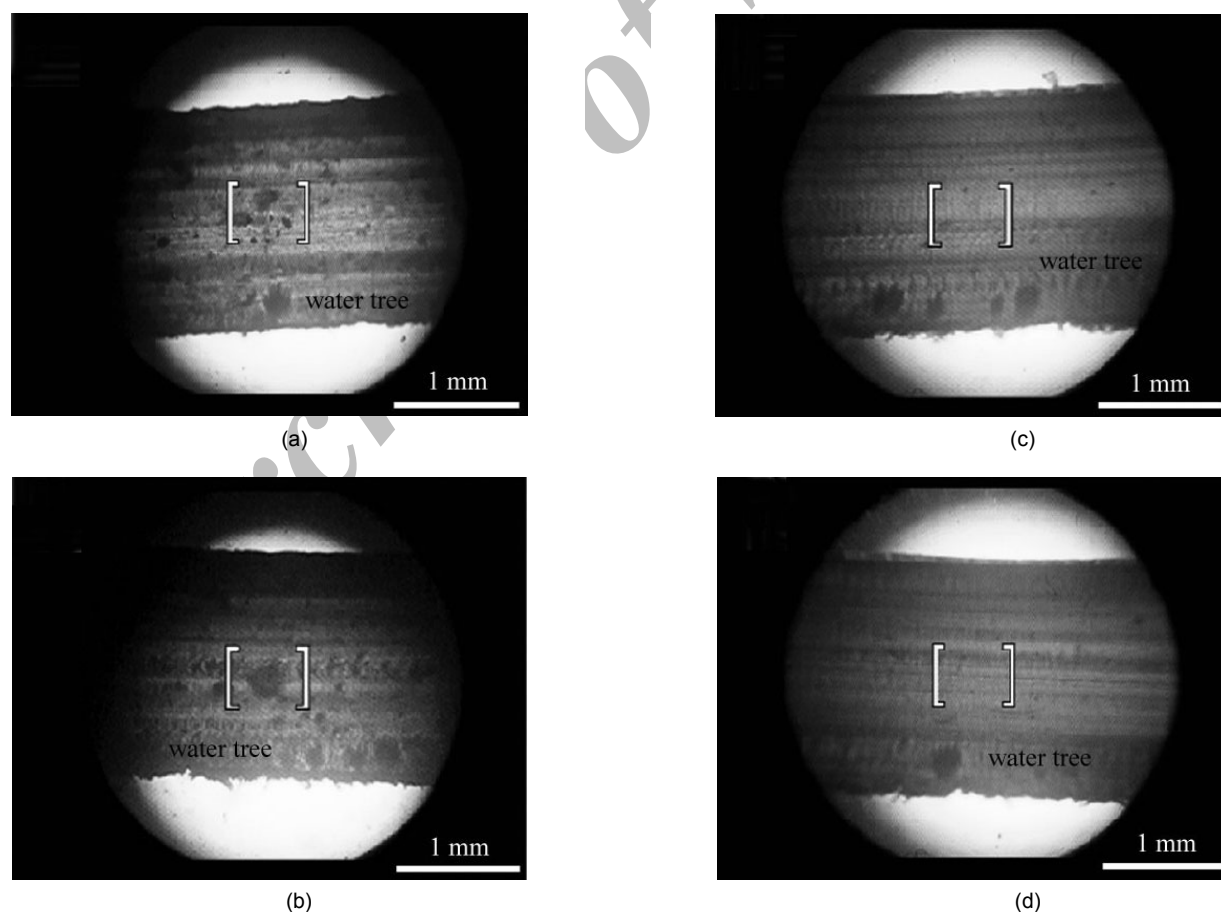
decrease of crystallinity may be attributed to the distortion of in the crystal structure resulting from the co-cross-linking between LDPE and SEBS. The fine dispersion of SEBS can increase the cross-link density and thus the crystallinity shows lower values in the blend samples prepared by process 2. Interestingly, the introduction of EVA can further cause a decrease of crystallinity, indicating that EVA is also co-cross-linked with LDPE.

### Morphology

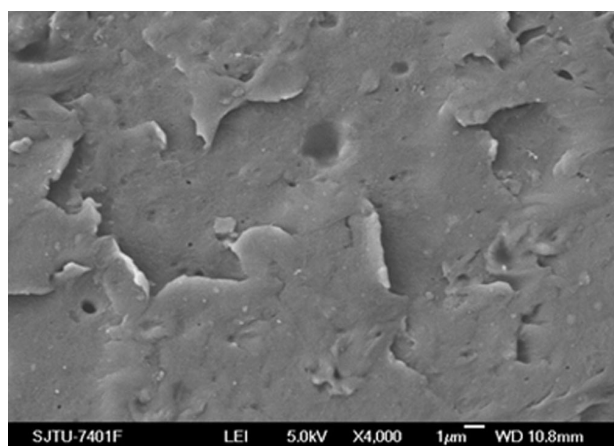
Figure 2 shows the SEM images of the etched blends. It can be seen that: (i) SEBS phase in LDPE blends was chemically etched by toluene and the non-cross-linked blends display sea-island morphology; (ii) after cross-linking reaction, two-phase morphology could not be found in the blends, indicating that SEBS could not be etched out after cross-linking with LDPE. The morphological change reveals that SEBS

can easily react with radicals generated from DCP decomposition.

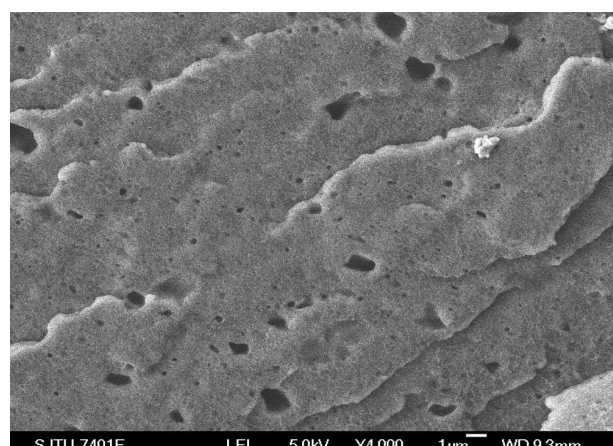
Comparison of the morphology of the blends prepared by different processes, reveals that the average diameter of the SEBS particles in the blends prepared by process 2 are smaller than that of the blends prepared by process 1. On the other hand, the blends prepared by process 2 show more uniform morphology when compared with the blends prepared by process 1. The results can be understood that the SEBS has a high melting onset, and the SEBS particles could not be melted in process 1. The aforementioned conclusion could be further confirmed by the samples stained with methylene blue after the water treeing process, and it can be seen from Figures 3a and 3c that the blends prepared by process 2 show a clear view, whereas the blends prepared by process 1 contain some opaque areas, which should be ascribed to the unmelted SEBS



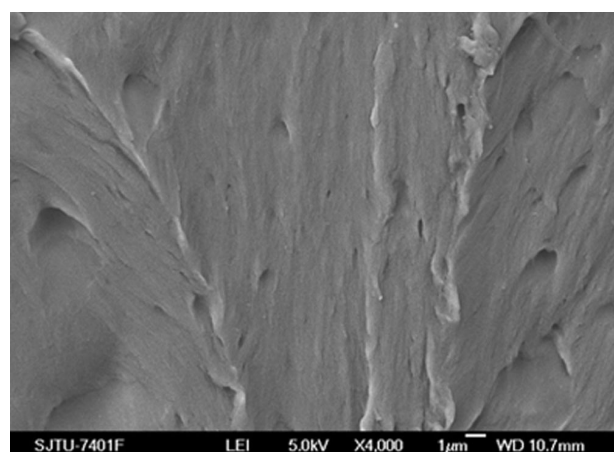
**Figure 3.** Optical microscopy images of the stained slices with methylene blue after the water treeing process: (a) XLPE/S15/P1, (b) XLPE/S15/E1/P1, (c) XLPE/S15/P2 and (d) XLPE/S15/E1/P2.



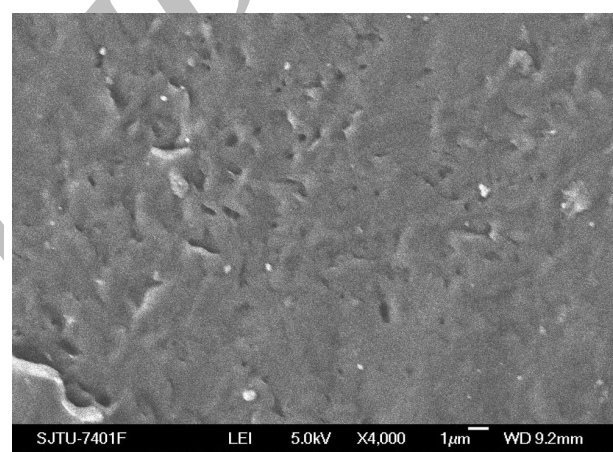
(a)



(c)



(b)



(d)

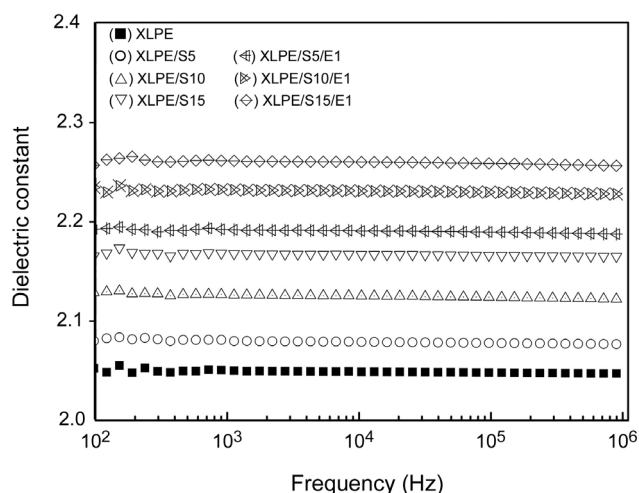
**Figure 4.** SEM images of the etched blends with EVA: (a) LDPE/S15/E1/P1, (b) XLPE/S15/E1/P1, (c) LDPE/S15/E1/P2 and (d) XLPE/S15/E1/P2.

particles.

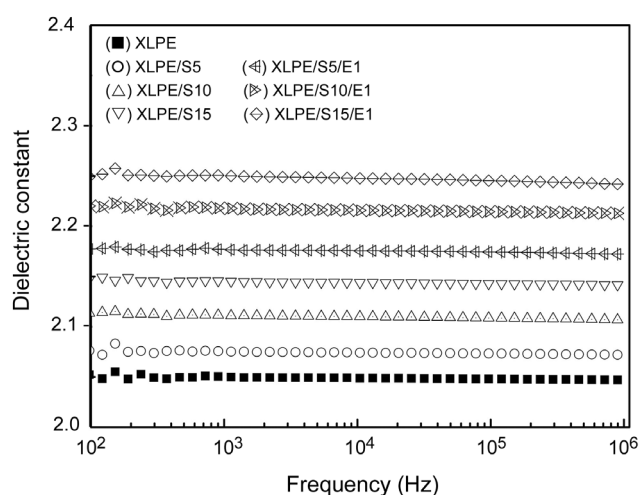
Figure 4 shows the SEM images of the etched blends with EVA. It can be found that: (i) the SEM images of the etched blends with EVA show the same morphological changes compared to that of the etched blends without EVA (ii) but, whether blends were cross-linked or not, the etched content of SEBS phase increased, indicating that EVA also has adverse effect on the compatibility between SEBS and LDPE, and EVA also can react with radicals generated from DCP decomposition. It can also be seen from Figure 4 that the XLPE/S15/E1 blends prepared by process 2 show a clear view, and the same blends prepared by process 1 contain some opaque areas.

### Dielectric Properties

Figure 5 shows the frequency dependency of dielectric constant of the blend samples obtained by different preparation methods. It can be seen from Figure 5 that all the samples have small frequency dependency of dielectric property, resulting in nearly the same value of dielectric constant in the total range of the measuring frequencies. The dielectric constant of the blends is increased with increase in SEBS content, and it may be explained that SEBS may contain a small amount of polar small-molecular additives, and the introduction of EVA will further increase the dielectric constant of XLPE/SEBS blends. The preparation by process 2 made SEBS



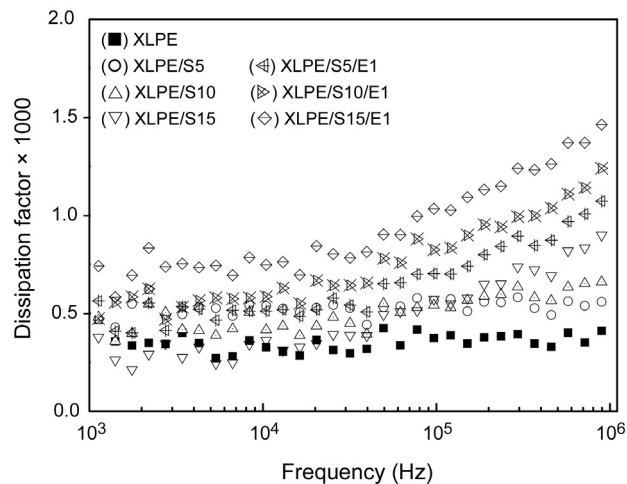
(a)



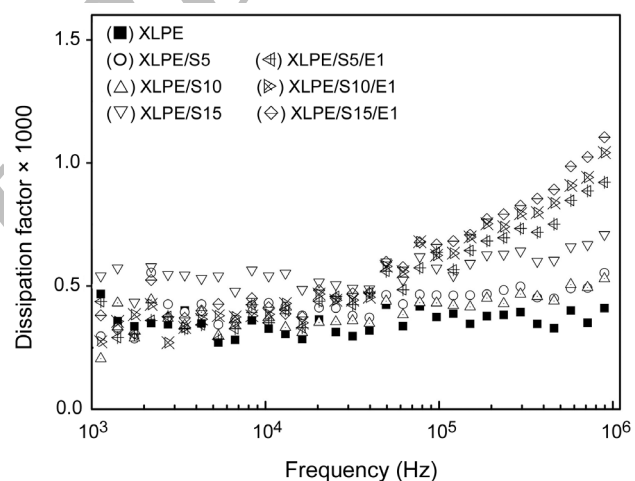
(b)

**Figure 5.** Frequency dependence of dielectric constant for the blend samples prepared by process 1 (a) and process 2 (b).

disperse more uniformly in LDPE matrix and enhanced the cross-linking of LDPE, while restraining the mobility of polyethylene molecular chains and lowering the polarized rates of XLPE, and as a result the dielectric constant of samples prepared by process 2 would be lower [20]. Figure 6 shows the frequency dependency of dielectric loss tangent of the blend samples obtained by different preparation methods. We can see that the XLPE/SEBS blends have small frequency dependency of dielectric loss, whereas the dielectric loss of XLPE/SEBS/EVA blends increase as the frequency increases. It is also noticeable from Figure 6 that, with the increase of



(a)



(b)

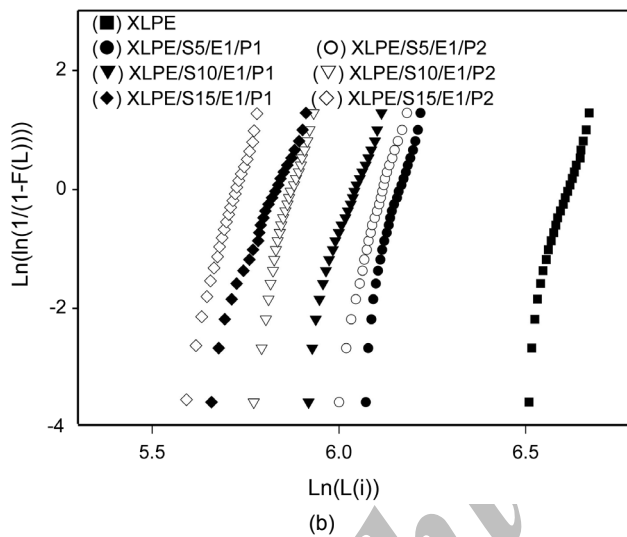
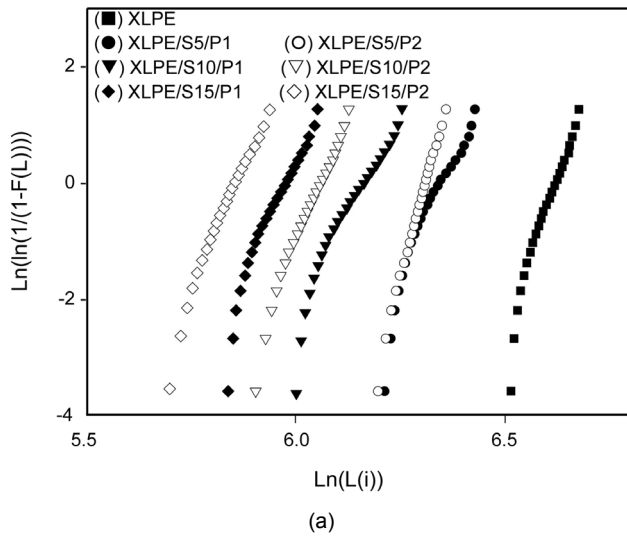
**Figure 6.** Frequency dependence of dielectric loss tangent for the blend samples prepared by process 1 (a) and process 2 (b).

SEBS content, the dissipation factor of several blends show similar trends to dielectric constant irrespective of which preparation method may be used, and SEBS disperses more uniformly in XLPE matrix resulting in relatively lower value of the dissipation factor.

The above results revealed that the blend samples still have the typical characteristics of dielectric behaviour for pure XLPE.

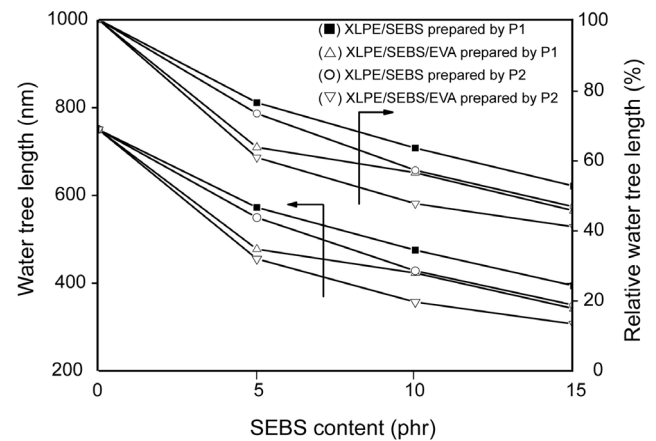
### Water Treeing Test

The water tree length (WTL) distributions were analyzed by using the two-parameter Weibull distribution, and the experimental data are shown in



**Figure 7.** The experimental data of water tree length of the blend samples prepared by different processes: (a) XLPE/SEBS blends and (b) XLPE/SEBS/EVA blends.

Figure 7, the  $\text{Ln}(\text{Ln}(1/(1-F(L))))$  was set as Y axis and  $L_t$  was set as X axis, according to these experimental data, the linear function curve was fitted, but, for the sake of convenience, the characteristic water tree length when the cumulative probability of water tree length reaches 69.03% it is used to evaluate the water treeing results. Therefore, the water tree resistance of the XLPE/SEBS and XLPE/SEBS/EVA blends prepared by different methods was evaluated and compared by the water tree length and relative water tree length as shown in Figure 8, which shows that: (i) the water tree length of the XLPE/SEBS blends decreases with the increased



**Figure 8.** Water tree length and relative water tree length of the blend samples prepared by different processes.

SEBS content, the relative water tree length will come to 52.7% when the SEBS content is 15 phr; (ii) SEBS and EVA can synergistically resist the water tree growth of XLPE, and the relative water tree length of SEBS/XLPE/EVA blend samples shows lower values when compared with the SEBS/XLPE blend samples having reached 45.7%; (iii) the sample preparation processes show clear influence on the relative water tree length of the blends samples, and the water tree length of samples prepared by process 2 will further decrease whether EVA is introduced or not, the relative water tree length will reach 46.8% and 41.1% for XLPE/SEBS and XLPE/SEBS/EVA blends, respectively. These results show that the addition of SEBS and EVA can synergistically improve the water tree resistance of XLPE and the sample preparation processes also have an important influence on water tree resistance of XLPE.

Water treeing in polyethylene is a complex phenomenon involving several processes with many factors, which are different according to different water tree mechanisms.

First, the water tree resistance of the XLPE/SEBS and XLPE/SEBS/EVA blends should be associated with the decrease in crystallinity [11]. Water tree usually grows from defects in amorphous regions. For the same amount of charge injected into the insulating system under the same electrical stress, materials with lower crystallinity are expected to have a lower local conductivity and would not be easy to reach the minimum value of electrochemical potential for



degradation compared to that with higher crystallinity. Therefore, the blend samples with lower crystallinity show shorter water tree length due to a low charge concentration. SEBS and EVA would decrease the crystallinity of XLPE, and more dispersivity of SEBS in XLPE matrix will further reduce the crystallinity of XLPE, thus SEBS and EVA can decrease the crystallinity of XLPE and the water tree resistance of conventional XLPE can be improved. The process 2 improves the dispersivity of SEBS in XLPE matrix, while the crystallinity of the blends is lowered, and therefore, process 2 also could improve the water tree resistance of XLPE from the viewpoint of crystallinity. Besides improving the anti-stress-crazing performance and crystallization behaviour of XLPE, SEBS is used as a voltage stabilizer. The ferrocene and its derivative also have a limited aromaticity and can be considered as effective voltage stabilizers to resist the growth of water tree, the electric field strength is the main factor influencing water tree growth [21]. The side chain of SEBS is phenyl and contains aromaticity to a certain extent with ionizing potential lower than that of polyethylene, therefore, it can also absorb high energy electrons to form stable particles, which make it possible to avoid the direct bombardment of the electrified particles and to prevent polyethylene from being broken down so as to resist the growth of water tree.

Another reason that the XLPE/SEBS blends have better water tree resistance than XLPE is that SEBS shows a stronger hydrophobicity when compared with XLPE. SEBS phase can prevent water molecules to penetrate into the samples, thus water tree in XLPE/SEBS blends cannot propagate as quickly as that in XLPE, and EVA with stronger hydrophobicity can absorb molecular water to prevent at sediments electric field enhancement points. Therefore, SEBS and EVA can synergistically improve water tree resistance of conventional XLPE. Another logical assumption is that SEBS has better water tree resistance than XLPE. The SEBS phase represents a randomly distributed set of points in XLPE matrix and stops the growth of water tree.

From the SEM of the blends, it is evident that the different preparation methods just lead to differences in dispersivity of SEBS in XLPE/SEBS and XLPE/SEBS/EVA blends. The blend samples that were

prepared by process 2 showed more uniform dispersion of SEBS phase, therefore, based on the above assumption that water trees propagate until they encounter a water tree retarding cluster, could be easily understood that the water tree resistance in XLPE/SEBS blends can be improved as the SEBS loading is increased.

## CONCLUSION

The XLPE/SEBS and XLPE/SEBS/EVA blends were prepared by two different processes and the water tree resistance of the samples was investigated.

The main findings from the water treeing experimental can be summarized as follows:

(i) SEBS shows a significant effect on improvement of the water tree resistance of XLPE. SEBS and EVA can synergistically improve the water tree resistance of XLPE and preparation method also has an important influence on water tree resistance property of XLPE.

(ii) SEBS and EVA have a small influence on the thermal properties of XLPE; while SEBS and EVA have a negative effect on the crystallinity of the blends and the preparation process 2 leads to a further decrease of crystallinity.

(iii) The morphology of the samples reveals that the difference between preparation processes changes the dispersion of SEBS in the matrix, but does not change the compatibility between SEBS and LDPE.

(iv) The XLPE/SEBS and XLPE/SEBS/EVA blends still possess excellent dielectric constant and dissipation factor as XLPE. The preparation process 2 that improved the dispersivity of SEBS has a positive effect on the dielectric properties.

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