A comparative study of electrical aging of multiwalled carbon nanotubes and carbon black filled cross-linked polyethylene

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ABSTRACT

In this work, XLPE/MWCNT and XLPE/CB nanocomposites have been prepared in order to investigate AC electric field and water effects on electrical aging of XLPE. The mechanical, AC breakdown strength and AC conductivity were tested and the morphologies after 30 days electrical aging were observed using an optical microscope. The results showed that all samples exhibit excellent insulation properties and mechanical properties. Compared with CB addition, the MWCNT composites exhibit better resistance to electrical aging, with the length of electrical aging-induced microcracks in the MWCNT blends decreasing from 104 to 22 \(\mu\)m, and the width decreasing from 87 to 17 \(\mu\)m, which means a reduction of \(\sim80\)% compared of values for neat XLPE in both length and width. However, the XLPE/CB composites have a tendency to promote electrical aging. The mechanism is revealed by comparing the results of the fibrous MWCNTs with the spherical CBs.

GRAPHICAL ABSTRACT

AC electric field and water effects on electrical aging of cross-linking polyethylene.

1. Introduction

Cross-linked polyethylene (XLPE) is widely applied for the insulation of power cables which have largely replaced paper/oil insulation and low-density polyethylene (LDPE) in medium and high-voltage cables, due to its good processability, convenient operation, excellent dielectric properties, low maintenance and cost \([1–4]\). However, in humid environment, the initiation and growth of water trees is the main electrical aging phenomena in XLPE cable. Water tree aging has become the biggest threat to the normal operation of high-voltage cables, seriously affecting the economic development of the region and threatening people’s lives. Water tree aging was first reported by Miyashita \([5]\), which is a degradation that occurs under the combined effects of moisture and the alternating electric fields. Because
the microscopic appearance after electrical aging resembles a tree branch, it is called ‘water tree’. By far, the majority of water tree aging research can be roughly divided into three aspects: (i) the mechanisms of water tree aging [6–9], (ii) the influence of factors on growth and propagation of water trees [10] and (iii) studying of water tree retardant XLPE [11]. The initiation and growth of water trees have been comprehensively studied, and many possible mechanisms have been proposed [6–10]. Numerous experiments have shown that the initiation and growth of water trees are not affected by a single factor, such as electric field intensity and frequency [12], aging time [12, 13], temperature [14] and type of defect [12], but also by the nature of the insulation compound used, such as its morphology [14, 15], and type and content of additives mixed into the compound [16, 17], etc. But not all properties of water trees are thoroughly studied. Generally, it is agreed that the water tree has the form of water-filled micro voids (micrometer scale) and interconnected channels (nano to micrometer scale) [2, 7], which are arranged roughly along the direction of the electric field. The conventional developing water tree retardant XLPE always introduces inorganic nanofillers, i.e., solution compounding and subsequent melt compounding. In this experiment, the maximum amount of MWCNT and CB additive is 0.2 wt%. The XLPE, XLPE/MWCNT and XLPE/CB nanocomposites were treated under accelerated water tree aging environment for 30 days at room temperature to investigate the effect of MWCNT and CB on the water tree resistance. The mechanical properties, AC conductivity and AC breakdown strength were tested to explain the effect of MWCNT and CB addition in XLPE insulating materials.

2. Experimental

2.1. Materials

LDPE (18 D) with a density of 0.92 g/cm³ was purchased from China Petrol Daqing Petrochemical Company. MWCNT (NC7000) with lengths of 1.5 μm and diameters of around 9.5 nm were supplied by Nanocyl S.A (Belgium). Commercial CB particles (VXC500) were provided from Cabot (United States), with an average particle size of 200 nm or less in diameter. Dicumyl peroxide (DCP) is supplied from Chengdu Chron Chemicals Co. Ltd, China. Xylene was bought from Chengdu Chron Chemicals Co. Ltd, China.

2.2. Samples preparation

The XLPE/MWCNT and XLPE/CB nanocomposites with 0, 0.05, 0.1, 0.15, 0.2 wt% nanoparticles were prepared through a two-step processing procedures, i.e. solution compounding and subsequent melt compounding. First, the LDPE was dissolved into 300 mL of xylene and stirred at 80°C for 1 h. The MWCNT powder was also added to 200 mL of xylene and sonicated for 2 h, then stirred for 0.5 h. Afterwards, the LDPE/MWCNT nanocomposites were precipitated out through cooling of the prepared solution by deionized ice water. After that, the nanocomposites were dried in a vacuum oven at 60°C for 48 h until the weights of the samples were...
constant to ensure that residual solvent was completely removed. Second, 3 wt% cross-linking agent (DCP) for LDPE was added by melt blending using a twin-roll Banbury mixer. These LDPE/MWCNT nanocomposites were hot pressed at the temperature of 170 °C for 30 min in a cuboid shape to become the XLPE/MWCNT composites. XLPE/CB nanocomposites were prepared in the same way.

2.3. Characterization

2.3.1. Scanning electron microscope
The morphologies of the MWCNT, CB and their nanocomposites were examined using a field emission scanning electron microscope (Nova NanoSEM450, FEI, USA) after cold fracturing in liquid nitrogen and coating the samples with gold.

2.3.2. Cross-linking degree and crystallinity
The cross-linking degree of all samples was tested by extracting in xylene at high temperature. Crystallization and melting of XLPE/MWCNT and XLPE/CB composites were studied in a Mettler-Toledo differential scanning calorimeter. The results of cross-linking degree and crystallinity are shown in Figure S1 and Table S1.

2.3.3. Mechanical property
Tensile property measurements were based on ASTM D 638-2003 in a SANS materials testing machine (INSTRON 5967), using a cross-head speed of 30 mm/min, and a specimen thickness of about 0.5 mm. Five specimens for each sample were used for the tests.

2.3.4. AC breakdown strength
The AC breakdown strength of the samples was measured under ball electrode at room temperature. Samples with a thickness of 0.4 mm were immersed in transformer oil to avoid surface flashover discharge. The AC voltage was continuously increased at the rate of 0.2 kV/s until the sample was punctured. Twenty breakdown tests were performed on each sample in order to avoid experimental errors. The two-parameter Weibull statistical distribution method was used to treat the breakdown test data. The formula was described as follows:

\[
F(U, \alpha, \beta) = 1 - \exp \left( - \frac{U}{U_0} \right)^\beta
\]

where \( F(U, \alpha, \beta) \) is the probability of breakdown, \( U \) represents the breakdown strength obtained from experiment, \( U_0 \) is the scale parameter representing the characteristic value of dielectric breakdown strength with a cumulative failure probability of 63.2%, and \( \beta \) is the shape parameter, reflecting the unpredictable data.

2.3.5. AC conductivity tests
The AC conductivity, dielectric constant and dielectric loss of the composite films (diameter of 16 mm) were tested by a broad frequency dielectric spectrometer Concept 50 (Novocontrol, Germany). Before characterization, silver paint was brushed on both sides of the samples.

2.3.6. Water tree behavior
Square specimens (50 × 50 × 3 mm) were prepared for water tree aging tests. Three lines of needle hole defects were made in the circular area with a diameter of about 25 mm in the middle of the sample, and the needle hole depth was 1.5 mm. Steel needle tip flat in triangle shape, with a slope length of 3 mm, tip of (17 ± 2) chamfer, tip radius of curvature (2.5 ± 0.5) um. The sample was placed in the water tree aging experimental device as schematically shown in Figure 1(b), and the needle hole defect of the sample was located in the center of the hole at the bottom of the device. A 20% sodium chloride solution was injected into the device and the copper electrode was submerged. The copper electrode under the water tree aging device was grounded with an input amplitude for the upper copper electrode of 7.5 kV and the frequency of 400 Hz. The accelerated
water tree aging experiment was carried out for 30 days at room temperature. The experimental principle of accelerated aging is shown in Figure 1(b).

3. Results and discussion

3.1. Scanning electron microscope imaging

Scanning electron microscope (SEM) was carried out to determine the morphologies of MWCNT, CB powders and their decentralization in the XLPE matrix. It can be seen from Figure 2(a,b) that the fibrous MWCNT and spherical CB were used in this experiment. The MWCNT and CB are dispersed individually and homogeneously throughout the XLPE matrix and very few agglomeration can be seen, as shown in Figure 2(c,d).

3.2. Cross-linking degree and crystallinity

From the Table S1 and the Figure S1, the introduction of MWCNT and CB did not cause changes in the degree of cross-linking and crystallinity of the material.

3.3. Mechanical property

The results of the mechanical properties for the XLPE blends are presented in Figure 3. It can be seen that the XLPE/MWCNT and XLPE/CB lead to an obvious increase in the tensile strength but a slight decrease in the elongation at break as the content of the MWCNT and CB increases. When the content of the MWCNT and CB is 0.2 wt%, the tensile strength of the XLPE/MWCNT is 23 MPa, almost 25% higher than that of XLPE and the XLPE/CB is 21.5 MPa, 16% higher than that of XLPE. The elongation decreases almost 35% in XLPE/MWCNT and 15% in XLPE/CB nanocomposites when the fillers addition is 0.2 wt%. The increase in tensile strength of the MWCNT composites is more pronounced than that of the CB blends. However, the elongation at break is also decreased significantly. This indicates that the interfacial interaction between the fillers and the XLPE is poor.

3.4. AC breakdown strength

The AC breakdown strength of the XLPE/MWCNT and XLPE/CB nanocomposites is estimated to directly assess the insulation performance of the materials. The Weibull probability plots for the AC breakdown strength of the XLPE, XLPE/MWCNT and XLPE/CB nanocomposites are illustrated in Figure 4. According to Figure 4(a), the values of characteristic breakdown strength of the XLPE/MWCNT nanocomposites
increase gradually with increasing MWCNT loading and reaches a maximum value 65.66 kV/mm, almost 20% higher than that of neat XLPE (54.68 kV/mm) at a MWCNT concentration of 0.2 wt%. The AC breakdown strength of XLPE/CB nanocomposites is lower than for the XLPE (i.e. 53.58 kV/mm vs 54.68 kV/mm) when the CB content is 0.05 wt% as shown in Figure 4(b). As the CB concentration increases the breakdown strength increases gradually. It can be seen that both the MWCNT and CB composites play a role in improving the breakdown strength. The impact of the MWCNT composites on the breakdown performance of the sample is more effective than CB at the same filler content. This may be attributed to the incorporation of π electron cloud that is introduced by the MWCNT which can reduce the carrier energy injected from the electrodes, capture space charge and then impede space charge conduction, decreasing the fracture probability of molecular chains by high energy carriers, and hence improving the AC breakdown strength [30].

3.5. AC conductivity

In addition to the AC breakdown strength, the AC conductivity is another important performance parameter for insulating materials. Figure 5 illustrates the AC conductivity of the XLPE, XLPE/MWCNT and XLPE/CB composites. It is found that the AC conductivity increases with MWCNT loading. Although, when the MWCNT content reaches 0.2 wt%, the AC conductivity of the XLPE/MWCNT sample increases by about two times compared with that of neat XLPE, the AC conductivity of all samples is in the same order of magnitude (i.e. 10^{-12} (S/cm)). Compared with XLPE/MWCNT composites, the CB composites at the same filler content exhibit a smaller impact on AC conductivity, which may be related to the inherent properties of the filler. At the same time, dielectric constant and dielectric loss of the composites are shown in Figure S2. As can be seen from Figure S2, XLPE/MWCNT and XLPE/CB composites exhibit low dielectric constant and dielectric loss compared to neat XLPE. The AC breakdown strength, AC conductivity and dielectric properties results show that the XLPE/MWCNT and XLPE/CB nanocomposites exhibit excellent electrical insulation performances at 0.2 wt% filler content.

3.6. Water tree behavior

The water tree resistance of the XLPE, XLPE/MWCNT and XLPE/CB samples is evaluated by the characteristic water tree length (WTL) and water tree width (WTW). The photographs of typical water trees in XLPE composites can be seen in Figure 6(a–i) and the WTL and WTW of the samples are shown in Figure 7. It can be seen from Figures 6(a–e) and 7 that the WTL and the WTW of XLPE/MWCNT composites after 30 days of accelerated electrical aging decrease remarkably with increasing of MWCNT concentration up to 0.15 wt%, while when the MWCNT concentration increases to 0.2 wt%, the WTL and WTW show an increasing trend. Samples incorporating 0.15 wt% MWCNT showed minimum dimensions for WTL and WTW, WTL decreased from 104 μm for neat XLPE to 22 μm, a 79% reduction compared to neat XLPE, while WTW decreased from 87 to 17 μm, i.e. a 80% reduction compared to neat XLPE. However, the CB composites accelerate electrical aging, while the results from Figures 6(a–i) and 7 manifest that
the doping of MWCNT into XLPE can greatly improve the water tree aging resistance of conventional XLPE insulation materials.

However, there has been no clear conclusion regarding the mechanism of water tree aging in XLPE up to now and there is no mechanism that can explain the experimental results under all environmental conditions [6]. Nevertheless, there seems to be growing consensus that the electro-mechanical properties are predominant and that the electro-chemical mechanism is essential [31].

According to the electro-mechanical mechanism of water tree propagation [6], the water droplets infiltrate by the electric field, moves toward the high electric field region and agglomerate to form a large stress concentration point under the effect of the high electric field. When the stress concentration exceeds the local yield strength of the polymer matrix, it causes the molecular rupture of the polymer chains and the microcracks are generated [7, 32, 33]. With the penetration of water droplets, the injection of ionic solution and the electro-oxidation can produce strings of cavities, thus resulting in the propagation of the water tree.

In this experiment, the high permittivity variation between the MWCNTs and the XLPE matrix results in the local enhancement of the electric field at the MWCNT interface under the high-voltage field. After the initiation of the water tree, the tree would grow through the basic polymer and move toward the MWCNTs. When the amount of filler introduced reaches 0.15 wt%, the inhibition of the water tree growth reaches an optimal value. A similar study on the growth of the lamellar graphene used in ceramics for tissue cracking has also been reported [34]. The effect of the fibrous MWCNTs and the spherical CB interface on the water tree growth could be explained by a physical model.
which is shown in Figure 8(a,b). As shown in Figure 8(a), after the initiation of the water tree, it starts to grow through the base XLPE and moves toward the MWCNTs because of an enhancement in the local electric field around the MWCNTs. The MWCNTs are too hard and strong to destroy but can disperse the electric stress as a stress carrier, resulting in an increasing time of water tree growth. On the other hand, under the action of electric field force, the water in the tip of the water tree will disperse around the MWCNTs on account of the weak interaction between the MWCNTs and the polymer matrix interface and the local enhancement of the electric field, so that the water will be dispersed around the MWCNTs instead of flocking together, thus stress concentration under the action of an electric field are avoided. Therefore, the water tree will bifurcate as it encounters a MWCNT and grows along the ends of a MWCNTs on account of the electric field at both ends of the rod-shaped packing being greater than in the middle, the branch of water tree will be obstructed by another MWCNT because of the randomly distributed MWCNTs. Finally, the tree will grow around the MWCNT, from the basic XLPE to another MWCNT, which indicates the aging resistance and cracking resistance of XLPE. In addition to this, the MWCNT composites increase the tensile strength and AC breakdown strength of the matrix material causing the higher fracture strength which indicated that more force is needed to break the matrix material, leading to an increase in resistance of water tree aging. The results of the XLPE/CB composites can verify this model indirectly. As shown in Figure 8(b), the water would grow through the base XLPE and move toward the CB because of an enhancement of the local electric field around the CB. Under the action of the electric field, the water penetrates downward along the weak interface between the CB and XLPE, and the water will gather at the bottom of the CB, then leading to stress concentrations and triggering new cracks. Thus, three-dimensional spherical CB particles would accelerate water penetration, and accelerate the water tree aging process. Doping of spherical CB nanoparticles promoted the growth of water trees to some extent, and further explains the mechanism of the inhibition effect of fibrous MWCNT on water tree aging.

From the viewpoint of electro-chemical mechanisms [7], there are two possible ways of chemical reactions in the aging area: one is the chemical reaction of ions in the water or the impurity of ions in the polymer at the water and polymer interface when the water tree is triggered, the other is water decomposition or electrolysis free radicals initiate reactions. Water molecules are first ionized to generate free radicals and free electrons and the free radicals react rapidly with the polymer to form polymer radicals. At the same time, free electrons may react with oxygen to form peroxide radicals. Oxygen reacts with polymer radicals to form carbonyl groups and hydroxyl groups, promoting the breakage of the molecular chain, resulting in local degradation of the polymer to form micropores. As the aqueous solution continues to permeate in the polymer, the polymer around the micropores is gradually degraded, eventually forming a water tree [35]. The effect of MWCNTs on water trees can also be explained by electro-chemical mechanism. Some researchers have reported that the carbon nanotube is able to act as a hydroxyl and hydrogen free radical scavenger [36, 37]. When the tree grows through the base XLPE and moves toward the MWCNT, free radicals generated by electrolyzed water will be trapped by the MWCNT instead of continuing to attack the XLPE molecular chain. Therefore, the electro-chemical action would be delayed and manifest itself as an inhibition of water tree growths.

4. Conclusions

In summary, in this work XLPE/MWCNT and XLPE/CB nanocomposites were prepared through a two-step processing procedure, i.e. solution compounding and subsequent melt compounding. With this simple method, nanocomposites with good filler
dispersion can be obtained at low filler loadings below 0.2 wt%. The AC breakdown strength of XLPE/MWCNT and XLPE/CB is higher than that of neat XLPE and maintains a low AC conductivity. The above results show that the introduction of conductive MWCNTs and CBs at very low loading levels can also maintain excellent insulation properties. The neat XLPE, XLPE/MWCNT and XLPE/CB nanocomposites were treated under accelerated electrical aging environment for 30 days and their morphologies were observed using an optical microscope. When the content of MWCNT is 0.15 wt%, the length of water trees in the samples was decreased from 104 to 22 μm, while the width of the water tree decreased from 87 to 17 μm, which means ~80% reduction compared of the value for neat XLPE in both length and width. This study shows that very low MWCNT loadings can greatly increase the water tree resistance of XLPE, extending the service life of high-voltage cables. Water tree inhibition of both types of composites were analyzed for electro-mechanical and electro-chemical mechanisms by comparing the results of MWCNT- and CB-based systems. The proposed simple but effective method can be used to extend the life of medium and high-voltage cables, thus saving resources and protecting the environment.

Disclosure statement
No potential conflict of interest was reported by the authors.

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