Study on the Regulation Mechanism of Octa-aminophenyl POSS on the Electrical Properties of Polypropylene

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Abstract. Elevated temperatures significantly contribute to the deterioration of electrical performance in polymeric insulation under HVDC conditions. To enhance the power transmission capacity, it's crucial for new cable designs to incorporate insulation materials with superior high-temperature resistance, involving heightened breakdown strength, minimized space charge accumulation, and reduced conductivity. In this study, a small quantity (~1.0 phr) of Octa-aminophenyl POSS (OaPOSS), a type of supernano, is blended into polypropylene (PP) to create OaPOSS/PP nanocomposites with upgraded electrical properties. Through thermally stimulated depolarization current (TSDC) analysis, it was clear that the incorporation of OaPOSS enhanced both the trap level and the density of deep traps within the PP matrix. The functionality of OaPOSS is elucidated by examining both the interface between OaPOSS and pristine PP, and the role of the phenyl group in isolated OaPOSS particles. Ultimately, the introduction of OaPOSS presents a novel approach to adjusting the electrical characteristics of pure PP, paving the way for the design of non-cross-linked, eco-friendly thermoplastic insulation materials tailored for HVDC cable applications with exceptional electrical performance.

Keywords: Nanocomposite; Polypropylene; Breakdown strength; Space charge accumulation; Thermally stimulated depolarization current; Trap

1 Introduction

A comparison of PP with LDPE and XLPE has revealed significant advantages in electrical performance exhibited by PP. These advantages make PP a highly promising candidate for use as an electrical insulation material [1]. Also, the manufacture of PP could avoid the pollutants released from the crosslinking and degassing process of XLPE, including phenol, acetophenone, and cumyl alcohol [2]. Uncrosslinked PP is a thermoplastic material that exhibits clear advantages over XLPE with regard to environmental protection, owing to its straightforward recycling properties. The recycling process of PP is uncomplicated, facilitates the recycling of resources and environmental protection, and is a more sustainable material option [3-4]. Additionally, due to its excellent

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mechanical properties under high temperatures, PP could be more suitable for the HVDC cable insulation under high temperatures. Therefore, PP has a large potential to develop a nenew-generationable insulation [5]. Whilst polypropylene (PP) has been shown to exhibit superior performance in comparison to both linear low-density polyethylene (LDPE) and high-density polyethylene (HDPE) when subjected to elevated temperatures, it is nevertheless the case that such conditions result in a compromise to its electrical properties. This is particularly evident in terms of a reduction in breakdown strength and an increase in space charge accumulation[6]. In order to ensure the stability of PP in high-temperature environments, it is essential to identify methods by which to enhance its electrical properties, thus counteracting the deleterious effects caused by high temperatures.

Different from inorganic nanofillers, POSS has the microstructure of nano-silica cage with the chemical bond of Si-O-Si and the functional side-group [7-9]. Research has demonstrated that decreasing the size of POSS with nano-fillers can enhance the electrical properties of materials more effectively [10]. This finding presents a novel approach to optimising electrical properties through size-based adjustment. As Figure 1 shows, the main cage of OaPOSS is connected with 8 phenyl groups and the diameter of OaPOSS is around 1.5 nm which is much smaller than traditional nanofillers [11]. Therefore, the higher interfacial area with the polymeric chain would be achieved by the addition of OaPOSS nanofillers. However, few research papers focused on the functional groups of POSS for DC insulations.

In this research, PP and OaPOSS nanofillers were selected to manufacture the nanocomposites. A systematic evaluation of the crystallisation degree, microstructure characteristics, and electrical properties of PP and OaPOSS/PP nanocomposites was conducted using SEM observation, TSDC analysis, and DC breakdown strength test. The objective of this study was to provide a theoretical foundation for enhancing material properties and facilitating practical applications.

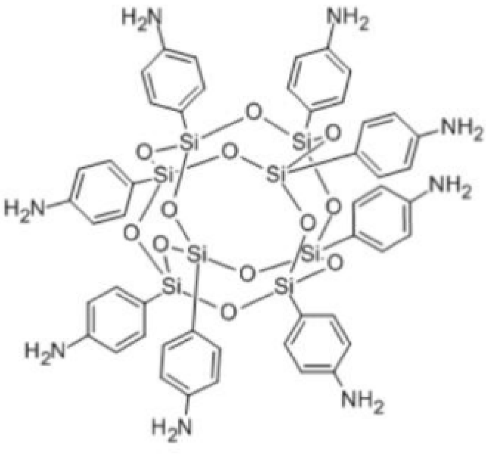

Fig. 1. The schematic diagram of OaPOSS

2 Materials and Experiments

2.1 Materials

The matrix material utilized was iPP with a density of 0.92 g/cm^3, and the nanofiller was OaPOSS, density 1.25 g/cm^3, both sourced from Aladdin Industrial Inc., China. Xylene, also procured from the same supplier, served as the solvent.

Initially, PP pellets were dissolved in xylene at 120°C. Varying amounts of OaPOSS nanofiller, as detailed in Table 1, were then incorporated into the PP-xylene mixture to ensure uniform dispersion of OaPOSS within the PP. The mixture was subjected to stirring for a period of 12 hours at a speed of 500 RPM at 120°C, with the objective of ensuring uniform mixing. Subsequent to this, the mixture was dried for a duration of 12 hours by a turbine blower operating at the same temperature environment, in order to meet the conditions required by the experiment. Subsequently, film samples of varying thicknesses were produced through compression molding at 200°C for 12 minutes under 15 MPa of pressure. These samples were then cooled to 25°C for 15 minutes under 10 MPa of pressure. For physical and electrical testing, film samples with nominal thicknesses of 100 μm and 200 μm were prepared. Lastly, in order to ensure the stability of the sample and the accuracy of the test results, the following measures have been taken: In order to completely remove impurities and ensure the purity of the material, samples of polypropylene (PP) and its nanocomposites containing different concentrations of OaPOSS (organic-inorganic hybrid polyhedral sesquisiloxane) were carefully placed in a vacuum oven at a pressure of 0.01 PASCAL. These samples are then heated to 80 degrees Celsius and held at this temperature for up to 24 hours to effectively evaporate and remove any volatile impurities or unreacted monomers that may be present. Once the heating process is complete, a precisely controlled cooling method is used to gradually cool the sample to room temperature at a slow rate of 0.1 Kelvin per minute, a step designed to minimise internal stresses or structural defects that may be caused by rapid temperature changes, thereby ensuring that the prepared nanocomposite sample has stable physical and chemical properties.

Table 1. Sample preparation and composition

Sample	OaPOSS (g)	PP (g)
PP	0	100
PP/1.0-phr OaPOSS	1.0	99.0

2.2 Scanning Electron Microscope

FE-SEM was utilised to examine the microstructure of polypropylene nanocomposites and the distribution of nano-fillers, thereby establishing an experimental foundation for the enhancement of material properties. The sample was fractured in liquid nitrogen to produce a brittle section, which was then coated with gold under vacuum conditions and observed under the electron microscope. The electron acceleration voltage was set at 12.0 kV, and the SEM magnification was 10,000. Given the significant influence of nanofiller dispersion on the enhancement of PP's electrical properties, it is crucial to evaluate the dispersion of nanofillers within the polypropylene matrix.

2.3 Thermal Stimulated Current Measurement

The measurements of Thermally Stimulated Depolarization Current (TSDC) were carried out using a wideband dielectric spectrometer (Novocontrol GmbH Concept 40, Germany). The cooling and heating processes were facilitated by liquid nitrogen release and a heating system, respectively. A series of experimental procedures have been meticulously planned and executed to investigate in detail and depth the charge release mechanism of polypropylene and its nanocomposites when heated. These processes cover several key steps: In the early stages of the experiment, important pre-treatment steps were taken to ensure the stability and reliability of the electrical connection. Specifically, the contact area between the sample and the test electrode is carefully and uniformly coated with a thin layer of metal-gold. This approach not only significantly improves the electrical conductivity of the contact interface, but also effectively prevents connection failure caused by oxidation, corrosion, and other factors due to the excellent electrical conductivity and chemical stability of gold. In addition, the uniform coating ensures that the current is distributed evenly across the contact surface, further improving the accuracy and repeatability of the test and providing a solid basis for subsequent electrical performance testing. Next, the samples were polarized under preset temperature and electric field conditions. The samples were then cooled to low temperatures at a constant rate to “fix” the charge state. Short-circuit operation was then used to remove the space charge from the sample surface. Finally, TSDC was measured through a slow heating process to monitor the dynamic transition of charge release. This series of steps provides significant information about the charge storage and release characteristics of polypropylene and its nanocomposites, and provides solid experimental support for the subsequent optimization and practical application of the material.

2.4 DC Breakdown Strength Measurement

The HVDC breakdown test system is a sophisticated setup that includes a high-performance HVDC power supply, a voltage test system, protective resistors, a ball-to-ball electrode structure, a silicone oil immersion environment, and a temperature controller, along with a controllable heating plate. This integrated system guarantees accurate and safe breakdown tests, offering an efficient platform for studying the breakdown strength of polypropylene and its nanocomposites. Prior to the breakdown strength test, the oil temperature was recorded with a thermometer to confirm an experimental temperature of 30°C. A rigorous, efficient, and accurate test procedure has been developed for the evaluation of the breakdown strength characteristics of polypropylene and its nanocomposites. This procedure involves the uniform marking of test points on the film sample, ensuring precise alignment with the electrode shaft, and the application of DC voltage at a constant rate of 1 kV/s until breakdown occurs.

3 Results and Analysis

3.1 Morphology

The dispersion of OaPOSS nanofiller with different contents in PP is shown in Figure 2. In the initial phase of the OaPOSS content, which was set at 1.0 phr, the material exhibited uniform dispersion within PP, with no agglomerates exceeding 200 nm in size. This outcome serves to substantiate the efficacy of the employed preparation and dispersion methodology in the mitigation of agglomeration and degradation processes. Furthermore, it paves the way for further research by establishing a foundation for subsequent studies.

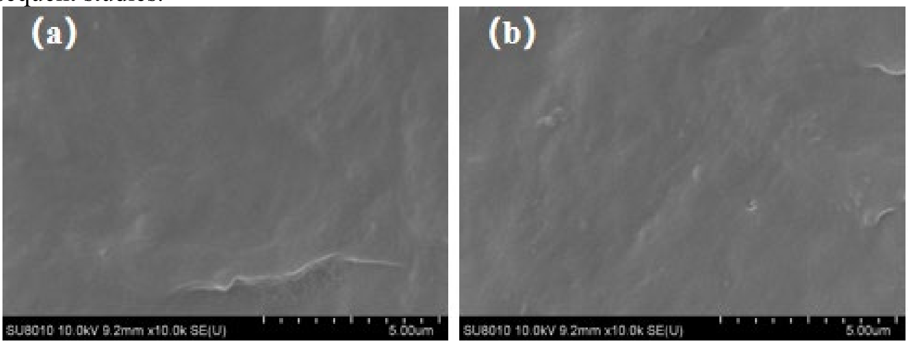

Fig. 2. The dispersion of OaPOSS in (a) pure PP (b) 1.0 phr OaPOSS/PP nanocomposites

3.2 Trapping Charaterisctics

Figure 3 presents the TSDC (Thermally Stimulated Depolarization Current) curves for PP and its nanocomposites. Both PP and its nanocomposites exhibit a peak in thermally stimulated current at -5°C, due to the transformation of polypropylene from a glassy state to a highly elastic state at the glass transition temperature. The peak value of the TSDC curve of PP is observed to be minimal at 64°C, attributable to the reduced density of deep traps. Conversely, OaPOSS/PP nanocomposites exhibit a pronounced peak value of 0.62 pA at 88°C, suggesting that OaPOSS enhances the deep trap density, thereby promoting enhanced charge storage and enhanced stability. This finding lends further support to the ongoing research and development of nano-filler mechanisms and materials.

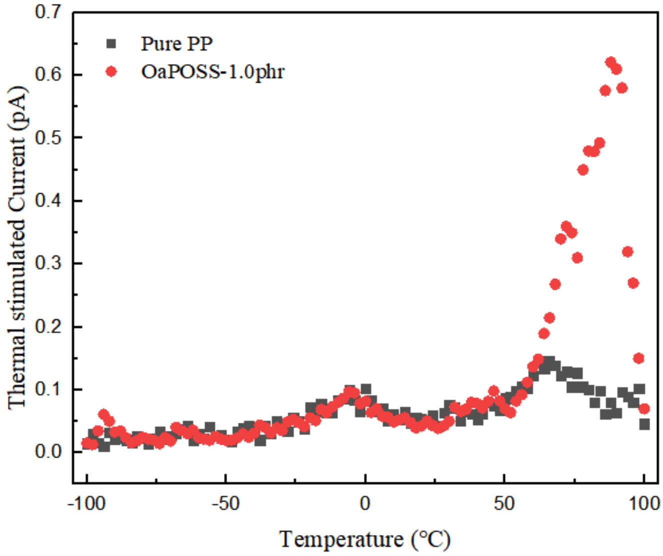

Fig. 3. The trapping level distribution shown in TSDC results

3.3 DC Breakdown Strength

The DC breakdown characteristics of polypropylene (PP) and its nanocomposites can be effectively characterized using the Weibull distribution, which is highly efficient for analyzing the breakdown strength of polymer-insulating materials. This model includes and critical value α and shape parameter β, both derived from fitting experimental data. This approach allows for a quantitative comparison of breakdown characteristics and offers a mathematical foundation for improving material performance and evaluating reliability:

$$P = 1 - \exp\left[-\left(\frac{E}{E_0}\right)^{\beta}\right]$$

Figure 4 illustrates that the incorporation of OaPOSS nanofiller enhances the direct current breakdown strength of polypropylene, increasing it from 442.1 kV/mm to 558.7 kV/mm. In comparison with pure PP, the breakdown strength of OaPOSS/PP nanocomposites exhibited an enhancement of 26.3%. This increase was attributed to the optimisation of the matrix structure by nanoparticles, the influence of charge distribution, and the potential promotion of crystallisation. Firstly, based on TSDC measurements, the enhanced breakdown strength is correlated with an increase in deep traps resulting from the addition of OaPOSS. These deep traps capture injected space charges, creating a potential barrier that restricts charge mobility.

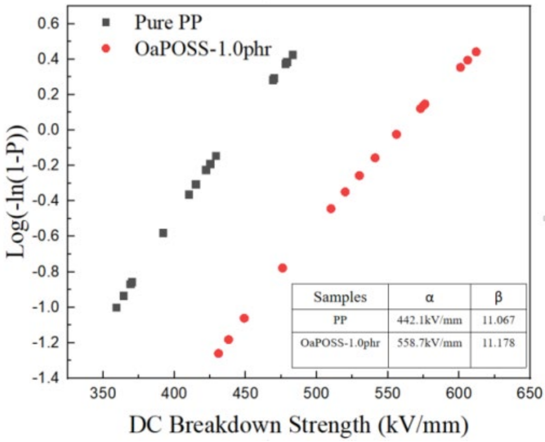

Fig. 4. The results of DC breakdown strength measurements

4 Conclusion

This study demonstrates that incorporating Octa-aminophenyl POSS (OaPOSS) into polypropylene (PP) significantly enhances its electrical properties. Specifically, OaPOSS introduces a high density of deep traps that effectively capture and immobilize charge carriers, leading to a substantial improvement in the electrical properties of PP. These results lay the groundwork for creating sustainable, non-cross-linked thermoplastic insulation materials designed specifically for high-voltage direct current (HVDC) cable applications.

Future investigations are essential to enhance the integration of OaPOSS into polypropylene (PP) and to examine the long-term stability and aging characteristics of OaPOSS/PP nanocomposites. Additionally, the study suggests investigating other nanomaterials with similar functional groups or microstructures to OaPOSS to identify alternative or complementary materials that could provide additional enhancements to the electrical characteristics of polypropylene.

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