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MODIFICATION OF POLYETHYLENE BY *in situ* FORMED SODIUM ACRYLATE*

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Abstract To improve the water tree resistance of PE, linear low-density polyethylene (LLDPE) was compounded with sodium acrylate (NaAA) for *in situ* polymerization, in which NaAA was formed through the neutralization of acrylic acid (AA) with sodium hydroxide (NaOH) before adding dicumyl peroxide (DCP) to initiate the *in situ* graft polymerization and homo polymerization. A series of LLDPE/NaAA compounds were investigated for their water absorption ratio (WAR) measurement, water treeing, mechanical and dielectric properties. The results strongly suggest that NaAA can improve the water tree resistance of LLDPE. In addition, the LLDPE/NaAA compounds possess satisfactory mechanical properties and dielectric properties. Characterization of LLDPE/NaAA compounds by using Fourier transform infrared spectrometry (FTIR) suggests that the neutralization and polymerization reaction could be achieved effectively. Using adequate DCP content is the key factor for controlling the polymerization of NaAA with precise conversion ratio.

Keywords: Polyethylene; Water trees; Cable insulation; Sodium acrylate.

INTRODUCTION

Polyethylene (PE) has been widely used in the extruded cable insulation because of its excellent dielectric and mechanical properties. Nevertheless, dielectric and mechanical resistances are the two most important polymer properties to be considered in choosing a material for cable insulation. A very important property for polymer-insulated power cables is the "water tree" resistance of the material[1]. Water treeing has been known as one of the most important causes for failure in power cables with polymeric insulation. Since 1969, when the discovery of degradation of PE under the combined action of water and electric stress was published by Miyashita, this kind of degradation has been called "water tree"; thereafter, much relevant research work has been carried out[2].

Over the last three decades, many speculations have been put forward to explain the processes and mechanisms of the formation and growth of water trees. But clearly, controversies still exist today[3–8]. Although they focused on the researches of the characteristics and mechanisms of water treeing, many researchers have attempted to improve the water tree resistance of cable insulation. However, water treeing is still the main cause for the insulation breakdown, especially in medium voltage cables. Due to such a great importance of water treeing as a cause of insulation breakdown, it is required to develop water tree resistant materials for power cables.

According to the literature, water tree resistance of insulating materials has been improved by chemical and physical modification, including the use of additives, blending, copolymerization and other techniques[2, 9, 10]. The water tree resistance of PE insulation has been improved by the reduction of the impurity of PE insulation. Lee and Turbett *et al.* improved the water tree resistance of polyethylene by blending it with ethylene vinyl acetate (EVA) or polyethylene glycol (PEG)[11–14]. But EVA and PEG are polar polymers and have poor

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compatibility with the polyethylene insulations, so they cannot be well dispersed throughout the blend. Ysauo *et al.* attempted to blend polyethylene with lipophilic surfactants[15, 16]. However, the surfactants are likely to be lost when in contact with water, which would deteriorate the water tree resistance of cable insulations. Ethylene-*co*-acrylic acid (EAA) was used as an additive to improve the water tree resistance of PE insulation because EAA has excellent compatibility with PE[17–19].

Following the previous researches, we have found that the improvement of water tree resistance can be obtained by using the additives that possess sufficient hydrophilicity to allow the polyethylene to link water molecules and prevent them from diffusing into the material, and possess sufficient compatibility with polyethylene to prevent loss or clustering of the additives and keep diffused water distributed evenly in the mass[20]. Having in mind that the carboxy group has strong hydrophilicity, the water tree resistance should be improved if the molecular chains of polyethylene could link carboxy groups.

Thus, the purpose of the present work is to explore a new approach for the preparation of water tree resistant insulating materials. In order to improve the water tree resistance, sodium acrylate was *in situ* formed in an LLDPE matrix through the neutralization of NaOH and acrylic acid (AA). The neutralization of AA with NaOH and the polymerization of resulting NaAA were investigated. Moreover, the effects of NaAA and DCP contents on the hydrophilicity, mechanical properties, dielectric properties and water treeing of LLDPE/NaAA blends were explored.

EXPERIMENTAL

Materials

The raw materials used in this work are given in Table 1.

Table 1. Raw materials

Materials	Properties	Source
Linear low-density polyethylene (LLDPE)	LL4004 Density 0.924 g/cm^3 Melt index 3.6 g/10 min	Exxon Mobil in Saudi Arabia
Acrylic acid (AA)	Chemical pure	Shanghai Wulian Chem. Co., Ltd., China
Sodium hydroxide (NaOH)	Chemical pure	Shanghai Chemical Co., Ltd., China
Dicumyl peroxide (DCP)	Purity 99.5%	Shanghai Gaoqiao Petroleum Co., Ltd., China
Sodium chloride (NaCl)	Chemical pure	Shanghai Chemical Co., Ltd., China

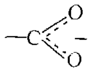
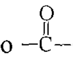
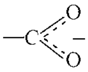
Sample Preparation

LLDPE was melted in a HAAKE Rheometer RC90 with a rotor speed of 60 r/min and a temperature of 170°C. During the compounding operation, NaOH was first added to LLDPE followed by AA. After the ingredients were finely dispersed, a polymerization initiator, DCP, was added. The whole compounding process lasted for about 20 min.

The NaAA contents shown in all the figures were the theoretical values calculated from the NaOH and AA contents. The neutralization degree was supposed to be 100%, and the NaOH/AA molar ratio was 1.0.

Characterization

Infrared spectroscopic measurement

IR spectra were recorded on a Paragon 1000 FTIR spectrometer (Perkin Elmer). The sample of AA was prepared *via* the casting of AA onto a KBr disc. The LLDPE and LLDPE/NaAA compounds were prepared into thin films at 170°C for 5 min. Then, for characterizing the polymerization of NaAA, one of the films of LLDPE/NaAA compounds was extracted with distilled water in a Soxhlet extractor for 3 days to remove the residual NaAA, and finally, it was dried in a vacuum oven at 80°C for 12 h. To characterize the graft polymerization of NaAA, another film of the LLDPE/NaAA compound was immersed in an excess amount of hydrochloric acid at room temperature for 3 days, and the  $-\mathrm{C}(=\mathrm{O})\mathrm{O}^-$ group was transformed into  $-\mathrm{C}(=\mathrm{O})-\mathrm{OH}$ group in the LLDPE/NaAA compounds by the reactions of the  $-\mathrm{COO}^{-}$ group and hydrochloric acid; then, it was extracted with methanol in a Soxhlet extractor for 3 days to remove the remaining AA, poly(acrylic acid) (PAA); finally, the film was dried in a vacuum oven at 60°C for 12 h[21].

Polymerization conversion ratio

The polymerization conversion ratio (CR) of NaAA in the LLDPE/NaAA compounds was determined as follows:

$$\text{Conversion ratio } (\%) = (W_0 - W_t)/W_0 \times 100$$

where W_t is the weight of the unreacted NaAA, and W_0 is the weight of the original NaAA in the LLDPE/NaAA compounds.

Gel content

Approximately 0.2 g of each sample was exactly weighed and placed in a 120 mesh copper net, which was extracted in a Soxhlet extractor for 48 h using xylene as extractant. After extraction, the copper nets were dried in an oven at 80°C until constant weight. The gel content is 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.

Hydrophilicity

Hydrophilicity of the sample was expressed in terms of water absorption ratio[20], which was measured by using a square sample (50 mm × 50 mm × 1 mm), by the immersion/gain method. Firstly, the samples were dried for 12 h at 80°C in a vacuum oven, and their original weights (M_0) were recorded. Then the samples were immersed in an excess amount of deionized water at room temperature for different periods of time until absorption equilibrium was reached. The weight of each specimen (M_t) was determined, after removing its surface water by blotting with filter paper.

The water absorption ratio (WAR) was calculated as follows:

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

Mechanical properties

The tensile tests of the 1 mm-thick specimens were performed according to ASTM D 638-2003 in an Instron series IX 4465 materials tester, with a grip separation rate of 250 mm/min.

Dielectric properties

The volume resistivity was measured on a ZC-36 megohmmeter (Shanghai Precision and Scientific Instrument Corp., Shanghai, China) according to ASTM D 6095-1999. The dielectric constant and the dissipation factor were measured on a QS30 high-voltage Bridge (Shanghai Huguang Corp., Shanghai, China) according to ASTM D 150-1998. The dielectric breakdown strength was measured on an AHDZ-10/100 AC dielectric strength tester (Shanghai Lanpotronics Corp., Shanghai, China) according to ASTM D 149-1997.

Water treeing

The cup-shaped samples were employed to carry out the experiment of the water treeing[18]. The schematic diagram of the test cell for the measurement of water tree length was shown in Fig. 1. The cup-shaped samples with 20 conical defects were prepared by compression molding at 170°C for 5 min using a mold. The distance from the needle tip to the bottom of cup was 1.5 mm and the tip radius of each needle was 5 μm. The experiment of water treeing was carried out under the action of the applied voltage of 5 kV at the frequency of 1 kHz and room temperature for 15 days. After the experiment, 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. The value averaged from 20 defects was taken as the water tree length (WTL).

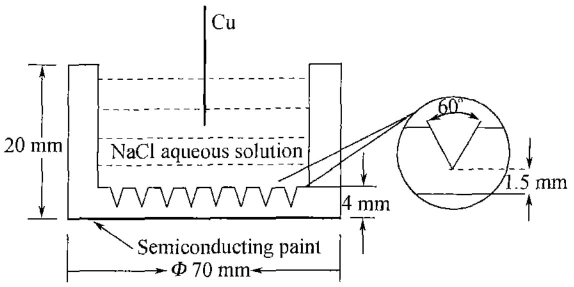


Fig. 1 Schematic diagram of the test cell for water tree length measurement
Electrolyte: 1.0 mol/L NaCl aqueous solution; test condition: 5 kV, 1 kHz, 360 h, room temperature

RESULTS AND DISCUSSION

Characterization of in situ Formed NaAA

The *in situ* preparation and polymerization of NaAA were confirmed by FTIR spectra in Figs. 2(a)–(d). AA has characteristic absorption peaks at 1712 cm^{-1} ($\nu_{C=O}$) and 1640 cm^{-1} ($\nu_{C=C}$)[22]. When the $-\overset{\overset{\displaystyle O}{\|}}{C}-OH$ group is transformed into $-C\langle^{O}_{O^-}$, its characteristic absorption peak appears at 1570 cm^{-1} (ν_{COO^-})[22]. This spectral change reveals that the $-COOH$ in AA (cf. Fig. 2a) has transformed into $-COO^-$ in LLDPE/NaAA (cf. Fig. 2c) when AA and NaOH were mixed in a molar ratio of 1:1 in LLDPE. It is worth noticing that in the FTIR spectrum of methanol extracted LLDPE/NaAA (cf. Fig. 2d), a small absorption peak at 1712 cm^{-1}, which is assigned to $\nu_{C=O}$ of $-COOH$ still remains, indicating that graft polymerization of NaAA on LLDPE and homo polymerization of NaAA occur simultaneously.

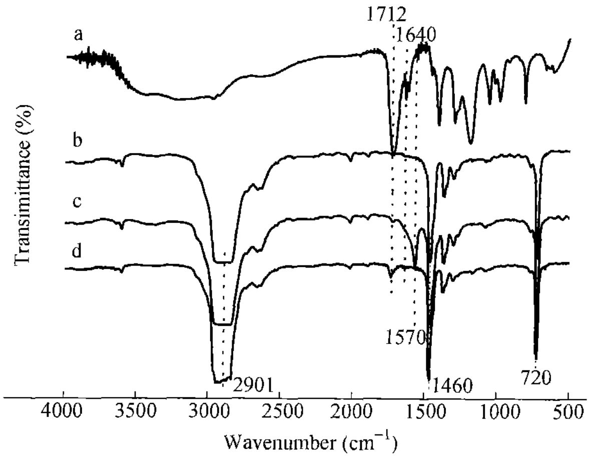


Fig. 2 FTIR spectra of (a) AA, (b) LLDPE, (c) a LLDPE/NaAA (100/1.0) compound (extracted with distilled water) and (d) a LLDPE/NaAA (100/1.0) compound (extracted with methanol)

Figure 3 shows the effect of DCP content on the polymerization conversion ratio of NaAA in the LLDPE/NaAA compounds. As expected, the polymerization conversion ratio of NaAA increases markedly until the DCP content reaches 0.1 phr (part of reagent per hundred parts of LLDPE); thereafter, the polymerization conversion ratio of NaAA increases slowly with the increase of DCP content. Since NaAA can be dissolved in water, it is necessary to use at least 0.1 phr of DCP in order to increase the polymerization conversion ratio of NaAA and to decrease the loss of NaAA.

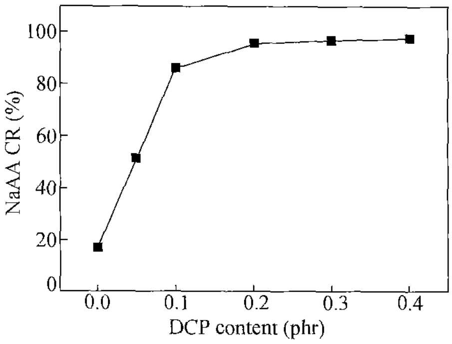


Fig. 3 Effect of DCP content on the polymerization conversion ratio of NaAA in the LLDPE/NaAA (100/1.0) compounds

However, crosslinking of LLDPE can be initiated by DCP at elevated temperatures; therefore, the effect of DCP content on gel content of the LLDPE/NaAA compounds is worth discussing. As is shown in Fig. 4, gel content of the compounds is relatively low when the DCP content does not exceed 0.1 phr, and then it would increase markedly with the DCP content. Because the gel content of the compounds would deteriorate the thermal and mechanical properties, and could accelerate the degradation of insulation[23]; therefore, a DCP content of 0.1 phr is adequate to be used in this study.

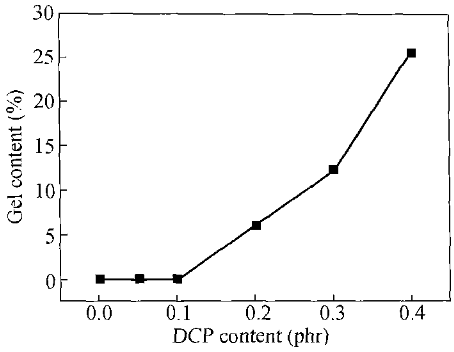


Fig. 4 Effect of DCP content on the gel content of LLDPE/NaAA (100/1.0) compounds

Hydrophilicity

The effect of the NaAA content on the water absorption ratio (WAR) of the LLDPE/NaAA compounds is clearly illustrated in Fig. 5. The WAR of LLDPE/NaAA compounds increases with the increase of NaAA content, indicating that the hydrophilicity of the compounds has been improved with the increase of NaAA content. Note that the —COONa groups in the compounds are hydrophilic in nature; and the —COONa groups in the sodium polyacrylate (PNaAA consisting of both grafted PNaAA and homo sodium polyacrylate) molecular chains are partially ionized when they are in contact with water[24]. Therefore, increasing the NaAA content results in an increase in the hydrophilic —COONa groups, which consequently leads to an increase of the water absorption ratio.

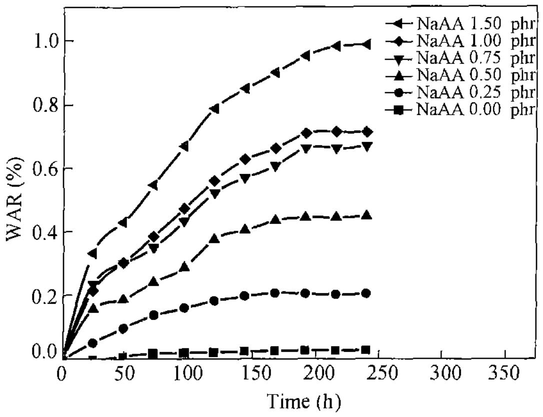


Fig. 5 Effect of NaAA content on the water absorption ratio of LLDPE/NaAA compounds

Water Treeing

Figure 6 shows the results of water treeing tests of the LLDPE/NaAA compounds. As is shown, the water tree length (WTL) reached a minimum value of 167 μm at a NaAA content of 0.5 phr, indicating that the water tree resistance of the compounds has been improved. According to the propagation mechanisms of water trees [6, 8, 20, 25], water and Maxwell stresses are the most crucial factors of water trees formation. Polyethylene is a hydrophobic non-polar polymer, so its electro-mechanical condensation of water occurs easily in electrically irregular points (voids, contaminants, *etc.*). Due to the inhomogeneous electric field of the faults in the polymer, water drops tend to move to the points of highest stresses. Then, crazing of the polymer would take place under the action of alternating Maxwell stresses and compressive stresses of the diffusion of water. Finally, with the growth of the craze, the craze-like structure of polymer would lead to the formation of the tree-like microchannels or tubes, which are water trees[26, 27]. Therefore, when the hydrophilic —COONa groups of the compounds are in contact with water, the water molecules would be prevented from diffusing in the compounds because of the strong interaction between the —COONa groups and water molecules. On the other hand, the condensation of water in electrically irregular points would also be prevented because the —COONa groups are scattered throughout the compounds. Finally, the propagation of water trees is retarded with the formation of NaAA in the LLDPE/NaAA compounds. However, with the increase of the NaAA content, the water concentration of the areas around the —COONa groups would increase gradually and these areas become new defects. Accordingly, it can be concluded that an extra amount of NaAA could result in an increase of WTL.

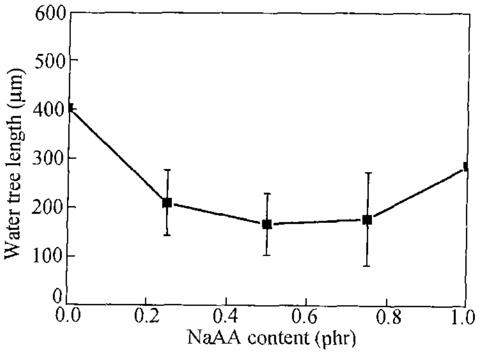


Fig. 6 Effect of NaAA content on the water tree length of LLDPE/NaAA compounds

Mechanical Properties

Figures 7 and 8 depict the effects of DCP and NaAA content on the mechanical properties of LLDPE/NaAA compounds. As is shown in Fig. 7, the DCP content has a slight effect on the mechanical properties of LLDPE/NaAA compounds until the DCP content reaches 0.1 phr. Beyond this value, the tensile strength decreased slightly while the elongation at break decreased markedly with the DCP content. The changes of mechanical properties for LLDPE/NaAA compounds are caused by the combined action of ionic crosslinks in the compounds and crosslinks of LLDPE. The ionic crosslinks formed by grafting of NaAA onto LLDPE chains can increase the tensile strength[22]; however, the gel content of the crosslinks of LLDPE lowers the tensile strength of the compounds. The decreasing of the elongation at break is mainly due to both the ionic crosslinks and the gel content in the compounds.

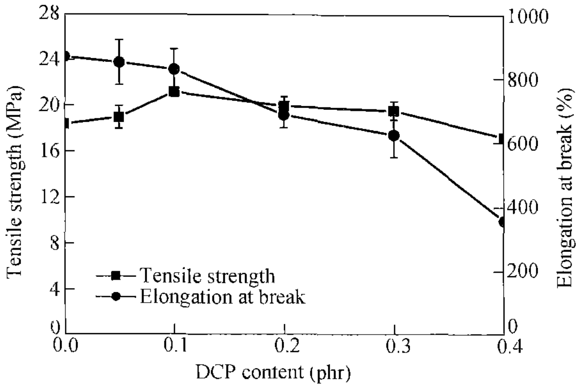


Fig. 7 Effect of DCP content on the mechanical properties of LLDPE/NaAA (100/1.0) compounds

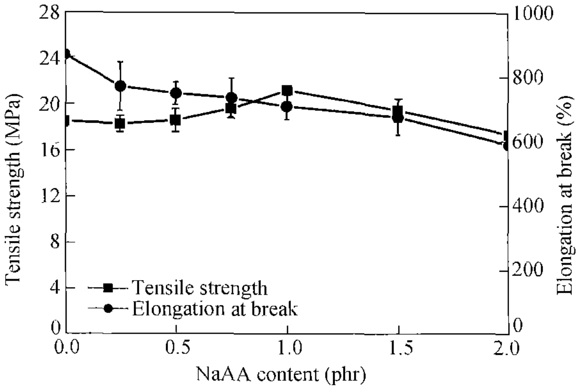


Fig. 8 Effect of NaAA content on the mechanical properties of LLDPE/NaAA compounds

The effect of NaAA content on the mechanical properties of LLDPE/NaAA compounds is exhibited in Fig. 8. The NaAA content has only a slight effect on the mechanical properties because the NaAA content is quite low. Although the ionic crosslinks can increase the tensile strength, yet the presence of insignificant amounts of unreacted NaAA and polysodium acrylate could decrease the tensile strength with the increase of NaAA content because of their poor dispersive capacity in the compounds. In addition, the ionic crosslinks, the unreacted NaAA and homo sodium polyacrylate (PNaAA) result in the decrease of the elongation at break with the increase of the NaAA content.

Dielectric Properties

The dielectric properties of LLDPE/NaAA compounds containing various amounts of DCP and NaAA were measured, and the results are shown in Tables 2 and 3. It can be seen that the DCP content has little influence on the dielectric properties of LLDPE/NaAA compounds until the content reaches 0.1 phr, and then the dielectric constant and breakdown strength decrease slightly with the increase of DCP content. The changes in dielectric properties are mainly due to the gel content in the compounds (Fig. 4), because crosslink can decrease the dielectric constant and the microgel can accelerate the degradation of insulation[23, 28]. From Table 3, we can observe that both the volume resistivity and breakdown strength decrease slightly while both the dissipation factor and dielectric constant increased slightly with the increase of NaAA content. It is well known that the polar impurity can definitely affect the dielectric properties by lowering both the volume resistivity and breakdown strength but increasing both the dissipation factor and dielectric constant increase[28, 29]. Although the unreacted NaAA and PNaAA in the LLDPE/NaAA compounds are regarded as polar impurities, the NaAA content has only a minor effect on the dielectric properties of the compounds because of the small amounts of NaAA until the content reaches 0.75 phr. When it exceeds 0.75 phr, the dissipation factor and dielectric constant increase markedly. Thus, the LLDPE/NaAA compounds still possess good dielectric properties when the DCP content does not exceed 0.1 phr, and the NaAA content does not exceed 0.75 phr.

Table 2. Effect of DCP content on the electrical properties of LLDPE/NaAA (100/1.0) compounds

DCP (phr)	0	0.05	0.1	0.2	0.3	0.4
Volume resistivity (Ω • cm)	1.6 (±0.3) × 10^{17}	1.3 (±0.2) × 10^{17}	1.3 (±0.5) × 10^{17}	1.3 (±0.4) × 10^{17}	1.2 (±0.2) × 10^{17}	1.1 (±0.3) × 10^{17}
Dissipation factor	0.0006 (±0.00004)	0.0006 (±0.00004)	0.0007 (±0.00006)	0.0007 (±0.00006)	0.0006 (±0.00008)	0.0006 (±0.00005)
Dielectric constant	2.26 (±0.03)	2.28 (±0.01)	2.27 (±0.02)	2.25 (±0.02)	2.24 (±0.04)	2.23 (±0.03)
Breakdown strength (kV/mm)	47 (±2)	46 (±2)	46 (±2)	41 (±4)	40 (±2)	36 (±3)

Table 3. Effect of NaAA content on the mechanical properties of LLDPE/NaAA compounds

NaAA (phr)	0	0.25	0.5	0.75	1.0	1.5
Volume resistivity (Ω • cm)	1.6 (±0.3) × 10^{17}	1.4 (±0.6) × 10^{17}	1.3 (±0.4) × 10^{17}	1.3 (±0.4) × 10^{17}	1.3 (±0.5) × 10^{17}	1.1 (±0.2) × 10^{17}
Dissipation factor	0.0001 (±0.00001)	0.0004 (±0.00003)	0.0004 (±0.00003)	0.0006 (±0.0001)	0.0007 (±0.00006)	0.0015 (±0.0004)
Dielectric constant	2.18 (±0.02)	2.22 (±0.03)	2.24 (±0.03)	2.27 (±0.01)	2.27 (±0.02)	2.28 (±0.02)
Breakdown strength (kV/mm)	52 (±1)	47 (±3)	46 (±2)	48 (±3)	46 (±2)	46 (±3)

CONCLUSIONS

The water tree resistance of LLDPE can be improved by the sodium acrylate *in situ* polymerization. When the DCP and NaAA contents are 0.1 and 0.5 phr, respectively, the water tree resistance of LLDPE/NaAA compound can be improved. Moreover, the compound still enjoys excellent mechanical and dielectric properties. FTIR spectra have revealed that NaAA, formed by the neutralization of NaOH and AA, can be polymerized. With the increase of the NaAA content, the hydrophilicity increases gradually in the LLDPE/NaAA compounds.

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