Effect of Silane-Grafting on Water Tree Resistance of XLPE Cable Insulation

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ABSTRACT: Water treeing is one of the main deterioration phenomena observed in the polymeric insulation of extruded crosslinked polyethylene (XLPE) cables, which can affect the service life of power cables. In this work, we investigated the effect of grafting of a silane (vinyl trimethoxysilane, VTMS) on the resistance of XLPE to water treeing. A series of water-treeing tests, the mechanical and dielectric measurements indicated that the silane-grafting could significantly improve the water tree resistance of the conventional XLPE cable insulation with little influences on its dielectric properties, e.g., the dielectric breakdown strength, dielectric constant and loss tangent, and its mechanical performance. It was found that there exists an optimum value of VTMS concentration (about 0.6 phr) corresponding to the minimum water tree length. The water tree resistance mechanism of silane-grafted XLPE was proposed on the basis of the process of silane hydrolysis and crosslinking. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 115: 3168–3176, 2010

Key words: crosslinked polyethylene; water treeing; vinyl trimethoxysilane; grafting; silane-crosslinking; cable insulation

INTRODUCTION

At present, crosslinked polyethylene (XLPE) is known as the most widely-used insulating material for extruded cable insulation, which has been largely replaced with paper/oil insulation and low-density polyethylene (LDPE) in medium and high-voltage cables, because of the well-known advantages such as adequate mechanical properties at high temperatures, good dielectric properties, easy manufacturing, and low cost.[1–4]

Water treeing has been known as one of the main deterioration phenomena affecting the service lifetime of the underground XLPE power cables. For underground cables, the water in the soil can slowly diffuse through the protective layers of the cable into the insulation under the action of high voltage across the cable insulation, forming tree-like structures (water tree), which are actually water-filled microcavities.[2]

Much effort has gone into attempting to clarify the water treeing phenomena and to minimize their influence in polymer-insulated power cables[5–7] since the first discovery of water trees in underground cables in 1968.[8] Up to now, many new speculations and theories have been brought forward for explaining the water treeing deterioration[6–11] and a number of studies have been carried out for developing new approaches to improve the resistance of XLPE to water-treeing.

Some researchers suggested that grafting hydrophilic groups onto polyethylene backbone chains,[12] or blending XLPE with polar polymers,[13,14] e.g., ethylene vinyl acetate (EVA), ethylene-acrylic acid (EAA), could improve the resistance of XLPE to water-treeing. Other researchers used low molecular weigh organic species as water tree retardant additive.[15] Our research group developed an effective method for increasing water tree resistance of XLPE by using non-polar polymers which are compatible with polyethylene,[16] e.g., polyolefin elastomer (POE).

There exist three methods for preparing XLPE: peroxide-, silane-, and irradiation-crosslinking.

Peroxide crosslinking involves the blend of polyethylene with peroxide during an extrusion process and the formation of free radicals which crosslink the polymer chains to form network structure during the subsequent thermal decomposition.[17,18] Silane crosslinking involves the grafting of vinyl silanes onto polyethylene chains with the help of small quantities of peroxides as radical initiators followed by a silane hydrolysis and silanol condensation reacton.[19–21] Silane crosslinking requires the

presence of water which diffuse into the polymer. The loss of water can reduce the possibility of forming water trees. On the other hand, the silanol groups formed during the process of silane hydrolysis can reduce the condensation of water.[19] Therefore, it is of great interest that the well-known silane-crosslinking mechanism may be effectively introduced into the development of a new approach for improving the water tree resistance of XLPE cable insulation.

This study aims to investigate the possibility of improving the water tree resistance of XLPE by using silane-grafting onto polyethylene chains.

EXPERIMENTAL

Materials

The polyethylene used in this study is LDPE (100 bw) from ExxonMobil in Saudi Arabia, with a melt flow index (MFI) of 2.0 g/10 min and a density of 0.9225 g/cm^3. Vinyl trimethoxysilane (VTMS Z-6300) was supplied by Dow Corning (Midland, MI). The dicumyl peroxide (DCP) is supplied from Shanghai Gaoqiao Petroleum, China, with the purity higher than 99.5%. Catalyst used here was dibutyltin dilaurate (DBTL), purchased from Atofina. NaOH and NaCl are from Shanghai Medicine Chemical, China.

Sample preparation

After different ratios of LDPE, VTMS and DCP were blended in a HAAKE Rheometer RC90 with the rotor speed of 60 rpm for 2 min at the temperature of 110°C, DBTL was added into Rheometer and then blended for 8 min so as to finish the sample preparation. Any antioxidant was not used in this work. All samples were pressed at 170°C and 10 MPa for 15 min. For the sake of convenience, the samples were denoted using the following notation: XLPE-silane content, thus XLPE-0.2 indicates XLPE with 0.2 phr silane.

Characterization

Solid-state nuclear magnetic resonance

^{13}C solid-state nuclear magnetic resonance (NMR) measurements were performed on a Bruker AVANCE III system (400 MHz) at room temperature. About 1000 mg of powder was used for measurements. Prior to the NMR measurement, the samples were not purified.

Infrared transmission spectra analysis

A Paragon 1000 Fourier transform infrared (FTIR) spectrometer from Perkin Elmer (Boston, MA) was used to perform Infrared transmission spectra analyses on the samples. The thickness of the samples is about 100 μm. Before FTIR measurements, the films were purified to remove non-grafted VTMS or silane oligomers, the films were immersed in acetone solution for 2 days and then were dried in vacuum atmosphere at 80°C for 8 h.

Gel content

The gel content was determined on thin films with thickness of about 0.2 mm. During the heat press process, radicals produced by peroxides were partly used to initiate the grafting reaction of silane, besides silane-grafting, peroxide-crosslinked structure formed. To make sure whether or not silane crosslinking had occurred during the accelerated aging (the water treeing test), we compared the gel content of samples before and after the hydrolyzed condensation. The speed of silane crosslinking is very slow even under the action of catalyst (DBTL) because accelerated aging is carried out at room temperature.[22] Gel contents were measured after all the samples were immersed in 1.0N sodium chloride solutions for 15 and 30 days, respectively. About 0.2 g of the films before and after silane-condensated crosslinking were exactly weighed and then placed in a 120-mesh copper net, respectively. Then, they were extracted in a Soxhlet extractor for 36 h using p-xylene as extractant. After extraction, the copper nets containing samples were dried in a vacuum oven at 80°C. Finally, 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 mass values of samples before and after extraction, respectively.

Differential scanning calorimetry

A Perkin Elmer Pyris 1 DSC Calorimeter was used to perform differential scanning calorimetry analysis in a flowing nitrogen atmosphere. Samples of 3.0 mg were initially heated from 20 to 150°C at the heat rate of 20°C/min, held at 150°C for 3 min to eliminate thermal history effects, and then cooled down to 20°C at the rate of 20°C/min.

Water absorption measurement

The measurement of water uptake was carried out by the immersion/gain method.[23] First, a sample was dried for 36 h at 90°C in the dry oven, and its original weight (M_0) was recorded. Then, the sample was immersed into 1.0N NaCl solution at the

temperature of 30°C. The weight of wet sample (M_t) was measured after removing the water on the surfaces by blotting with the filter paper at different times until absorption equilibrium was reached.

The water absorption ratio (WAR) can be calculated as follows:

$$WAR(\%) = (M_t - M_0)/M_0 \times 100$$

Mechanical properties

According to ASTM D 638–2003, the tensile tests were performed using an CMT 5305 materials tester (SANS, Shenzhen, China) with a crosshead speed of 250 mm/min. Dumbbell shaped specimens with cross-sectional area of 4 × 1 mm and gage length of 20.0 mm were used for the measurements. Five specimens for each sample were used and the arithmetic mean of all values obtained was taken as the average value representing the mechanical characteristics of each sample. In addition, the standard deviation was calculated according to ASTM D 638–2003.

Dielectric properties

The dielectric constant and the dissipation factor were measured by means of QS30 high-voltage Bridge (Shanghai Huguang, Shanghai, China) according to ASTM D150–2004 (the thickness of specimens is about 1.0 mm).

The dielectric strength was measured using an AC dielectric strength tester AHDZ-10/100 (Shanghai Lanpotronics, Shanghai, China) according to ASTM D 149–2004. During the testing, the applied voltage was raised step by step, and the voltage at each step was kept for 1 min. The voltage ratio between adjacent steps is 1.06. The initial test voltage was 10 kV and the rate-of-rise of the test voltage was 120 kv/min.

Water-treeing test

The circular specimens for water-treeing test were prepared based on the method and molding apparatus according to ASTM D6097–97. Each circular specimen with the diameter of 25.4 mm and the thickness of 6.35 mm contains a conical defect for the accelerated water-treeing test at the center of one side, and the conical defect with the diameter of 3.2 mm and the height of 3.2 mm has the tip angle of 60° and the cone-tip radius of 3 ± 1 μm. After 10 specimens of the same kind were fixed in PMMA specimen holder, the holder was filled with 1.0N NaCl solution, and then, the test specimen holder was also placed in the bath containing 0.01N NaCl solution. Water-treeing test was carried out at the

room temperature for 30 days under the action of the applied voltage of 5 kV and 1500 Hz. The high voltage and the zero potential (earth-ground) are applied to the NaCl solutions inside and outside the specimen holder, respectively. After the accelerated water-treeing test was performed for a defined time, all samples were stained with methylene blue, and the water tree length was determined by measuring the length of the longest branch of the tree using the polarized microscopy. The value averaged from the values of 10 measurements was taken as the water tree length (WTL).

RESULTS AND DISCUSSION

NMR and FTIR characterization

For investigating the grafting reaction, ^{13}C solid-state nuclear magnetic resonance was used to qualitatively determine the total branch points. Figure 1 presented the ^{13}C-NMR spectra obtained for XLPE and VTMS-grafted XLPE. Branch content was determined by numerical peak integration of branching signals. The results revealed that the number of the total branch points for VTMS-grafted XLPE is clearly larger that of XLPE, indicating that the silane is grafted onto the polyethylene chain backbones.

To characterize the occurrence of the silane-condensation reaction, the XLPE samples containing 1.0 phr VTMS were immersed into 1.0N NaCl solution for 20 days and then FTIR measurement was performed (Fig. 2). For comparison, the FTIR measurement was carried out for the samples before immersion. According to the FTIR spectra measurements (Fig. 2), two absorption peaks centered at about 1150 and 1050 cm^{-1} can be found after the silane-condensation reaction, which corresponds to the Si—O—Si crosslinked bonds.
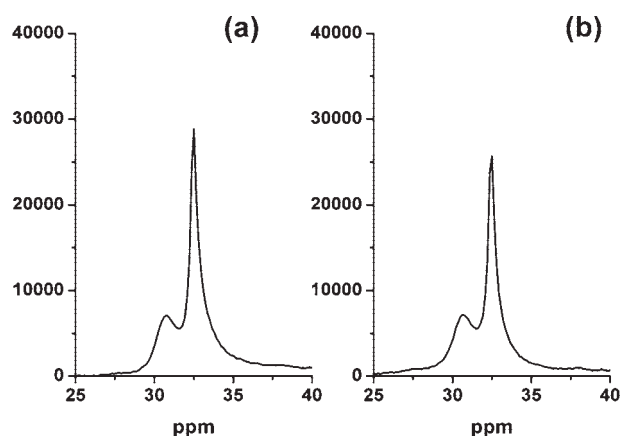

Figure 1 ^{13}C spectra of XLPE (a) and 5.0% VTMS-grafted XLPE (b).
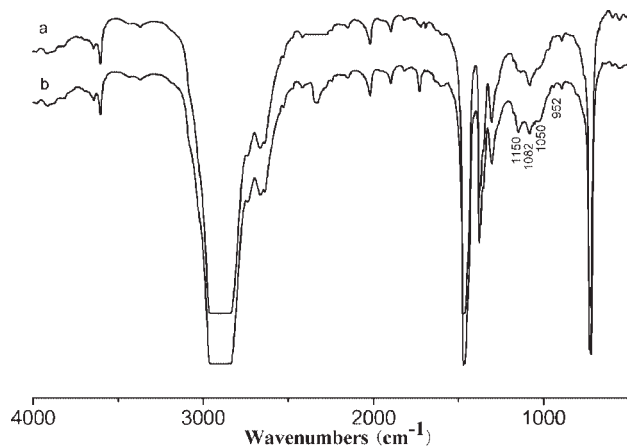
Figure 2 FTIR spectra of the silane-grafted XLPE samples (a) before and (b) after the silane-condensation reaction.

Gel content

To investigate the influence of the VTMS content on the degree of peroxide crosslinking of LDPE and on the silanol condensation reaction of the silane-grafted XLPE, the gel content was measured for the samples containing different VTMS before and after the silanol condensation reaction (for 15 and 30 days, respectively).

As can be seen in Figure 3, the gel content of the silane-grafted XLPE samples show high values after the hydrolysis and condensation reactions, which is likely attributed to the silane-crosslinking reaction with the help of water. The results of gel content measurement before and after hydrolyzed condensation strongly indicate that the hydrolyzed crosslinked reaction surely occur during water-treeing process.

As we can see from Figure 3, the gel content obtained in this work was quite higher than 65–75% gel results of industrialized crosslinked polyethylene. The reasons might be ascribed to the difference between the laboratory preparation process and practical industry cable production process in which antioxidants were usually used. Antioxidants can capture free radicals which hinder crosslinking process. Any antioxidant was not used in our sample processing, and therefore, high values of gel content were obtained.

It can be also seen in Figure 3 that, the gel content values of silane-grafted XLPE samples after the hydrolyzed condensation for 15 and 30 days tends to increase slightly with the increasing of VTMS concentration, while the longer time for the hydrolyzed condensation seems to further increase the gel content of silane-grafted XLPE.

As can be expected, the existence of the peroxide-crosslinked network may probably show the so-called "inhibiting effect" on the formation of silane-crosslinked structures. Not only is the water

penetration into XLPE rather difficult, but also the VTMS concentration is relatively low. These may be likely the main reasons both why the increment rate of the gel content is not so sensitive to the increase of the VTMS concentration and why the gel content behaviors of the silane-grafted XLPE samples are not clearly different from each other after the hydrolyzed condensation for 15 and 30 days, respectively.

It should be noted in Figure 3 that the gel content of the silane-grafted XLPE before the hydrolyzed condensation decreases with increasing of VTMS in the VTMS concentration range of 0 to 0.4 phr, whereas it begins to increase beyond 0.4 phr. These results may be ascribed to the competition between the peroxide-crosslinking and the silane-grafting reactions of polyethylene when the samples were crosslinked at 170°C and 10 MPa: the radicals produced by peroxides could be partly consumed by the silane-grafting as well as by the peroxide-crosslinking.

In range of the relatively low VTMS concentration (<0.4 phr), the higher the concentration, the further the silane-grafting reaction of the radicals caused by peroxides could occur. However, the ratio of the grafted VTMS is so low that the silane-grafting can become saturated with the increase of VTMS concentration, resulting in that the gel content does not decrease anymore.

On the other hand, it can be also excepted that the gel content could increase beyond 0.4 phr VTMS, which may be explained as follows.

1. Little peroxides take parts in the silane-grafting reaction because of its saturation characteristics, whereas most of the peroxides would be consumed by the peroxide-crosslinking reaction.
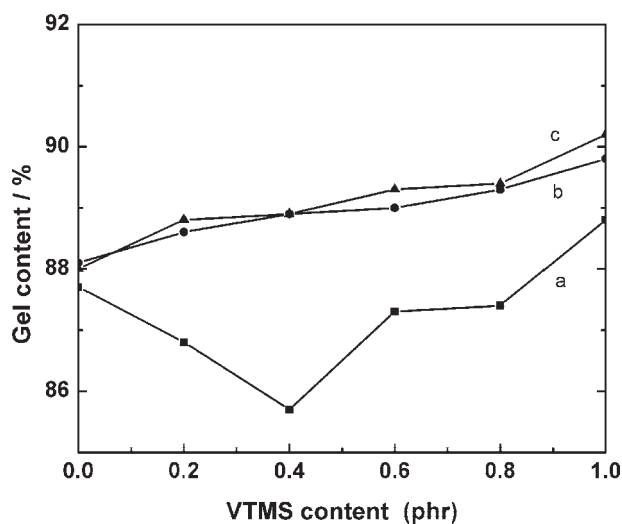

Figure 3 Plots of the gel content of XLPE versus the VTMS content: before the hydrolyzed crosslinking (a); after hydrolyzed crosslinking for 15 (b) and 30 (c) days.
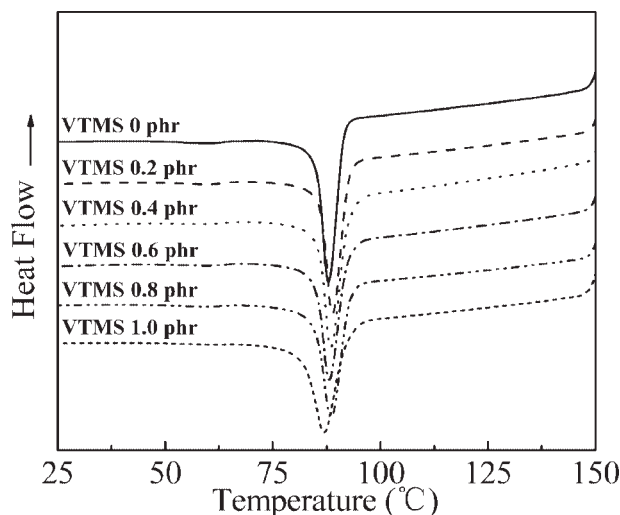
Figure 4 DSC cooling curves of silane-grafted XLPE samples.

2. With the silane-grafting onto the polyethylene chains, the radicals formed in the main chains of polyethylene might be transferred to the ethylene side-groups of VTMS, leading these radicals to be linked with other radicals.

DSC characterization

The DSC melting and crystallization curves of silane-grafted XLPE with different VTMS contents are exhibited in Figure 4.

It is clearly seen from Figure 4 that the peak of crystallization temperature rapidly shifts to higher temperature and then shifts slowly again to the lower temperature. Silane groups are grafted onto XLPE chain and destroy the three-dimensional network structure formed by crosslinking between carbon free radicals. However, since the grafted silanes
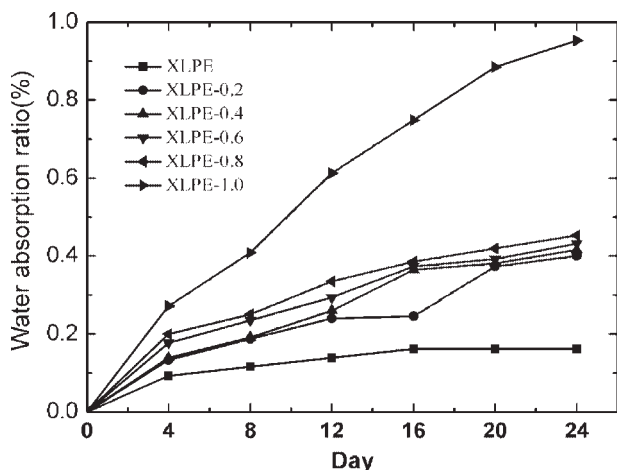

Figure 5 The behaviors of water absorption for the silane-grafted XLPE samples.

occupy the space of carbon radicals belonging to the crosslinked network and the volume of the grafted VTMS is larger, the destroyed crosslinked structure can still inhibit thickening of the lamellar. On the other hand, although the structure of the three-dimensional network can give an obstacle to the movement and arrangement of molecular chains, the silane groups grafted onto the polymer can partly destroy this three-dimensional network structure, allowing the crystallization to become easier.

Water absorption behavior

The aforementioned physico-chemical behaviors of the silane-grafted XLPE samples can be confirmed through their water absorption behaviors. The effects of the VTMS contents on the water absorption ratio (WAR) of the silane-grafted XLPE samples are shown in Figure 5. The neat XLPE sample is found to absorb little amount of water, whereas the WAR
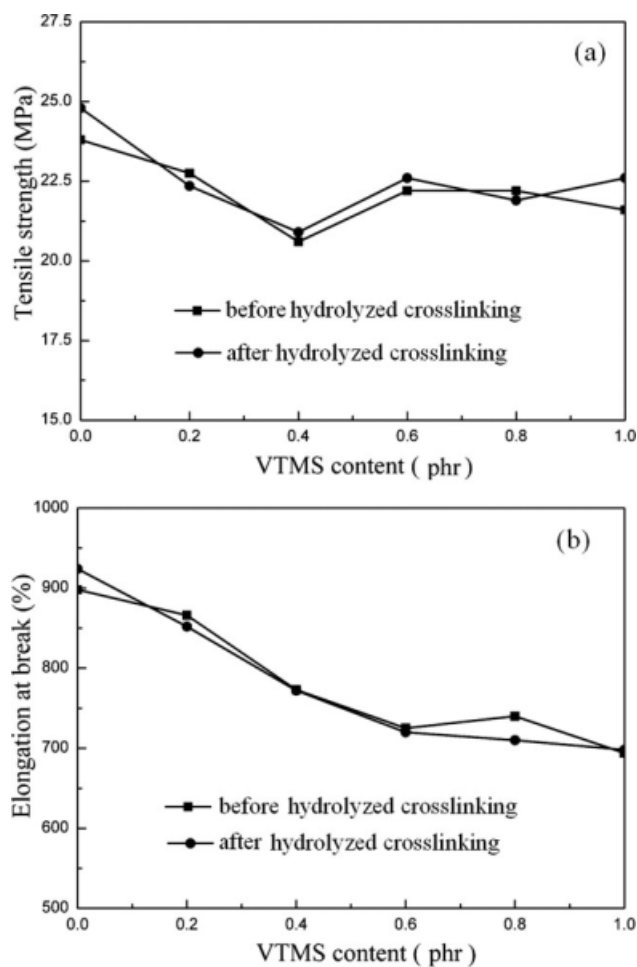

Figure 6 (a) Effect of VTMS content on the tensile strength of XLPE samples before and after the hydrolyzed crosslinking. (b) Effect of VTMS content on the elongation at break of XLPE samples before and after the hydrolyzed crosslinking.
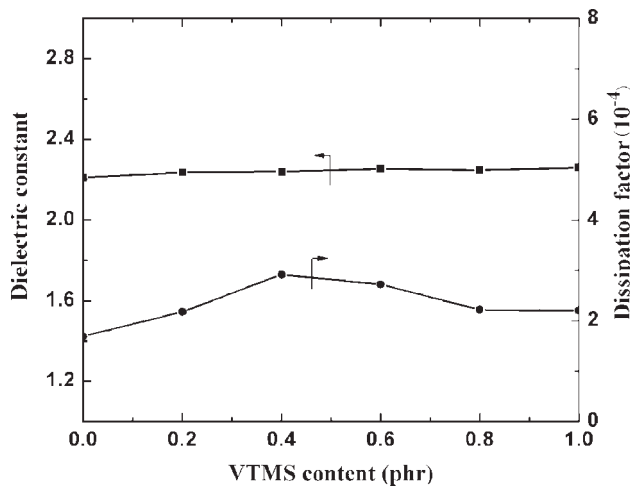
Figure 7 Effect of VTMS content on the dissipation factor and dielectric constant of XLPE.

Figure 8 Characteristic dielectric strength for silane-grafted XLPE samples.

of the XLPE-*g*-VTMS samples increases with the increase of the VTMS content, which may be ascribed to the stronger hydrophilicity of the Si—(OCH$_3$)$_3$ group. This hints at that the hydrophilicity of the samples used has been improved because of the silane group grafted onto polyethylene chains.

Mechanical properties

The effects of the VTMS concentration on the mechanical properties of the peroxide-crosslinked XLPE are shown in Figure 6, which shows that before silane-hydrolyzed crosslinking, the tensile strength of the silane-grafted XLPE decreases significantly with the phr ratio of VTMS content increasing up to 0.4, mainly due to that the silane groups grafted onto XLPE consume a part of radicals caused by the decomposition of peroxides (DCP), resulting in the decrease of the degree of crosslinking. In this case, the elongation at break of the silane-grafted XLPE also displays the similar behavior with the tensile strength. However, beyond the phr ratio of 0.4, the tensile strength begins to increase again, and tends to keep nearly the constant value starting at the phr ratio of 0.6. The similar trends of tensile strength and elongation at break can be also observed after all the specimens were immersed in 1.0*N* sodium chlorides for 30 days at room temperature, but the tensile strength values the silane-grafted XLPE before and after the hydration condensation are surely different from each other, that is, the latter tends to be higher than the former. Such a mechanical behavior also hints at that the additive increase of the density of crosslinking network in the silane-grafted XLPE, caused by the silane crosslinking and also can be expected during the accelerated water-
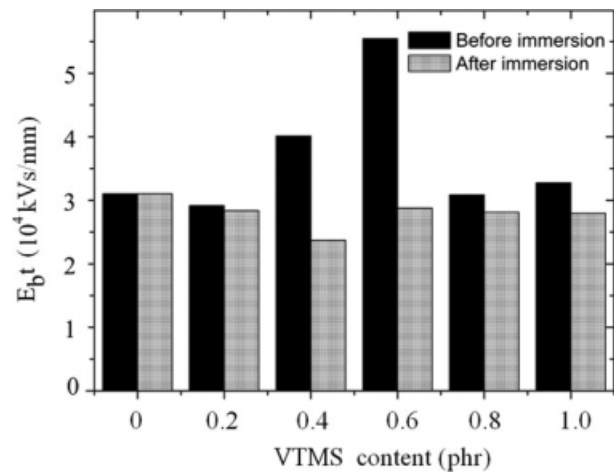
treeing experiment which is performed under the condition of the 1.0*N* NaCl solution and the AC high voltage.

Dielectric properties

It is well known that the addition of impurity can cause the modification of the dielectric properties of matrix, including the dielectric permittivity, the dissipation factor, dielectric breakdown strength, etc.[1]

The measurement results of the dielectric properties for silane-grafted XLPE specimens containing different concentrations of VTMS are shown in Figure 7. It can be clearly seen from Figure 7 that the functional groups of VTMS (Si—OCH$_3$) have a little influence on the electrical property of the silane-grafted XLPE. The permittivity of the silane-grafted XLPE tends to slightly increase with the VTMS
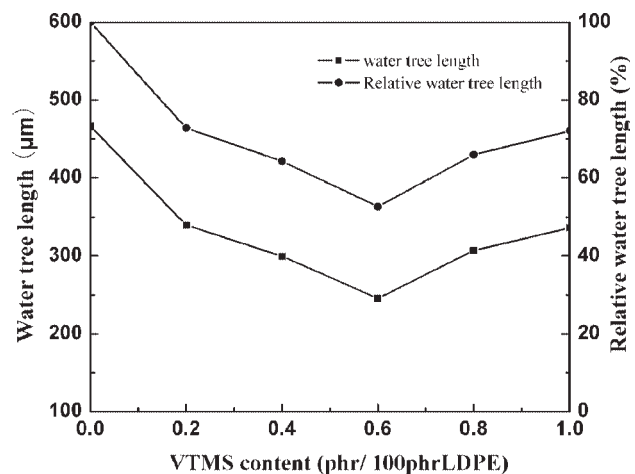

Figure 9 Effect of the VTMS concentration on the water tree length and relative water tree length of XLPE compounds.
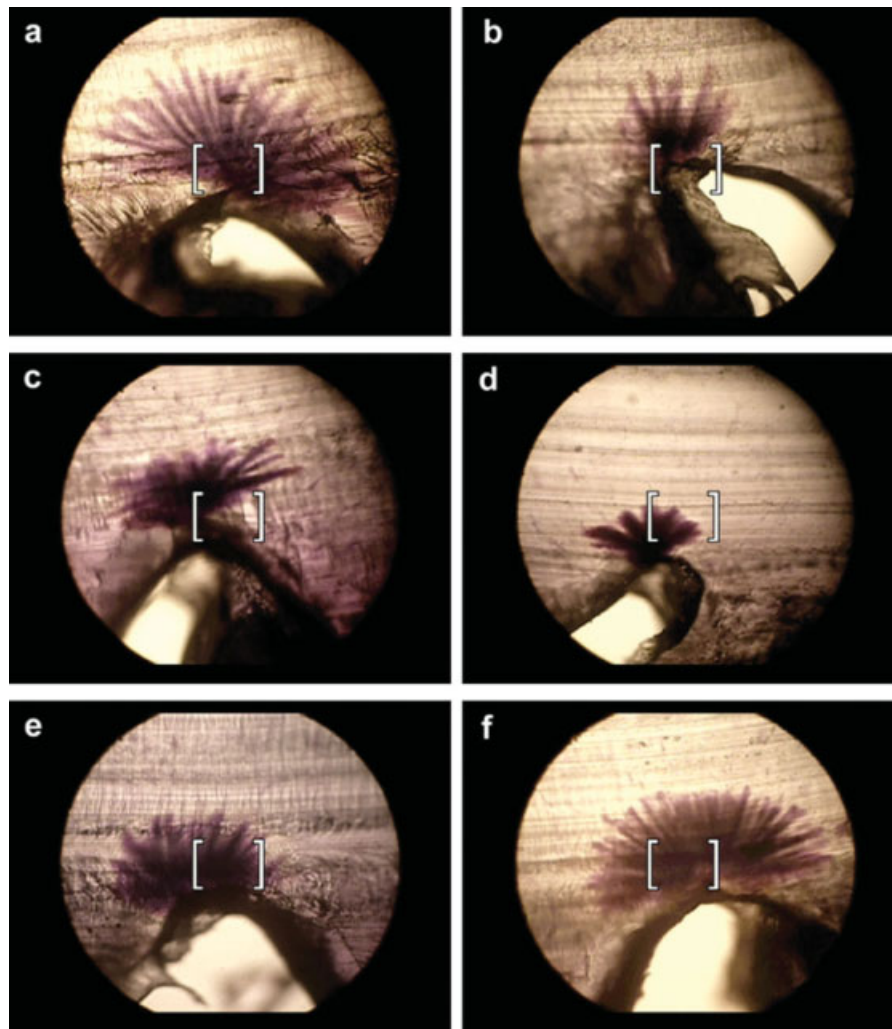
Figure 10 Photographs of typical water trees in the silane-grafted XLPE samples: (a) XLPE, (b) XLPE-0.2, (c) XLPE-0.4, (d) XLPE-0.6, (e) XLPE-0.8, and (f) XLPE-1.0. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

concentration increasing, which may be ascribed to the increase of the density of the crosslinking resulting from the additional silane crosslinking. It is well known that the crosslinked polyethylene has the higher permittivity than the non-crosslinked one.

To investigate the dielectric breakdown behavior of the silane-grafted XLPE in details, the dielectric strength measurements were performed at the room temperature for the silane-grafted XLPE samples before and after immersion in distilled water (30°C) for 30 days (Fig. 8).

It is well known that the dielectric breakdown results for different aging conditions show good agreement with the Weibull distribution. In the case of step or ramp voltage test, the cumulative Weibull distribution function is given by

$$F(Et) = 1 - \exp\left[-\left(\frac{Et}{Et_S}\right)^{\beta}\right]$$

where Et_S is the scale parameters or the characteristic parameter; Et is the random variables; β is the shape parameter or slope parameter; $F(Et)$ indicates the cumulative failure probability for the Weibull distribution.

It should be noted that the dielectric breakdown behavior of the silane-grafted XLPE before hydration is quite similar to its dependence of gel content on VTMS concentration and strongly supports the aforementioned considerations. It is well known that non-crosslinked polyethylene shows higher dielectric strength than crosslinked one at 30°C.[24] Crosslinking weakens the molecular motion and hinders crystallization, which is one of the main reasons why the lower the degree of crosslinking, the higher the dielectric strength becomes for the silane-grafted XLPE investigated in this study.

Also, very interesting is that the values of dielectric breakdown strength corresponding to the VTMS concentrations of 0.4 to 0.6 phr before hydration are
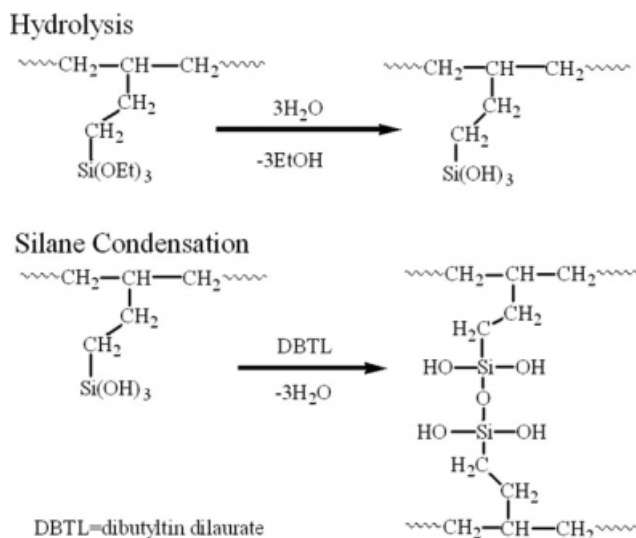
Hydrolysis

Silane Condensation

DBTL=dibutyltin dilaurate

Figure 11 Schematic of a silane hydrolysis and silanol condensation reaction.

apparently reduced again after hydration and that after hydration, any significant differences of the dielectric breakdown strength can be hardly observed in the VTMS concentration range of 0–1.0 phr. As considered earlier, with the aid of water absorbed by the silane-grafted XLPE, the silane-crosslinking network can be additively formed in XLPE during hydration and, as a result, the higher degree of crosslinking may reduce the dielectric breakdown strength. It is of great practical significance that the dielectric breakdown strength values after hydration are nearly independent of whether or not the silane groups are grafted onto XLPE.

Water-tree resistance

The water tree resistance of the silane-grafted XLPE samples studied is evaluated by the water tree length and relative water tree length as shown in Figure 9. Figure 10 shows the photographs of typical water trees in the silane-grafted XLPE samples. It can be seen from Figures 9 and 10 that the silane-grafting can considerably improve the water tree resistance of the conventional XLPE cable insulation: the water tree length decreases from 466 µm to 245 µm when the VTMS content increase from 0 phr to 0.6 phr, while beyond 0.6 phr VTMS, it increases again up to 335.9 µm at 1.0 phr VTMS. According to our experiment, the number of water trees is also smallest when VTMS content is 0.6 phr. It can be also seen from Figure 10 that the water trees have the similar shape, indicating that the water tree initiation and development characteristics are almost the same.

The results obtained are likely due to the grafting of hydrophilic VTMS onto the basic chains of the

peroxide-crosslinked polyethylene and to the silane-condensation reactions occurring during water-treeing test.

According to the propagation mechanisms of water trees, water and electrical field are the most crucial factors for water tree formation.[7,9,25] Because of the inhomogeneous electric field around the surfaces of the defects in the polymer, water drops tend to move towards the points of highest electrical stresses. Then, crazing of the polymer would take place under the action of the alternating Maxwell stresses and compressive stresses due to the diffusion of water. Finally, with the growth of the craze, the craze-like structure of the polymer would lead to the formation of the tree-like microchannels or tubes, which are called water trees.

The principles of a silane hydrolysis and silanol condensation reaction are shown in Figure 11. Silane-crosslinking consumes a part of penetrated water molecules during accelerated aging process. In the case of the relatively low VTMS concentrations, e.g., below 0.6 phr, the hydrophilic $Si-(OCH_3)_3$ groups of VTMS grafted onto the crosslinked polyethylene can react with water so as to be turned into $Si-(OH)_3$, and then the crosslinking between $Si-(OH)_3$ groups occurs under the action of catalyst (DBTL). Silane-crosslinking reaction could lead to the production of water molecules, but more water molecules could be consumed during the change of $Si-(OCH_3)_3$ groups into silanol groups $[Si-(OH)_3]$, that is, the total number of penetrated water molecules decreased and the effect of penetrated water on water tree growth would be depressed during the water tree growing process. Besides crosslinked silanol groups, there existed some silanol groups which did not taken part in the silanol condensation and had a strong interaction with water molecules. These silanol groups were dispersed uniformly in the matrix and could prevent the aggregation of water molecular in matrix, resulting in the decrease of water tree length.

Beyond the VTMS concentration of 0.6 phr, the water tree length begins to increase again with the VTMS content increasing, which can be explained by the agglomeration of water drops caused by the residual silanol groups that have not taken part in the silane-crosslinking reaction and the increase of the total degree of crosslinking. If the VTMS concentration exceeds 0.6 phr, it can be easily expected that excessive silanol groups can cause the water molecules to be aggregated in the matrix and consequently lead to electric field distortion. Because of the considerable difference of the permittivity between the host insulating material (about 2.2–2.3) and water (about 80), the electric field can be so amplified around these aggregated water molecules that water tree can be easily grown. On the other

hand, water-treeing deterioration has been known to mainly occur in the amorphous region of XLPE material.[26] As shown in Figure 3, the increase of the crosslinking network density beyond the VTMS concentration of about 0.4–0.6 phr can widen the amorphous region, which may also give an advantageous condition to water-treeing.

CONCLUSIONS

The effect of silane-grafting on the water tree resistance of XLPE cable insulation has been experimentally investigated together with the analyses of physico-chemical and dielectric characterization.

Our experimental results show that the silane-grafting has a significant effect on the improvement of the water tree resistance of the XLPE cable insulationand. Of practical significance is that the water tree resistance can be considerably improved by grafting VTMS of about 0.6 phr onto the conventional XLPE cable insulation with little influences on its dielectric properties, e.g., the dielectric breakdown strength, dielectric permittivity, and loss tangent. And also, the silane-grafting of XLPE in the VTMS concentrations below 1.0 phr has been found to display insignificant influences on the mechanical properties of the cable insulation.

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