

# Effect of Styrene–Ethylene–Butadiene–Styrene and Its Synergetic Effect with Ethylene Vinyl Acetate on the Mechanical, Thermal, Dielectric, and Water-Treeing Behaviors of Crosslinked Polyethylene

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**ABSTRACT:** In this study, we investigated the effect of an aromatic polymer, styrene–ethylene–butadiene–styrene (SEBS), on the water-tree resistance of crosslinked polyethylene (XLPE), and the synergetic effect of SEBS and ethylene vinyl acetate (EVA) was also investigated. The XLPE/SEBS and XLPE/SEBS/EVA samples were characterized by means of differential scanning calorimetry, scanning electron microscopy, mechanical measurements, and an accelerated water-treeing experiment, and the obtained results clearly show the relevant influence of SEBS and EVA, and as expected, the addition of SEBS and EVA was found to synergistically influence the water-tree resistance of XLPE more positively in comparison with

that without the addition of EVA. In addition, it also indicated that the blends possessed excellent dielectric behaviors, such as the dielectric constant and dissipation factor. The crystallization of the blends decreased with increasing SEBS content and addition of EVA. However, the melting temperature of the blends increased with the addition of SEBS and EVA, but the melting temperature of the blends decreased with increasing SEBS content. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 118: 2350–2357, 2010

**Key words:** blending; conjugated polymers; degradation; high performance polymers; polyethylene (PE)

## INTRODUCTION

Nowadays, crosslinked polyethylene (XLPE) has been widely used in underground transmission cables because of its excellent mechanical and dielectric properties. However, the electrical aging of insulation in power cables is inevitable, and water treeing has been known to be one of the main deterioration phenomena that finally cause the dielectric breakdown in XLPE power cables, especially in medium-voltage cables.<sup>1,2</sup>

Over the last 4 decades, much work has been devoted to understanding the initiation or growth mechanism of water treeing and the effect of test conditions, such as electric field strength, frequency, and temperature.<sup>2–6</sup>

To improve the service life of power cables, it is necessary to improve the water-tree resistance of insulating materials, and it could be improved by chemical and physical modification, such as blending, filling additives, copolymerization, and other

techniques.<sup>1,7–11</sup> The general method is to improve the hydrophilicity of XLPE by hydrophilic modification (acrylic acid) and ethylene vinyl acetate (EVA) or ethylene acrylic acid (EAA) blending.<sup>9,11–13</sup> The blending of XLPE with polar polymers has been widely used to create new water-tree-retardant insulating materials for power cables.<sup>14–16</sup> Although this method can improve the water-tree resistance, the polar polymers increase the dissipation factor and deteriorate other properties of the final materials. Therefore, nonpolar polymers with good compatibility with XLPE, such as polyolefin elastomers, have also been used to improve the water-tree resistance of XLPE.<sup>17</sup> Styrene–butadiene rubber was confirmed by Nagasaki to resist the growth of a bowtie tree.<sup>7</sup>

Lee et al.<sup>18</sup> used styrene–ethylene–butadiene–styrene (SEBS) to improve the compatibility of low-density polyethylene (LDPE)/polystyrene blends and to investigate the effect of SEBS on the electrical properties of LDPE/polystyrene blends.

In this study, the effect of SEBS on the water-tree resistance of XLPE was investigated, and the synergetic influence of SEBS and polar additives, such as EVA, was also considered on the basis of the different water-tree-resistance mechanisms of EVA and SEBS.

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## EXPERIMENTAL

### Materials

The LDPE (100bw) used in this study was purchased from Exxon Mobil Chemical Co. (Saudi Arabia) and had a melt flow index of 2.0 g·10/min and a density of 0.9225 g/cm<sup>3</sup>. SEBS (Kraton G1651H) was purchased from Shell Chemical Co., had a density of 0.91 g/cm<sup>3</sup>, and contained 31 wt % styrene. EVA (VA600) was supplied by Hyundai Petrochemical Corp. (South Korea) and had a melt flow index of 6.0 g·10/min and a density of 0.95 g/cm<sup>3</sup>. Dicumyl peroxide (DCP) was supplied by Shanghai Gaoqiao Petroleum Co., Ltd. (China) and had a purity higher than 99.5%. Sodium hydroxide (NaOH; analytical reagent) and sodium chloride (NaCl; analytical reagent) were provided by Shanghai Medicine Chemical Co., Ltd. (Shanghai, China).

### Sample preparation

Different ratios of LDPE, SEBS, and DCP were blended in a Haake rheometer RC90 (Shanghai Kechuang Machinery & Equipment Co. Ltd., Shanghai, China) at a temperature of 115°C with a rotor speed of 60 rpm for 10 min. All of the samples were first pressed at 120°C for 5 min and then pressed again at 175°C and 10 MPa for 15 min to obtain the XLPE/SEBS series, and the XLPE/SEBS/EVA series were obtained with the same process plus the addition of EVA during the blending process. For the sake of convenience, the samples were labeled XLPE-S<sub>x</sub>-E<sub>y</sub>, where S stands for SEBS and x is the content of added SEBS, E stands for EVA and y is the content of EVA; for example, XLPE-S15-E1 indicates XLPE with 15 phr SEBS and 1.0 phr EVA.

### Characterization

#### Gel content

The gel content was determined on thin films with a thickness of about 0.2 mm. Approximately 0.2 g of films were exactly weighed and then placed in a 120-mesh copper net. Then, they were extracted in a Soxhlet extractor (Shanghai Hongli Instrument Co., Ltd., Shanghai, China) for 36 h with *p*-xylene as an extractant. After extraction, the copper nets were dried in a vacuum oven at 80°C until a constant weight was reached, and the remaining gel was weighed and calculated as follows:

$$\text{Gel content (\%)} = W_t/W_0 \times 100$$

where  $W_0$  and  $W_t$  are the sample masses before and after extraction, respectively.

#### Differential scanning calorimetry (DSC)

A PerkinElmer Pyris 1 differential scanning calorimeter (PerkinElmer Instruments (Shanghai) Co. Ltd.,

Boston, USA) was used to take the DSC measurements in a flowing nitrogen atmosphere. Samples ranging from 3.0 to 4.0 mg were initially heated from 20 to 150°C at a rate of 20°C./min, held at 150°C for 3 min to eliminate thermal history effects, and then cooled to 20°C at a rate of 20°C./min. A value of 290 J/g was used as the melting enthalpy of 100% crystalline polyethylene to calculate crystallinity of polyethylene from the melting enthalpies.<sup>19</sup>

#### Mechanical measurement

The tensile tests of the 1 mm thick specimen were performed with an Instron series IX 4465 materials tester (Instron Corp., Shanghai Representative Office, Shanghai, China) according to ASTM D 638-2003 with a crosshead speed of 250 mm/min. Five specimens for each sample were measured, and the arithmetic mean of all values obtained was reported as the average value for the particular properties of each sample.

#### Scanning electron microscopy

The morphology of the fractured surfaces of the samples was observed with a Hitachi S520 scanning electron microscope (Hitachi Ltd., Tokyo, Japan). All samples were prepared by immersion of the test pieces in liquid nitrogen before breaking. Then, the fractured surfaces were etched with toluene to remove the dispersed SEBS phase and then coated with gold before examination.

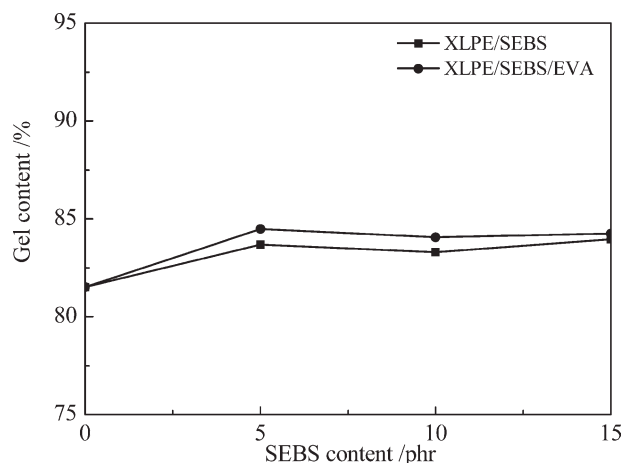
#### Dielectric properties

The dielectric constant and the dissipation factor were measured by means of a QS30 high-voltage bridge (Shanghai Huguang Corp., Shanghai, China) according to ASTM D 150-2004 (the thickness of specimens was 1.0 mm).

The dielectric strength was measured with an ac dielectric strength tester AHDZ-10/100 (Shanghai Lanpotronics Corp., Shanghai, China) according to ASTM D 149-2004. During testing, the applied voltage was raised step by step, and the voltage at each step was retained for 1 min. The voltage ratio between adjacent steps was 1.06. The initial test voltage was 10 kV, and the rate of rise of the test voltage was 120 kV/min. The value averaged from the values of six points measured was taken as the dielectric strength.

#### Water-treeing test

The water-treeing test was carried out according to ASTM D 6097-97. Each circular specimen with a diameter of 25.4 mm and a thickness of 6.35 mm



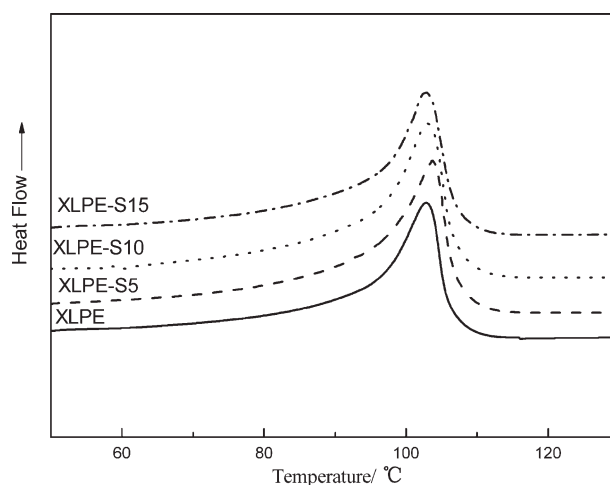
**Figure 1** Effect of the SEBS content on the gel content of the XLPE/SEBS and XLPE/SEBS/EVA blends.

contained a conical defect for the accelerated water-treeing test at the center of one side, and a conical defect with a diameter of 3.2 mm and a height of 3.2 mm had a tip angle of  $60^\circ$  and a cone-tip radius of  $3 \pm 1 \mu\text{m}$ . After 10 specimens were fixed in a poly(methyl methacrylate) specimen holder, the holder was filled with a 1.0N NaCl solution, and then, the test specimen holder was also placed in a bath containing a 0.01N NaCl solution. The water-treeing test was carried out at room temperature for 30 days under the action of the applied voltage of 5 kV and 1500 Hz. After the defined time, all samples were stained with methylene blue, and the water-tree length was determined by measurement of the length of the longest branch of the tree with polarized microscopy. The value averaged from the values of 10 measurements was taken as the water-tree length.

## RESULTS AND DISCUSSION

### Gel contents

The gel contents of the blend samples containing SEBS and SEBS/EVA were measured, and the results are shown in Figure 1. We found that (1) the blends showed higher values of gel content compared with the XLPE sample, (2) the gel content values of the blends were almost independent of the SEBS content, and (3) the introduction of EVA slightly increased the gel content of the blends. The results obtained might have been due to the enhancing crosslinking effect of SEBS and the formation of the cocrosslinked network of SEBS and LDPE because the C=C double bonds of the SEBS molecular chains could have reacted easily with the radicals created by DCP and, thus, take part in the crosslinking process of polyethylene. The  $-\text{CH}_2-\text{CH}_2-$  group of EVA could have also joined in the cross-



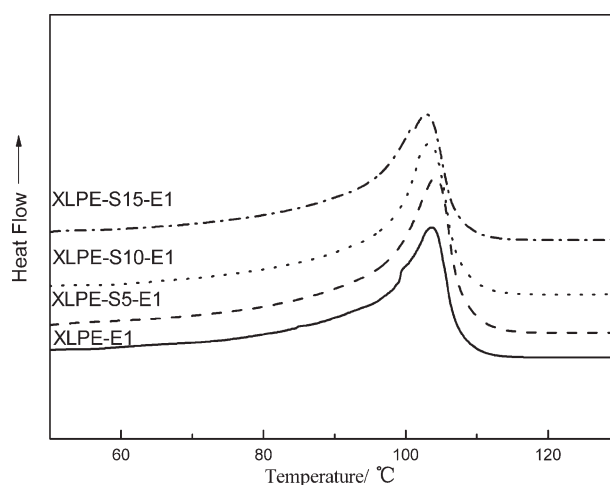
**Figure 2** Effect of the SEBS content on the melting curves of the XLPE/SEBS blends.

linking processes, and the slight increase in the gel content after the addition of EVA indicated that EVA had a higher crosslinking efficiency in comparison with polyethylene and could be attributed to the acetyl group on the copolymer, which stabilized a radical on the attached carbon by resonance.

### DSC characterization

Other studies noted that the temperature of the melting peak is correlated with the crystal perfection of polyethylene and the area of the melting peak is directly proportional to the crystallinity.<sup>20,21</sup> Therefore, the melting behaviors of the XLPE/SEBS and XLPE/SEBS/EVA blends were measured, and the melting curves are shown in Figures 2 and 3, respectively.

As shown in Figure 2, the melting peak temperature of the blends first increased with increasing



**Figure 3** Effect of the SEBS content on the melting curves of the XLPE/SEBS/EVA blends.

**TABLE I**  
Summary of the DSC Data for the XLPE/SEBS and XLPE/SEBS/EVA Blends

Sample	$T_m$ (°C)	$T_c$ (°C)	$\Delta H_m$ (J/g)	$X_m$ (%)
XLPE	102.94	87.99	77.05	26.7
XLPE-S5	103.69	86.81	68.97	23.8
XLPE-S10	103.07	88.22	72.74	25.1
XLPE-S15	102.87	87.39	71.35	24.6
XLPE-E1	103.81	87.12	66.01	22.8
XLPE-S5-E1	104.13	86.83	66.44	22.9
XLPE-S10-E1	103.49	87.58	67.05	23.1
XLPE-S15-E1	103.01	87.35	64.14	22.1

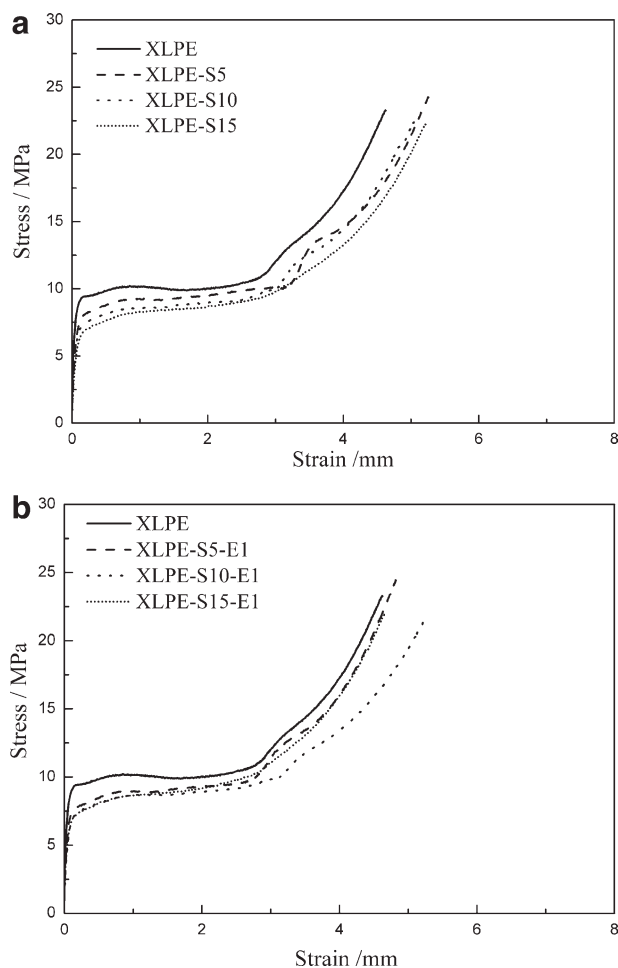
$\Delta H_m$  = enthalpy of melting;  $T_c$  = crystallization temperature;  $T_m$  = melting temperature;  $X_m$  = degree of crystallinity.

SEBS and then decreased again with further increasing SEBS; this indicated that a small amount of SEBS did not prevent the lamellar thickening of polyethylene during crystallization; however, part of the radicals in the blends were consumed by SEBS during the crosslinking process, and fewer radicals approached the polyethylene molecular chains, which, in turn, led to the formation of more perfect lamellas. Thus, the melting temperature slightly shifted to a higher temperature.<sup>1</sup> With further increasing SEBS content, the crosslinking reaction between SEBS and polyethylene began to influence the crystallization of the polyethylene, which resulted in a decrease of the melting temperature. The addition of EVA further consumed radicals during the crosslinking processes; therefore, the XLPE/SEBS/EVA blends showed higher melting peak temperatures compared with the XLPE/SEBS blends (Table I).

The crystallinity of blends is related to the water-tree resistance of the materials; the thermal data of the XLPE/SEBS and XLPE/SEBS/EVA blends are listed in Table I. The crystallinity of the blends gradually decreased with increasing SEBS content, which was consistent with the increase in the gel content shown in Figure 1. With the addition of EVA, the crosslinked degree further increased, and thus, the crystallinity of the blends decreased. According to Figure 1, the blends only showed a slight increase in gel content; therefore, it was reasonable that the crystallinity of the blends decreased a little.

### Mechanical measurements

The mechanical behaviors of the XLPE/SEBS and XLPE/SEBS/EVA blends contents are worth discussing because they are one of the most important properties for the choosing of polymer materials. Figure 4(a,b) shows the stress-strain curves of the XLPE/SEBS and XLPE/SEBS/EVA blends, respectively. Furthermore, the tensile strength and elonga-

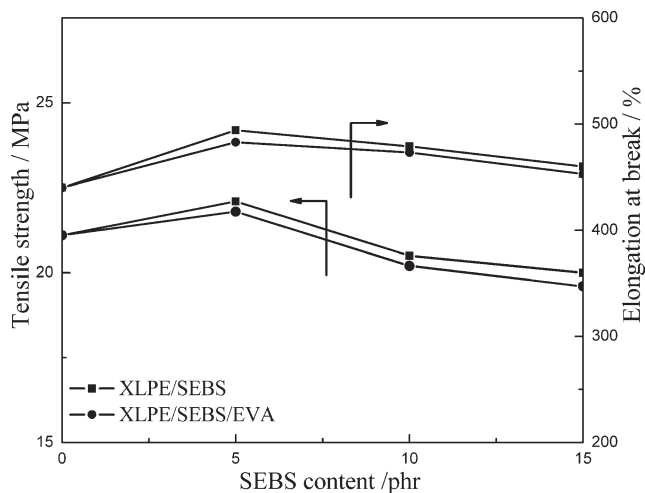


**Figure 4** Tensile stress-strain curves of (a) XLPE/SEBS and (b) XLPE/SEBS/EVA blends.

tion at break of the samples are also presented in Figure 5.

As shown in Figure 4, the broken points of the XLPE/SEBS and XLPE/SEBS/EVA blends shifted to higher strains with increasing SEBS content, and the strain degree became larger. The stress when the specimen was broken first increased and then decreased again. This may have been due to the enhancing crosslinking of polyethylene and the elastomeric properties of SEBS. EVA could also enhance the crosslinking of polyethylene, and the broken stress of the XLPE/SEBS/EVA blends was lower than that of the XLPE/SEBS blends. We concluded from the stress-strain curves that the antistress crazing was slightly improved with the addition of SEBS and EVA, and we also considered it as a possible factor for improving the water-tree resistance of XLPE.

As shown in Figure 5, the tensile strength of XLPE/SEBS blends first increased and then decreased again with increasing SEBS content. When the SEBS content was 5.0 phr, the tensile strength reached the maximum; this may have been due to the enhanced crosslinking of polyethylene, but when



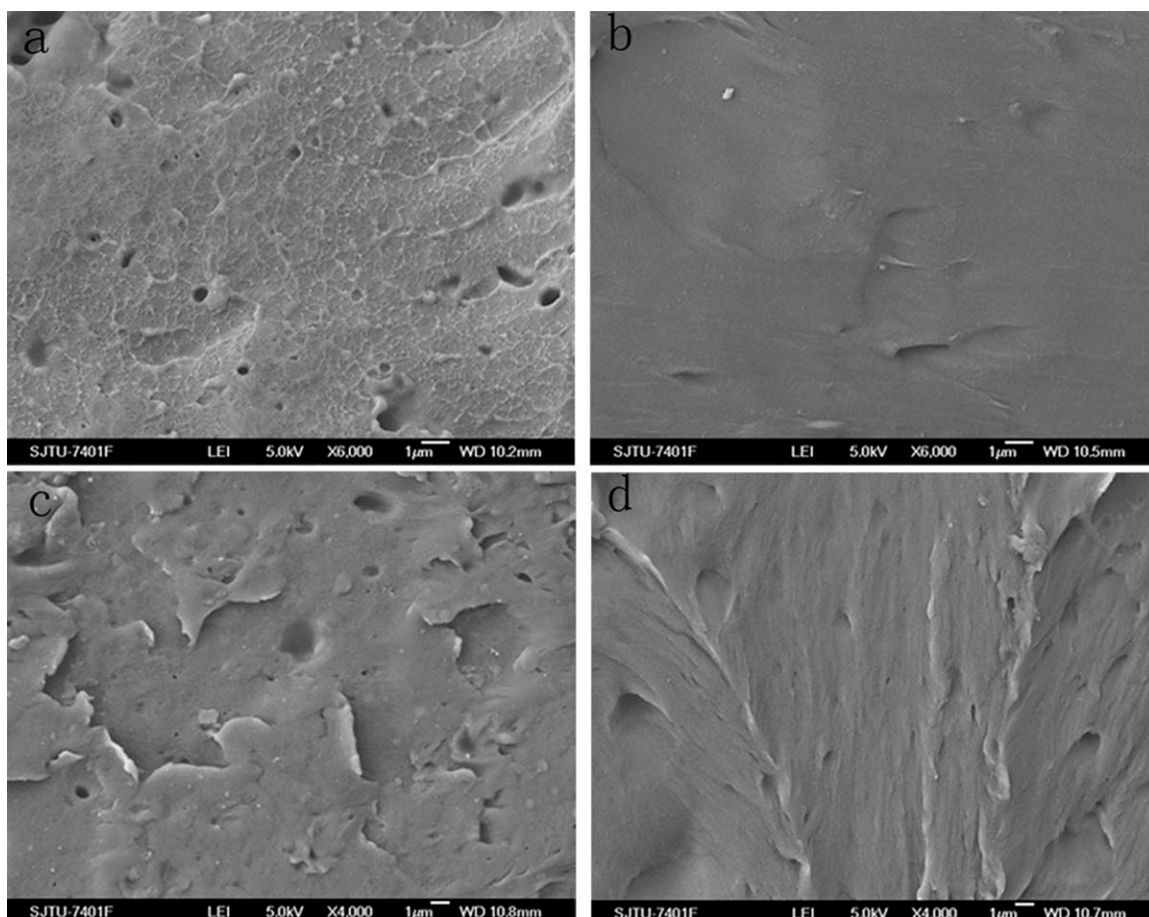
**Figure 5** Effect of the SEBS content on the mechanical properties of the XLPE/SEBS and XLPE/SEBS/EVA blends.

its content exceeded 5.0 phr, SEBS led to a decrease in the tensile strength in contrast, possibly because of the relatively low tensile strength of SEBS. The variation rules of the elongation at break of the

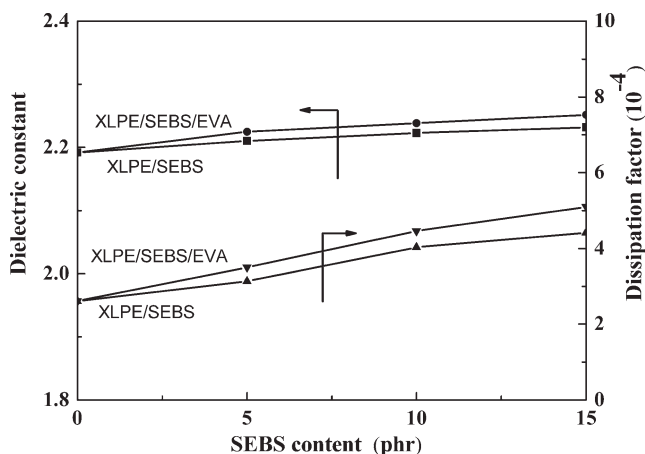
blends were similar to that of the tensile strength. Although the mechanical performance of blends decreased when the SEBS content exceeded 5.0 phr, the tensile strength and elongation at break of the blend still came to 20 MPa and 459%, respectively, when the SEBS content was 15.0 phr and satisfied the mechanical performance of cable insulation. For the XLPE/SEBS/EVA blends, the tensile strength and elongation at break had a similar variation rule to the XLPE/SEBS blends, but because of the further enhancing crosslinking effect of EVA, the values of tensile strength and elongation at break of the XLPE/SEBS/EVA blends were lower than those of the XLPE/SEBS blends. As stated previously, SEBS had little influence on the tensile properties of XLPE, and when the addition amount of EVA was small, the XLPE/SEBS and XLPE/SEBS/EVA blends still possessed excellent mechanical performances.

### Morphology

Figure 6 depicts the morphology of the LDPE/SEBS, LDPE/SEBS/EVA, XLPE/SEBS, and XLPE/SEBS/EVA blends, respectively. As shown, (1) the SEBS



**Figure 6** Scanning electron microscopy images of samples with different DCP and SEBS contents: (a) LDPE-S15, (b) XLPE-S15, (c) LDPE-S15-E1, and (d) LDPE-S15-E1.



**Figure 7** Effect of the SEBS content on the dissipation factor and dielectric constant of the XLPE/SEBS and XLPE/SEBS/EVA blends.

phase in the LDPE blends was chemically etched by toluene, and the uncrosslinked blends displayed a sea-island morphology [Fig. 6(a)]; (2) after the crosslinking reaction, a two-phase morphology could not be found in the blends; this indicated that SEBS could not be etched out after crosslinking with LDPE [Fig. 6(b)]; and (3) after comparing Figure 6(c,d) to Figure 6(a,b), respectively, we observed that the volume of the dispersed phase increased after the addition of EVA. The morphological change revealed that SEBS easily reacted with radicals generated from DCP and was cocrosslinked with polyethylene. Although the addition of EVA had a negative effect on the compatibility between SEBS and PE, after it was crosslinked, SEBS was hardly etched out, and the phase structures hardly showed any great change.

### Dielectric properties

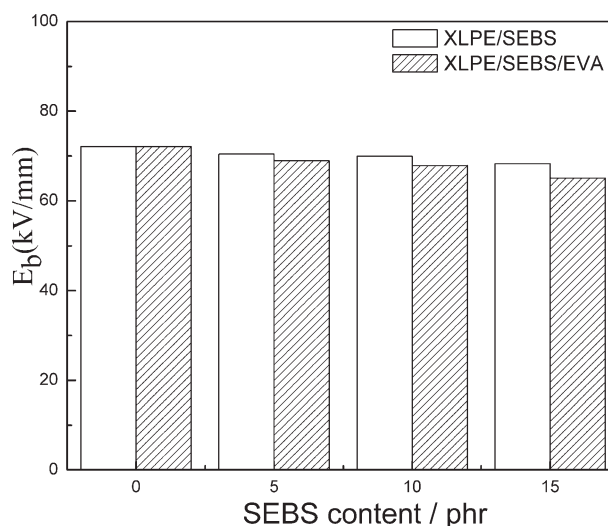
For cable insulation, the dielectric properties of materials are another important decisive factor. The effects of the SEBS content on the dielectric constant and dissipation factor of the XLPE/SEBS and XLPE/SEBS/EVA blends were examined, and the results are exhibited in Figure 7. As shown in Figure 7, the dissipation factor of the XLPE/SEBS blends increased slowly, possibly because the SEBS may have contained a small amount of polar small-molecule additives, but SEBS enhanced the crosslinking of polyethylene and restrained the mobility of the polyethylene molecular chain and lowered the polarized rates of XLPE. Thus, the dissipation factor just had a slight increase. However, the dielectric constant first decreased and then increased again with increasing SEBS content. This may also have been due to the integrative results of crosslinking enhancement and polar small-molecule additives.

For the XLPE/SEBS/EVA blends, the dissipation factor and dielectric constant of the XLPE/SEBS/EVA blends also had similar variation trends, but the values were obviously higher than that of the XLPE/SEBS blends; because of that, the addition of a polar copolymer accelerated the tropistic polarization and introduced new dipolar dissipation, which thus led to increases in the dissipation factor and dielectric constant.<sup>22</sup> It was shown clearly by the results of the dissipation factor and dielectric constant that the XLPE/SEBS and XLPE/SEBS/EVA blends still possessed excellent dielectric properties.

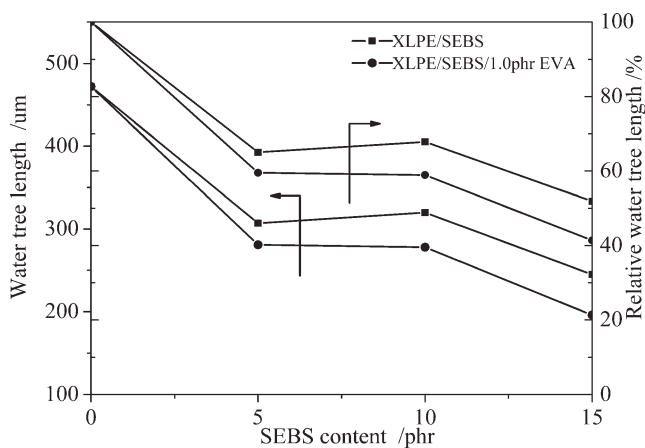
Figure 8 depicts the relationship between the SEBS content and the dielectric strength of the XLPE/SEBS and XLPE/SEBS/EVA blends. The dielectric strength of the XLPE/SEBS blends decreased with increasing SEBS content, which may have been due to the enhancement of the crosslinked degree and the decrease in crystallinity, and the dielectric strength decreased gradually.<sup>23</sup> The introduction of the polar copolymer (EVA) further decreased the dielectric strength of XLPE, but because the added amount of EVA was very small, the influence was very small. As shown by the results of dielectric strength, the XLPE/SEBS and XLPE/SEBS/EVA blends still possessed excellent insulation performance.

### Water-treeing test

The water-tree resistance of the XLPE/SEBS and XLPE/SEBS/EVA blends was evaluated by the water-tree length and relative water-tree length, as shown in Figure 9, which shows that (1) the water-tree length of the XLPE/SEBS blends decreased with increasing SEBS content, and the relative water-tree



**Figure 8** Characteristic dielectric strength ( $E_b$ ) for XLPE/SEBS and XLPE/SEBS/EVA blends with different SEBS contents.



**Figure 9** Effect of the SEBS content on the water-tree length and the relative water-tree length of the XLPE/SEBS and XLPE/SEBS/EVA blends.

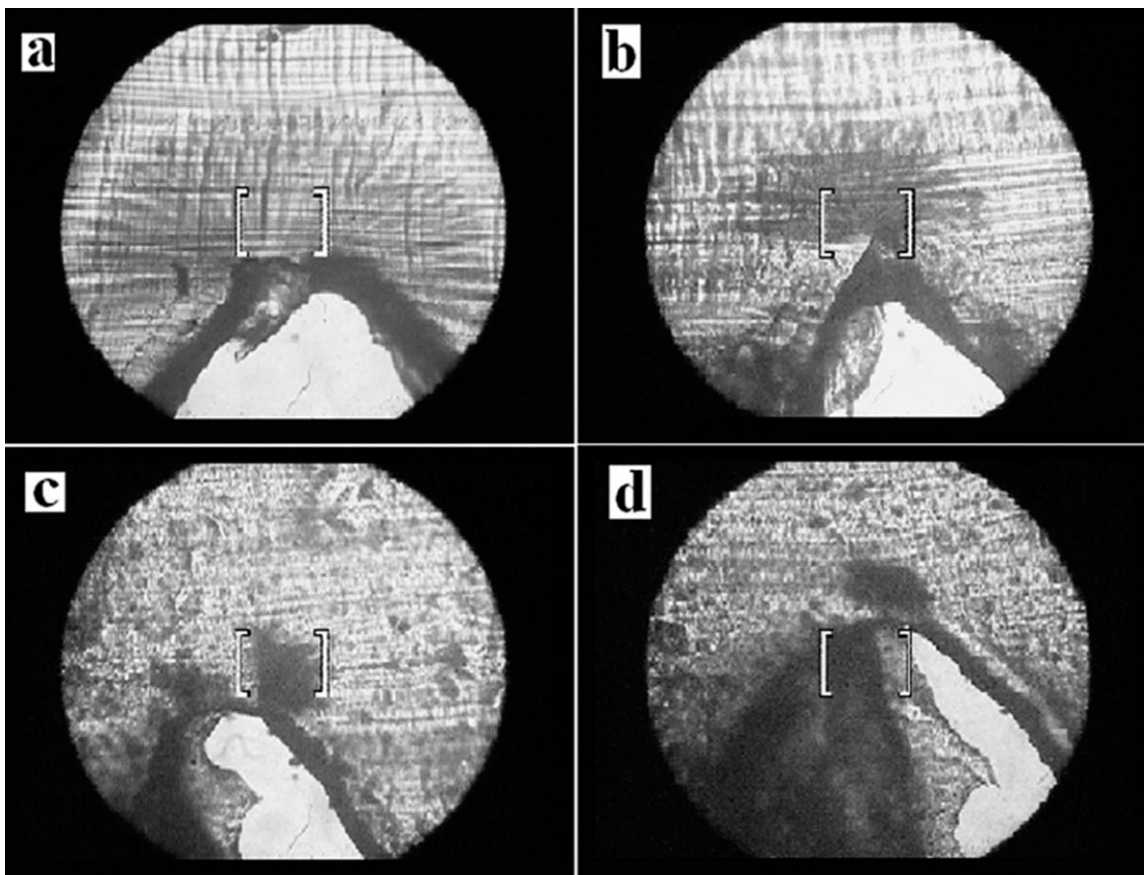
length came to 52% when the SEBS content was 15.0 phr, and (2) SEBS and EVA synergistically resisted the water-tree growth of XLPE, and the relative water-tree length of the SEBS/XLPE/EVA blend samples showed lower values when compared with those of the SEBS/XLPE blend samples and came to 41.5%. Figure 10 shows the photographs of typical

water trees in the XLPE/SEBS and XLPE/SEBS/EVA samples.

At first, the water-tree resistance was related to the crystallinity of the blends in general; water trees usually initiate at the defects in amorphous regions, and the introduction of SEBS and EVA decreased the crystallinity of XLPE. The charge density at each defect decreased, the water-tree growth rate decreased, and thus, the water-tree length became shorter, although this effect was too small to be considered a main factor. However, from the viewpoint of crystallinity, the water-tree resistance was synergistically improved by SEBS and EVA.

Second, as shown in the previously mentioned stress-strain curves of the blends, we concluded that the antistress crazing was slightly improved with the addition of SEBS and EVA, and this was considered a possible factor in the improvement of the water-tree resistance of XLPE.

Similar to the electric-tree-resistant mechanism of voltage stabilizers,<sup>24</sup> ferrocene and its derivatives have aromaticity, and their ionizing potentials were lower than that of polyethylene. Therefore, high-energy electrons were absorbed before it reacted with polyethylene, which made it possible to avoid



**Figure 10** Photographs of water trees in the XLPE/SEBS and XLPE/SEBS/EVA blends: (a) XLPE, (b) XLPE-E1, (c) XLPE-S15, and (d) XLPE-S15-E1.

the direct bombardment of the electrified particles and to prevent polyethylene from being broken down so as to resist the growth of the electric tree.

Therefore, with reference to the electrochemical reaction and electric-tree-resistant mechanism, SEBS could be used as a voltage stabilizer. The side chain of SEBS was phenyl and contained aromaticity to a certain extent and whose ionizing potential was lower than that of polyethylene, and it could also absorb high-energy electrons to form stable particles and prevent the direct bombardment of the electrified particles and increase the electric field strength of water-tree initiation. Therefore, water-tree growth was depressed, which may be considered as the main mechanism of resisting the growth of water trees by SEBS. On the other hand, the SEBS phase could be considered a randomly distributed set of points in the XLPE matrix and prevented the continuous penetration of water molecules into the electric enhancement points. On the basis of this assumption that water trees propagate until they encounter a water-retardant cluster, water molecules could not penetrate into the electric enhancement points, and the growth speed of the water trees slowed down. From the two viewpoints of the previously discussed electrochemical reaction and water penetration mechanism, the water-tree resistance in the XLPE/SEBS blends was improved as the SEBS content increased.

The tree-resistance mechanism of polar copolymers such as EVA is well known, and it can prevent the water molecules from diffusing into the insulation and, thereby, prohibit the condensation of water in electrically irregular points. Therefore, SEBS and EVA may have had a synergistic effect on water-tree growth.

### CONCLUSIONS

The water-tree resistance of the XLPE/SEBS and XLPE/SEBS/EVA blends were investigated along with the physicochemical and dielectric properties.

The main findings from the water-treeing experiments are summarized as follows:

1. SEBS showed a significant effect on the improvement of the water-tree resistance of the cable insulation, and SEBS and EVA synergistically improved the water-tree resistance of XLPE.
2. SEBS and EVA had a negative effect on the crystallinity of the blends, but the influence was small.
3. The morphological observation revealed that SEBS was partially compatible with PE, and

the addition of EVA deteriorated the compatibility of SEBS with PE, but the influence was small, especially after blends were crosslinked.

4. From the results of the mechanical properties and dielectric properties of the blends, the XLPE/SEBS and XLPE/SEBS/EVA blends still possessed excellent mechanical and dielectric properties.

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