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Study on Water-Tree Aging Characteristics of MAH Grafted Crosslinked Polyethylene

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Abstract

Modifying crosslinked polyethylene (XLPE) insulation materials through Physical or chemical means can improve their water-tree resistance. To explore this enhancement, MAH-grafted XLPE (XLPE-g-xphrMAH, where x=1.0, 1.5 and 2.0) was prepared using a melt blending and grafting modification method, employing low-density polyethylene (LDPE) with varying MAH doping contents. The accelerated aging experiments on water trees were conducted using the water blade electrode method. The findings indicated that the modification through MAH grafting notably suppresses water tree aging in XLPE. Among the samples tested, XLPE-g-1.0phrMAH demonstrated the most effective inhibition of water tree formation, displaying the shortest water tree length. Characterizing the dielectric properties of XLPE-g-xphrMAH samples, revealed that MAH-grafted XLPE leads to an increase in both the relative dielectric constant and the dielectric loss factor. At 50 Hz power frequency, XLPE-g-xphrMAH samples exhibit a relative permittivity below 2.28 and a dielectric loss factor below 0.001. Moreover, the AC breakdown field strength increases by 3.89%, suggesting that XLPE-g-1.0phrMAH displays outstanding insulation performance.

Keywords: Crosslinked polyethylene; Maleic anhydride; Water-tree; Grafting; Dielectric properties

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1. Introduction

Crosslinked polyethylene (XLPE) insulated power cables demonstrate exceptional electrical performance, high transmission capacity and ease of processing, rendering them extensively utilized in power cable insulation[1-5]. When in operation, electro-mechanical stress prompts the infiltration of water molecules into the amorphous phase voids and interfacial imperfections of the crystalline phase within XLPE insulation. Due to their polarity, water molecules have a propensity to cluster within the non-polar polyethylene, thereby compromising the insulation efficacy of the cables and reducing their operational longevity[6-10].

Since the discovery of water trees in the 1960s, numerous experts and scholars have focused on researching anti-water tree insulation materials. Studies have shown that

incorporating polar additives or blending polar polymers into XLPE insulation materials can effectively suppress water trees formation[11-16]. Uehara et al.[17] successfully impeded water dendrite growth in polyethylene by introducing hydrophilic additives. The robust hydrogen bonding between nitrogen atoms in the hydrophilic additives and water molecules in polyethylene hinders the permeation and aggregation of water molecules, thus, effectively inhibiting water dendrite growth. The Insulation Center of Xi'an Jiao-tong University[18-19] discovered that incorporating polyethylene glycol (PEG) into polyethylene can effectively suppress water tree formation, with optimal inhibition achieved at a concentration of 0.5wt%. Lee et al.[20] observed that the addition of 2wt% ethylene vinyl acetate copolymer (EVA) to polyethylene exhibited superior inhibitory effects on water trees. Dang Zhimin et al.[21] reported that introducing 1wt% ethylene acrylic acid copolymer (EAA) into modified XLPE led to reduced water tree initiation probability and branch length. Zhang Chengcheng et al.[22] demonstrated that the inclusion of antioxidants enhances water tree resistance in metallocene polyethylene cross-linked polyethylene (MPE-XLPE) blends, impeding water branch growth. Yang Junsheng and colleagues[23] conducted a melt blending of styrene ethylene butene styrene block copolymer (SEBS) and maleic anhydride grafted styrene ethylene butene styrene block copolymer (SEBS-g-MA) with polyethylene. They observed that a higher mass fraction of SEBS resulted in a more effective inhibition of water tree formation in the blend. Additionally, they noted that the shortest water tree branch length occurred at a mass fraction of 4.76wt% for SEBS-g-MA. Previous research has indicated that while polar additives can impede water tree growth in insulation materials, they have a tendency to precipitate, thereby compromising their effectiveness in water tree inhibition. Mixing polar copolymers with XLPE insulation material can hinder water tree development but may also diminish the electrical performance of XLPE insulation material. This study aims to identify a polar additive capable of inhibiting water tree growth without precipitation through interaction with XLPE, while still meeting the electrical performance criteria.

Maleic anhydride (MAH) molecules feature polar anhydride groups and easily excitable carbon-carbon double bonds, which break during reactions. The high steric hindrance of MAH's cyclic structure hinders self-polymerization. Consequently, most activated MAH molecules can graft onto XLPE polymer chains, significantly enhancing their hydrophilicity. This study involved the preparation of MAH-grafted XLPE-g-MAH polymers with varying compositions through melt blending. Their grafting efficacy was assessed via infrared spectroscopy, while the polymer's crosslinking degree was evaluated through thermal elongation and gel extraction tests. Mechanical and dielectric properties were analyzed using an electronic tensile tester and broadband dielectric spectrometer, respectively. Accelerated water branch aging experiments were performed on polymer samples employing a water blade electrode. A comparative analysis of the morphology and length of water branches in MAH-grafted polymers with different compositions was conducted. The study also delved into the inhibitory impact and mechanism of MAH-grafted XLPE with polar groups on water branches.

2. Experimental materials and methods

The Materials and Methods should be described with sufficient details to allow others to replicate and build on the published results. Please note that the publication of your manuscript implicates that you must make all materials, data, computer code, and protocols associated with the publication available to readers. Please disclose at the submission stage any restrictions on the availability of materials or information. New methods and protocols should be described in detail while well-established methods can be briefly described and appropriately cited.

2.1. Main raw materials and equipment

Low-density polyethylene (LDPE, LD200GH) pellets, characterized by a density of 0.922 g/cm^3 and a melt flow index (MFI) of 2.0 g/10 min , were manufactured by Sinopec Beijing Yanshan Company in China. Dicumyl peroxide (DCP), appearing as white crystals with a molecular weight of 270.37 was sourced from Sinopec Shanghai Gaoqiao Petrochemical Co., Ltd. Antioxidant (Irganox1010), having a relative molecular weight of 1177.63 and a melting point of 115°C , was supplied by Dongguan Shanyi Chemical Co., Ltd. Maleic anhydride (MAH), provided by Shanghai Maclean Biochemical Technology Co., Ltd., is depicted in Figure 1. All the aforementioned raw materials and reagents were utilized without undergoing any additional purification steps.

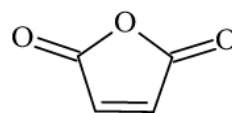


Figure 1 MAH structural formula

The primary equipment includes the RM-200A torque rheometer from Harbin Hapu Electrical Technology Co., Ltd., the FT/IR-6100 Fourier Transform Infrared Spectrometer from Jiashike Trading Co., Ltd., the Alpha-A wideband dielectric spectrometer from Novocontrol in Germany, and the C60/3 YDK test transformer Operation box from Suzhou Huadian Electric Co., Ltd.

2.2. Sample preparation and performance testing

Blended polyethylene containing varying amounts of maleic anhydride (MAH) was prepared by melt blending LDPE, MAH, Irganox1010, and DCP in a torque rheometer operating at 40 rpm and a melting temperature of 110°C for 15 minutes. Initially, LDPE was introduced into the torque rheometer and mixed for 3 minutes, followed by the addition of MAH and 0.3 parts per hundred by weight (phr) of Irganox1010, mixed for 7 minutes, and lastly, 2 phr of DCP was added and mixed for 3 minutes. The resulting blends were compression molded at 110°C under a pressure of 15 MPa using a plate vulcanizing machine, then crosslinked at 175°C under the same pressure for 35 minutes to produce MAH grafted XLPE specimens of varying sizes for different experiments. The MAH grafted XLPE samples with 1.0, 1.5, and 2.0 phr MAH were denoted as XLPE-g-1.0phrMAH, XLPE-g-1.5phrMAH, and XLPE-g-2.0phrMAH, respectively.

A Fourier transform infrared (FTIR) spectrometer was utilized to validate the melt grafting reaction using a 10cm diameter specimen with a thickness of $300 \mu\text{m}$ within the wavenumber range of $500\text{--}4000 \text{ cm}^{-1}$.

The thermal elongation test adhered to Standardization Administration of China (SAC) Publication No. GB/T 2951.21-2008, focusing on insulating and sheathing materials of electric and optical cables, specifically elastomeric compounds for ozone resistance, hot set and mineral oil immersion tests[24]. A dumbbell-shaped specimen, $1.0 \pm 0.2 \text{ mm}$ thick, was employed, as illustrated in Figure 2, applying a test tensile stress of 0.2 N/mm^2 at

200 °C. The average value of three samples determined the thermal elongation and permanent deformation rate of the material.

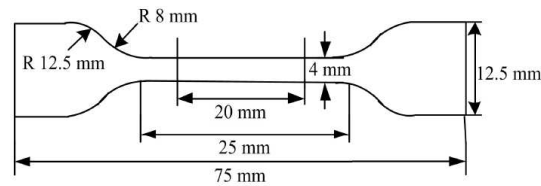


Figure 2 The size schematic diagram of the dumbbell shaped specimen

The gel content test followed the procedure outlined in Appendix A of the Machinery Industry Standard of China (MISC) Publication No. JB/T 10437-2004 for cross-linkable polyethylene insulating compounds used in wires and cables. A specimen was immersed in xylene solvent maintained at a slight boiling state for 10 hours. Subsequently, the specimen underwent drying in a vacuum oven at 150 °C for 2 hours until reaching a constant weight. Two specimens of each material type were tested, and the average value was considered the gel content.

The static contact angle θ of a water droplet on a clean film specimen with a thickness of 300 μm was determined using a contact angle measuring instrument (JC2000C1). Five specimens of each material type were tested, and the average static contact angle θ was recorded.

An aluminum electrode with a diameter of 25 mm was vapor-deposited on both sides of a specimen measuring 35 mm in diameter and 200 μm in thickness using a vacuum coating machine. The relative dielectric constant and dielectric loss factor were evaluated using a broadband dielectric spectrum analyzer (Alpha-A, Novocontrol, Germany) within an experimental frequency range of 10^{-1} – 10^6 Hz.

The breakdown field strength of the sample with a diameter of 80 mm and a thickness of 70 μm , underwent testing using an AC experimental transformer with a linear boost of 500 V/s. Twelve samples were tested for each material and three breakdown points were selected for measurement per sample. Subsequently, the experimental data on breakdown field strength were fitted using the Weibull statistical method.

2.3 Water-tree aging experiments

The experimental setup for the water blade electrode method is illustrated in Figure 3. Prior to testing, a 30 μm thick steel blade with a tip curvature radius of 10 μm was vertically inserted into a sheet-shaped specimen measuring $100 \times 100 \times 4$ mm to create a blade-like cut defect on the surface. The distance between the tip of the cut defect and the opposite surface of the specimen was approximately 2 mm, with a separation of 10 mm between the two cut defects. The polypropylene pipe has a height of 70 mm and an inner wall diameter of 65 mm. The sodium chloride solution concentration is 1.8 mol/L. The specimens were subjected to a continuous of high voltage at 4 kV effective value and 3 kHz frequency for 192 hours. Specimens aged for 24, 48, 96, 144, and 192 hours were examined to observe water-tree morphology. Prior to the water-tree aging experiment, the experimental setup was placed in a vacuum oven for 30 minutes to remove residual air. Following the aging test, thin slices approximately 180 μm thick were vertically cut along the sample blade defect, then immersed in methylene blue solution at 90 °C for 4 hours. The water-tree morphology in each slice was analyzed using a polarized light microscope and documented.

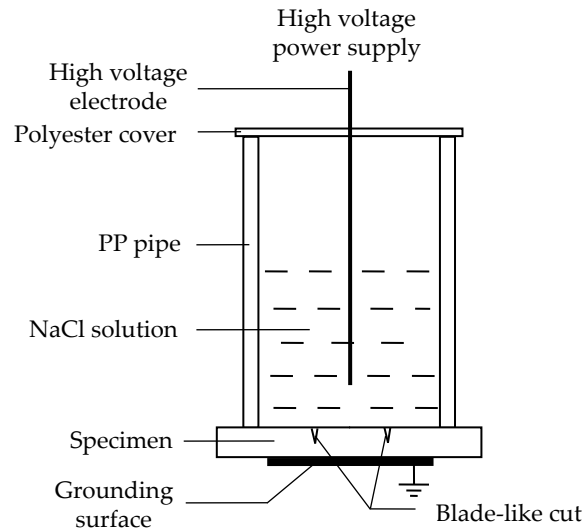


Figure 3 The schematic diagram of the experimental setup for water tree tests

3. Results and discussion

3.1. Infrared spectrum

Figure 4 display the FTIR spectra of LDPE + DCP, LDPE + DCP + MAH and XLPE-g-MAH both pre- and post- degassing. In comparison to LDPE + DCP, the FTIR spectra of LDPE + DCP + MAH in Figure 4 exhibit an absorption peak at 1735 cm^{-1} corresponding to the C=O stretching vibration in MAH, and an absorption peak at 870 cm^{-1} representing the out-of-plane bending vibration of C=C-H[25]. Follow vacuum degassing, the intensity of these two absorption peaks notable decreased, suggesting the escape of MAH from the blend specimens during degassing, indicating that the blend of MAH and LDPE was merely a result of mechanical mixing. In Figure 4, the FTIR spectra of XLPE-g-MAH showed a slight reduction in the C=O absorption peak at 1735 cm^{-1} , while the absorption peak at 870 cm^{-1} vanished entirely. This suggests that MAH remained in the sample, but the C=C double bond in MAH was consumed, indicating grafting of MAH onto XLPE through a grafting reaction. These results directly demonstrate the successful preparation of XLPE-g-MAH during the experiment.

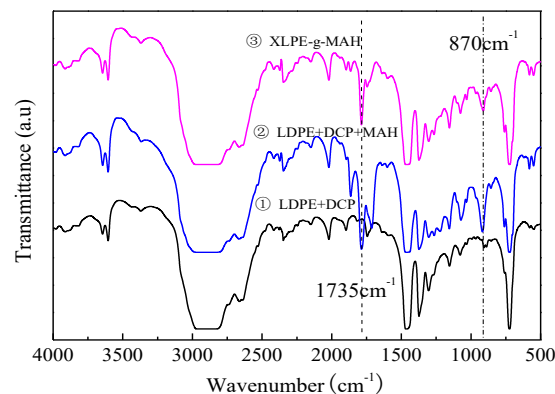


Figure 4 Infrared spectra of MAH grafted modified XLPE and its reference samples

3.2 Crosslinking extent

The thermal elongation and gel extraction tests were conducted to assess the crosslinking degree of XLPE. Table 1 presents the thermal elongation and gel content of XLPE and XLPE-g-MAH with varying MAH concentrations.

Table 1 Thermal elongation and gel contents test results of XLPE and XLPE-g-MAH

Material	Thermal elongation (%)	content (%)
XLPE	42.5	90.20
XLPE-g-1.0phrMAH	43.3	89.68
XLPE-g-1.5phrMAH	43.7	88.59
XLPE-g-2.0phrMAH	55.6	85.42

The results from Table 1 indicate that incorporating 1.0 phr and 1.5 phr MAH into XLPE has negligible effects on its thermal elongation properties, with the crosslinking degree remaining largely unchanged. The variation in gel content suggests that DCP serves as an initiator for both grafting and crosslinking reactions, with the crosslinking process unaffected by DCP consumption during grafting when MAH content is low. The thermal stability of the grafted material is maintained. However, at a MAH mass fraction of 2.0 phr, a high MAH concentration leads to increased grafting reactions between DCP-initiated LDPE macromolecular radicals and MAH, reducing crosslinking points in XLPE. Consequently, the thermal elongation of XLPE-g-MAH rises while the crosslinking degree decreases, yet still meeting the operational requirements of power cables. Furthermore, all samples returned to their original length post experiment, indicating no permanent deformation upon cooling.

3.3 Static contact angles

Water droplet images on the sample's surface were collected using a static contact angle measuring instrument. Measurement software was employed to create a tangent line on the water droplet's surface at the intersection of air, water and material. The angle θ formed between this tangent line and the material surface represents the static contact angle. Figure 8 illustrates the static contact angles of XLPE and XLPE-g-MAH samples.

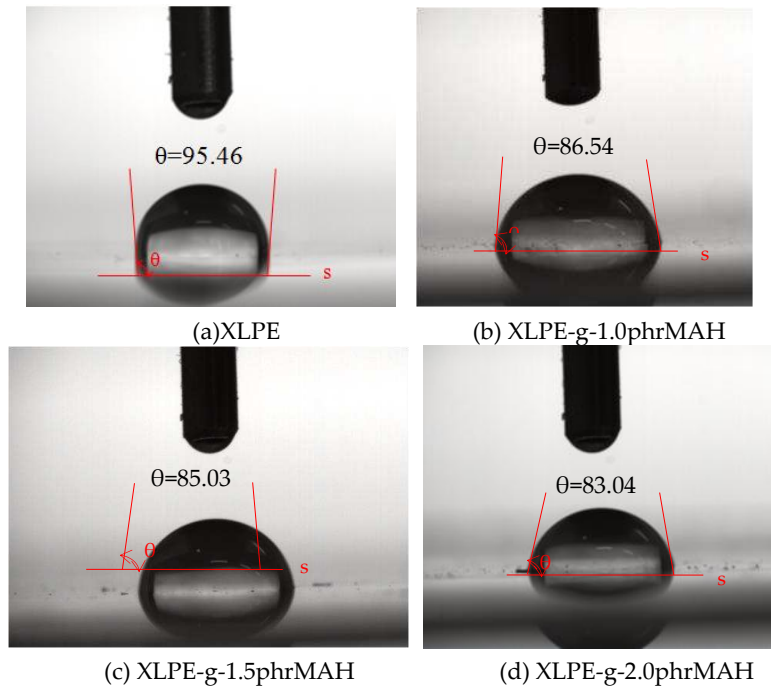


Figure 5 Static contact angles of XLPE and XLPE-g-MAH

Figure 5 illustrates that the contact angle θ of cross-linked polyethylene exceeds 90° , indicating a hydrophobic surface. Subsequently, the contact angle θ notable reduces upon the grafting of MAH onto XLPE. As the MAH content rises, the contact angle of decreased further, with all contact angles of XLPE-g-MAH measuring less than 90° , signifying hydrophilicity on the solid surface. This shift towards hydrophilicity can be attributed to incorporation of polar groups during MAH grafting, leading to an increased polarity of

the polymer chain, heightened hydrophilicity, and diminished contact angle of XLPE-g-MAH. The elevated hydrophilicity of polar groups promotes the dispersion of water molecules within the polymer, hindering water aggregation.

3.4 Dielectric properties

Figure 6 illustrates the frequency-dependent curves of the relative dielectric constant and dielectric loss factor for XLPE and XLPE-g-MAH. XLPE displayed the lowest relative dielectric constant, while the dielectric constant of XLPE-g-MAH showed an increasing trend with the incorporation of MAH, correlating positively with the MAH content. This behavior stemmed from XLPE being a nonpolar dielectric characterized by a relatively small dielectric constant. The introduction of MAH led to the incorporation of polar groups into the XLPE polymer matrix through grafting reactions. Consequently, the grafted polymer displayed interfacial polarization under the influence of an electric field, thereby elevating the relative permittivity of XLPE-g-MAH. Nonetheless, the marginal maleic anhydride in XLPE-g-MAH limited the extent of the permittivity increase.

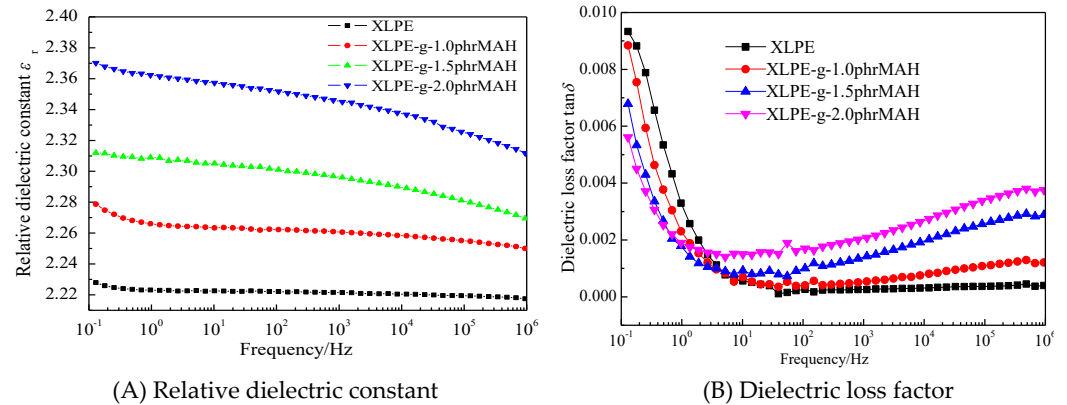


Figure 6 Dielectric spectra of crosslinked polyethylene (XLPE) and chloroacetic acid allyl ester grafted XLPE (XLPE-g-MAH).

Figure 6 (B) illustrates a notable increase in the dielectric loss factor of XLPE-g-MAH in comparison to XLPE, which escalates with higher MAH content. Dielectric loss results from the conductivity and relaxation polarization of the dielectric material. Internal leads to energy dissipation through heat generation within the dielectric. In the polarization phase, the movement of charged groups results in energy absorption of this energy to neighboring groups. This conversion of electro-magnetic energy into thermal energy induces heat production within the dielectric[23]. The significant increase in dielectric loss of XLPE-g-MAH can be attributed to two main factors. Firstly, impurity particles and residual maleic anhydride introduced during the grafting process can migrate under an electric field, Leading to leakage current and conductivity loss. Secondly, the relaxation polarization of XLPE-g-MAH rises, with the extent of increase being dependent on the grafting level. Higher grafting levels result in greater dielectric loss due to relaxation polarization. At a power frequency of 50 Hz, the dielectric loss factor of XLPE-g-1.0phrMAH is below 0.001, similar to un-grafted XLPE. XLPE modified with 1.5 phr MAH and 2.0 phr MAH exhibit dielectric loss factors not exceeding 0.005, meeting the specification for water-resistant XLPE cable insulation materials outlined in the power cable industry standard DL/T 1070-2007[26].

Figure 7 illustrates the dielectric strength variation curves of XLPE and XLPE-g-MAH. The Weibull distribution was utilized to calculate the scale parameter α and shape parameter β , where β characterizes the dispersion of the test outcomes. A higher β value indicates lower dispersion. It is evident that the dielectric strength of XLPE-g-MAH exhibits a pattern of initial increase followed by a decrease compared to XLPE samples. Incorporating a small quantity of MAH at 1.0 phr and 1.5 phr enhances the AC breakdown field strength

of XLPE-g-MAH by 3.89% and 4.15%, respectively. The presence of anhydride groups in MAH enhances its ability to capture or impede high-energy electrons in strong electric fields, thereby reducing their mobility. This effect diminishes the likelihood of high-energy electron bombardment on XLPE polymer chains, consequently elevating their breakdown field strength. The addition of 2.0 phr of MAH results in a 0.98% decrease in the AC breakdown field strength of XLPE-g-2.0phrMAH. However, an excessive amount of MAH leads to a slight reduction in the AC breakdown field strength. This decrease may stem from the ionization of un-grafted MAH when subjected to high electric fields, causing local electric field distortion, elevated carrier concentration, localized discharge, and a subsequent decline in dielectric breakdown strength.

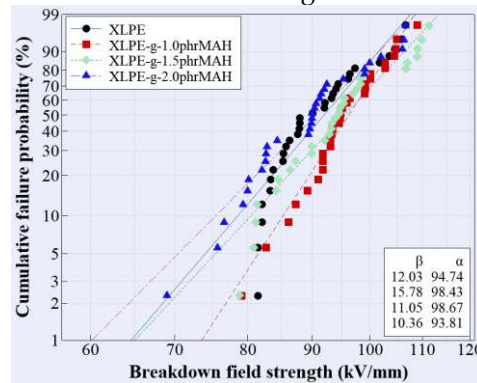


Figure 7 Dielectric strength of XLPE and XLPE-g-MAH

3.5 Aging performance of water trees

Concurrently performing water tree culture experiments on XLPE and XLPE-g-MAH, Figure 8 illustrates the morphology of water tree branches on both materials following 192 hours of applied voltage aging. Evidently, the water tree length of XLPE-g-MAH is notably shorter than that of XLPE. Figure 9 depicts the water tree length variation curves for various samples subjected to identical experimental conditions.

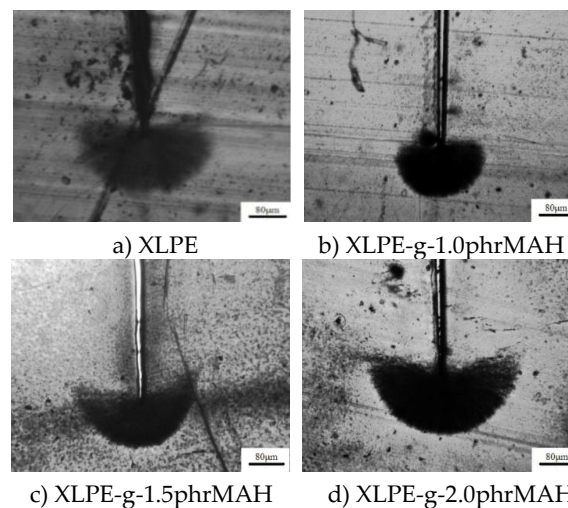


Figure 8 Water tree morphology before and after XLPE graft modification observed with optical microscopy

Figure 8 illustrates fan-shaped water branches near the blade tip, growing radially under an optical microscope. XLPE water branches exhibit higher density compared to XLPE-g-MAH, with longer dendritic discharge channels, indicating more pronounced aging characteristics. The inhibition of water tree branches in XLPE-g-MAH is influenced by the mass fraction of MAH. Adding 1.5 phr and 2.0 phr MAH to XLPE shows no significant impact on water tree inhibition when the applied voltage time is under 48 hours. As the

duration of voltage application increases, the impact of MAH grafting modification progressively intensifies. Following a 192-hour voltage application, the development of water branches in XLPE-g-1.0phrMAH was notably suppressed, with the length of water branches decreasing from 132 μm in XLPE to 91 μm in XLPE-g-1.0phrMAH, representing a relative reduction of 31.1% and indicating a substantial inhibitory effect. XLPE grafted with 1.5 phr and 2.0 phr MAH exhibited some inhibition of water branch growth, albeit not significantly.

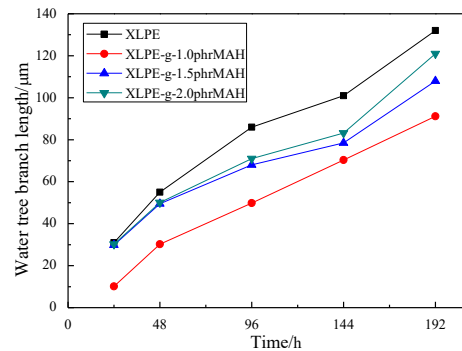


Figure 9 Water-tree length curves of XLPE-g-MAH and XLPE

The growth of water trees in insulating materials can be viewed as an electrical deterioration process, characterized by the migration of charges (electrons, ions, water, etc.) within the material matrix leading to irreversible damage to the molecular chain structure[27]. Cross-linked polyethylene (XLPE) is a semi-crystalline polymer comprising both crystalline and amorphous phases. The molecular chains in the amorphous phase lack order, while the crystalline phase exhibits a well-structured lattice with high stability, facilitating the initiation and propagation of water dendrites from the amorphous phase. In cases where the externally applied electrical stress matches the injected charge, a higher volume fraction of the amorphous phase in XLPE results in a decreased average charge density within the material, reducing the likelihood of reaching the electrochemical potential for polymer degradation. Consequently, grafting MAH is prone to disturbing the symmetry of polyethylene molecular chains, thereby elevating the amorphous regions in XLPE-g-MAH. This in turn, reduces the average charge density, resulting in a decline in both polymer degradation rate and water tree growth rate. However, surpassing 2.0 phr of added MAH leads to an excessive increase in the amorphous phase despite the decrease in average charge density. Paradoxically, this excessive increase facilitates water molecules' ingress, thereby fostering water branch growth.

In the electro-mechanical stress growth mechanism of water-trees in polymers, small water drop-lets (WDs) undergo a transformation from a spherical to an ellipsoidal shape when subjected to an electric field. Simultaneously, they exert a compressive force in the direction of the electric field on the material. Exceeding the bond energy of molecular chains in the polymer leads to deformation or even rupture of these chains. Water molecules permeate into the polymer matrix, penetrating the free volume voids via the amorphous regions, gradually aggregating to form larger microcavities. These water-filled cavities expand and multiply, some coalescing to a micrometer scale, exerting Maxwell stress on the adjacent XLPE molecular chains[28].

XLPE, a non-polar polymer with hydrophobic properties, facilitates the accumulation of water molecules within microcavities under an electric field, leading to the formation of larger water-filled cavities. The interaction between the hydrophilic acid anhydride groups in MAH and water molecules effectively disperses micro water droplets clustered in the amorphous regions amid flake crystals, hindering water condensation within the polymers[11, 29]. Consequently, the adverse electric-mechanical effects of

micro water droplets on insulation defects are mitigated when subjected to an alternating electric field, as illustrated in Figure 10.

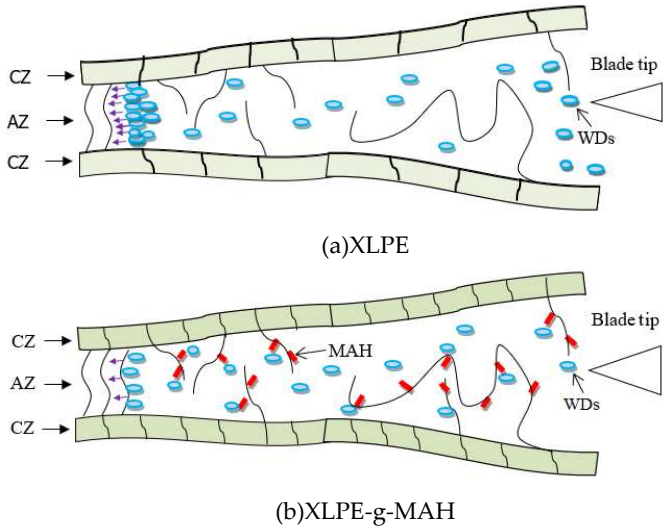


Figure 10 Schematic electro-mechanical mechanism of water micro-beads breaking through the amorphous areas in XLPE-g-MAH

Simultaneously, the grafting of MAH onto XLPE macromolecules increases the interaction energy between the macromolecular chains. This enhancement strengthens the entanglement effect in the amorphous region rendering the molecular chains less susceptible to rupture and enhancing the material's water resistance to some degree.

Based on the experimental findings presented above, the XLPE modified with 1.0 phr of MAH graft exhibits moderate hydrophilicity, leading to a more uniform dispersion of internal water molecules and effective suppression of water branching within the polymer matrix. Nevertheless, exceeding 1.5 phr of MAH for XLPE grafting results in compromised compatibility attributed to distinct molecular polarities. This leads to the formation of multiple incompatible interfaces within the polymer matrix, causing structural imperfections and facilitating the initiation and proliferation of water dendrites.

4 Conclusions

This article extensively investigated the water tree aging behavior of XLPE modified with MAH grafting using the water blade electrode method. The dielectric properties of XLPE-g-MAH were assessed through dielectric spectroscopy and AC breakdown tests. The grafting and surface hydrophilicity of the modified polymers were analyzed via infrared spectroscopy and static contact angle measurements. Based on the experimental findings, the mechanism of water branch suppression in MAH-grafted XLPE was elucidated. The following specific conclusions were drawn:

- 1) The electrical characteristics of XLPE grafted with MAH exhibit a minor influence, leading to a rise in relative dielectric constant and loss tangent. The AC breakdown strength of XLPE modified with 1.0phr and 1.5phr MAH increases, yet slightly diminishes at a 2.0 phr MAH grafting level. Despite MAH modification, XLPE retains favorable insulating properties.
- 2) Incorporating the polar MAH molecule onto XLPE polymer chains enhances the polarity of XLPE-g-MAH, notably boosting the hydrophilicity of the altered XLPE. The polar entities disperse water molecules, reducing their tendency to aggregate.
- 3) The MAH-grafted XLPE enhances polymer resistance to water treeing and restrains water branch formation. Particularly, XLPE-g-1.0phrMAH demonstrates the slowest water branch growth rate and the most effective suppression of water branching.

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