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## Material Properties

## Crosslinked polyethylene/polypyrrole nanocomposites with improved direct current electrical characteristics

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## ABSTRACT

Space charge accumulated in insulation materials could result in local field distortion, accelerate the material aging and threaten the security of high voltage direct current (HVDC) cable. To suppress the space charge accumulation and improve the direct current (DC) electrical characteristics of crosslinked polyethylene insulation materials, crosslinked polyethylene/polypyrrole (XLPE/PPy) nanocomposites were prepared, and the DC conduction and breakdown strength were investigated. The XLPE/PPy nanocomposites were characterized by Fourier transform infrared (FTIR) spectroscopy and X-ray diffraction (XRD). The DC electrical characteristics measurement results showed that the introduction of nano-polypyrrole (nano-PPy) could reduce the DC electrical conduction current density of XLPE/PPy nanocomposites, improve the space charge distribution but not affect the crosslinking reaction and crystallinity of XLPE. While assuring excellent electrical conduction and space charge distribution characteristics, there was an optimum nano-PPy content to reduce the DC breakdown strength of XLPE/PPy nanocomposites insignificantly.

## 1. Introduction

Polymer nanocomposites have received special attention due to the improved direct current (DC) dielectric properties for high voltage direct current (HVDC) cable insulation materials. Space charge accumulated in insulation materials could result in local field distortion, accelerate the material aging and threaten the security of HVDC cable [1]. Some inorganic nanofillers such as MgO, SiO<sub>2</sub>, ZnO, TiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, BaSrTiO<sub>3</sub>, carbon black and graphene are introduced into the polymer matrix to suppress space charge accumulation and inhibit field distortion in insulation materials [2–9]. For additional improvement, coupling agents are employed to modify the nanofillers. Nano-Al<sub>2</sub>O<sub>3</sub> particles modified with vinyl silane coupling could suppress space charge accumulation more effectively and present better breakdown strength when compared with low density polyethylene (LDPE)/Al<sub>2</sub>O<sub>3</sub> [10]. SiO<sub>2</sub> nanoparticles surface modified with titanate and silane coupling agent could introduce more and deeper trap sites at the interface due to the better dispersion of SiO<sub>2</sub> nanoparticles within crosslinked polyethylene (XLPE), and effectively improve DC conductivity and DC

breakdown strength and suppress the injection and movement of space charge [11,12]. Dimethyloctylsilane coupling agent could improve the compatibility between TiO<sub>2</sub> nanoparticles and XLPE matrix to reduce the agglomeration of TiO<sub>2</sub> nanoparticles. The deep traps introduced by dimethyloctylsilane modified TiO<sub>2</sub> nanoparticles could make a large number of charges injected from the electrodes be fixed, forming homocharge near the electrodes to reduce the interfacial electric field and suppress the electrons and holes injected from the electrodes further [13].

The influence of organic nanofillers on the DC dielectric properties of polyethylene (PE) has been rarely studied. In our previous work, the introduction of polyaniline (PANI) nanofibers reduced the DC conductivity of LDPE and weakened the DC breakdown strength, but could not suppress space charge accumulation when the nanofibers content was 0.6, 2.4 and 4.8 phr [14]. The introduction of a small quantity of polypyrrole (PPy) nanobowls could considerably improve the space charge distribution and reduce the DC conductivity of LDPE significantly in the whole measurement temperature range, but not decrease the DC breakdown strength [15]. However, van der Waals force

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between PE molecules weakens with temperature and PE is prone to deform. After crosslinking, the crosslink bonds replace the van der Waals force and the network structure is formed between the molecules, which gives XLPE better thermal and chemical stability, and insulating and mechanical performance. Hence, alternating and direct current plastic insulation cables mainly adopt XLPE as the main insulation material. However, the crosslinking by-products in XLPE are hard to remove completely and will affect the space charge distribution, DC conductivity and breakdown strength. In this paper, nano-poly-pyrrole (nano-PPy) was prepared and doped in crosslinkable PE by melt blending, and XLPE/PPy nanocomposites were obtained after crosslinking. The nanocomposites demonstrated excellent DC dielectric properties with low amount of nano-PPy doping, and the crosslinking degree of the nanocomposites was almost unaffected by the doping of nano-PPy.

## 2. Experimental

### 2.1. Materials

Ferric chloride hexahydrate ( $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ), cetyltrimethylammonium bromide (CTAB), methylene orange and ethanol were supplied by Tianjin Guangfu Fine Chemical Research Institute, China. Pyrrole monomer was purchased from Aldrich and stored at  $-4^\circ\text{C}$  before use. All the above chemicals were analytical grade and used as received without further purification. Dicumyl peroxide (DCP) was obtained from Shanghai Gaoqiao Petroleum Co. Ltd., China. Low density polyethylene (LDPE, LD200BW) pellets with a density of  $0.922\text{ g/cm}^3$  and a Melt Flow Index (MFI) of  $2.0\text{ g/10 min}$  were supplied by Beijing Yanshan Petroleum Co. Ltd., China.

### 2.2. Preparation of nano-PPy and XLPE/PPy nanocomposites

Nano-PPy was synthesized via in situ chemical oxidative polymerization directed by self-degradable templates.  $0.324\text{ g FeCl}_3 \cdot 6\text{H}_2\text{O}$  was dissolved in  $40\text{ mL}$  of  $5\text{ mM}$  methyl orange deionized water solution at room temperature. Then,  $0.058\text{ g CTAB}$  and  $115\text{ }\mu\text{L}$  pyrrole monomer were added sequentially into the above solution, and the reaction mixture was stirred magnetically at room temperature for  $30\text{ h}$ . The black PPy precipitate was centrifuged, washed with deionized water and ethanol several times, and finally dried in a vacuum oven at  $50^\circ\text{C}$ .

The as-prepared nano-PPy was dispersed in ethanol solution with ultrasonication. LDPE was added in a torque rheometer at  $110^\circ\text{C}$  for melting, and then the nano-PPy suspension was added gradually. After the blend of LDPE and nano-PPy continued to be mixed for about  $30\text{ min}$  at  $110^\circ\text{C}$  and the ethanol and  $\text{H}_2\text{O}$  were evaporated absolutely, LDPE/PPy nanocomposites containing  $0.2$ ,  $0.5$  or  $1.0\text{ phr}$  (parts per hundreds by weight) nano-PPy were obtained.

A certain amount of LDPE or LDPE/PPy nanocomposites was put into the mixer at  $110^\circ\text{C}$ . After melting,  $0.3\text{ phr DCP}$  was added into the mixer and blended for about  $3\text{ min}$ . The obtained crosslinkable LDPE or LDPE/PPy nanocomposites were hot compression moulded using the mouldings with different shapes and thicknesses at  $110^\circ\text{C}$  under a pressure of about  $15\text{ MPa}$  and then crosslinked under the same pressure at  $175^\circ\text{C}$  for  $30\text{ min}$ . XLPE, XLPE/PPy-0.2 XLPE/PPy-0.5 and XLPE/PPy-1.0 nanocomposite specimens for different tests were obtained, indicating the nanocomposites containing  $0$ ,  $0.2$ ,  $0.5$  and  $1.0\text{ phr}$  nano-PPy, respectively. Aluminum electrode was deposited on both sides of the tested specimens by vacuum coating for the measurement of electrical characteristics.

### 2.3. Characterization

The morphology of nano-PPy was characterized by a Hitachi SU8020 scanning electron microscopy (SEM). Fourier transform

infrared (FTIR) spectra of nano-PPy and XLPE/PPy nanocomposites were recorded on a SHIMADZU IR Pres-tige-21 spectrometer with the wavenumber of  $4000\text{--}400\text{ cm}^{-1}$  and a resolution of  $2\text{ cm}^{-1}$ . For the nano-PPy, a potassium bromide (KBr) wafer was used, and the weight percentage of nano-PPy in KBr was about  $0.5\text{--}1.0\%$ . For the XLPE/PPy nanocomposites, the specimen with average thickness of  $100\text{ }\mu\text{m}$  was used for the measurement. The structures of nano-PPy and XLPE/PPy nanocomposites were characterized by a D8 Advance X-ray diffraction (XRD) equipment using  $\text{CuK}\alpha$  radiation under  $40\text{ kV}$  and  $40\text{ mA}$  in the scan range between  $10^\circ$  and  $100^\circ$ .

### 2.4. Crosslinking degree measurement

The gel content is usually adopted to characterize the crosslinking degree of XLPE. In this paper, the gel content of XLPE/PPy nanocomposites was measured by the Soxhlet extraction method. First, a specimen of about  $0.300 \pm 0.015\text{ g}$  was weighed accurately as  $M_1$  with precision of  $0.001\text{ g}$ , cut into  $1 \times 1\text{ cm}^2$  pieces, put in a filter holder and refluxed for about  $12\text{ h}$  in boiling xylene solution at about  $140^\circ\text{C}$ . During refluxing, the non-crosslinked fraction in the XLPE composites was supposed to be dissolved fully in the xylene and the remaining insoluble fraction was considered to be crosslinked elastomer matrix. The insoluble residue was dried at  $150^\circ\text{C}$  for  $24\text{ h}$  in a vacuum oven followed by the determination of its net weight ( $M_2$ ). The gel content was calculated on the basis of the following equation [16]:

$$\text{Gel Content (\%)} = M_2/M_1 \times 100$$

### 2.5. Electrical performance measurement

The space charge distribution measurement of the specimen with average thickness of  $300\text{ }\mu\text{m}$  was performed in a pulse electro-acoustic (PEA) system. Silicone oil was used as acoustic coupling to ensure good acoustic transmission between the specimen and the electrode. Calibration was conducted at a DC field of  $3\text{ kV/mm}$  for  $5\text{ min}$  to minimize the influence on space charge accumulation. Then, a DC electric field of  $40\text{ kV/mm}$  was applied to the specimen for  $40\text{ min}$  at room temperature and removed. The measurement signals both in polarization and depolarization process were recorded over time, and the data were processed using a calibration trace and a deconvolution technique to restore the original signal.

DC electrical conduction current measurement of the specimen was performed with a three-electrode system for relatively uniform electric field under the measurement electrodes. The three-electrode system consisted of the measurement electrode of  $50\text{ mm}$  in diameter, guard electrode of  $54\text{ mm}$  in inner diameter and  $74\text{ mm}$  in outer diameter, and high voltage electrode of  $74\text{ mm}$  in diameter. The test system and the specimen with average thickness of  $200\text{ }\mu\text{m}$  were placed in an oven, which could eliminate the interference of external signals and control the measurement temperature. Quasi-stationary current under different DC electric fields between  $1$  and  $50\text{ kV/mm}$  at the measurement temperatures of  $30$ ,  $50$  and  $70^\circ\text{C}$  were recorded using EST122 picoammeter with a precision of up to  $10^{-14}\text{ A}$ .

The nano-PPy powder was compressed into a pellet with a thickness of about  $300\text{ }\mu\text{m}$ . The electrical conductivity of the nano-PPy pellet was determined by a four-point probe method at room temperature.

The DC breakdown test was conducted by placing the test specimen with average thickness of  $100\text{ }\mu\text{m}$  between two opposing cylindrical electrodes immersed in transformer oil to prevent surface flashover and control the temperature. A DC voltage with a ramp rate of  $600\text{ V/s}$  was applied until the specimen failed. Ten breakdown tests were performed on each type material and the resulting breakdown data were statistically treated by using two-parameter Weibull statistical distribution method [17].

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