

THE INFLUENCE OF ORGANIC ADDITIVES ON THE DIELECTRIC PROPERTIES, FREE VOLUME, AND CRYSTALLIZATION KINETICS OF HPPE

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Abstract. In this study, for the first time, a composition based on high-pressure polyethylene (LDPE) grade 10803-020 has been developed, notable for its exceptionally low additive content (0.05 wt% each) and excellent technological compatibility. The results of experiments investigating the effect of small amounts of phthalic anhydride (PhAn) and phthalic acid (PhAc) additives on the lifetime and dielectric properties ($\text{tg}\delta$, ρ_v) of high pressure polyethylene (HPPE) film grade 108 are presented. It was found that the optimal concentration is 0.05 mass% PhAn + PhAc, which increases the electrical strength by about 50% and significantly improves the dielectric properties compared to the original HPPE. The dielectric characteristics of the loss tangent, specific volume electrical resistivity (ρ_v), and dielectric permittivity (ϵ) were studied. In the optimal HPPE composition, there is a significant increase in specific volume electrical resistivity and a decrease in the loss tangent ($\text{tg}\delta$), indicating a strong correlation between these characteristics. To identify structural changes in the polymer materials before and after the introduction of small additives and exposure to discharges, research was conducted using scanning electron microscopy and dilatometry methods. These methods were used to study the effect of the small additive (PhAn + PhAc) on the crystallization rate and mechanism of high pressure polyethylene. It was found that the introduction of 0.05% by mass of PhAn + PhAc into HPPE influences the increase in polymer crystallization rate and improves polymer processing productivity.

Keywords: HPPE, phthalic acid, lifetime, specific free volume.

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1 Introduction

One of the primary challenges faced by researchers working in the field of high-molecular-weight compounds is the development of new polymer modifications with a predetermined set of properties that remain relatively stable under external influences and that depend on the supramolecular structure. Additionally, the technology for producing new polymer composite materials with enhanced physical, electrical, mechanical, and technological properties requires an understanding of the effects of additives and external factors on molecular-structural changes in polymers (Al-Raei et al., 2021; Slutsker, et.al. 2003; Van der Beek, 2005; Mammadli et al., 2019; Greco,

et al., 2003). In the study (Mehrabova et al., 2024a), the electrical strength of HPPE film and its optimal composition were examined, showing an increase in its electrical strength by approximately 50%. This study investigates the dependence of the loss tangent and specific volume electrical resistivity on temperature, as well as the kinetics of physico-chemical changes under the influence of electrical discharges in air and UV irradiation. The authors of studies (Cubric et al., 2022; Zuev et al., 2011) appropriately note that irradiation can result in the formation of internal voids and gas inclusions, which weaken the dielectric electrical strength ($\mathcal{E}$) and may lead to internal discharges.

Zeynalov et al. (2023) presents the results of an experimental investigation into the temperature-time dependence of the electrical durability of LDPE films and its optimal composition (LDPE + 0.05 wt% PhAn + 0.05 wt% PhAc). It was found that the addition of these components in small amounts (0.05 wt% each) significantly enhances the electrical strength of the material at various temperatures. It was shown that the threshold voltage U_0 corresponds to the activation energy of chemical bonds, indicating that the electrical breakdown of the polymer predominantly occurs along these bonds.

A comprehensive study of the structural properties of HPPE and its optimal modification was conducted to obtain detailed information on the specific mechanisms behind the observed improvement in electrical properties with the addition of low-molecular-weight organic additives. This research involved scanning electron microscopy as well as an investigation into the influence of small additives on the crystallization rate and mechanism of polyethylene. Dilatometry studies of microcomposites made it possible to not only determine the effect of compositional and structural changes on the nature of phase transitions but also to identify potential differences in the crystallization mechanism itself.

The objective of this study is to determine the optimal composition of organic additives that improve the dielectric properties of HPPE while preventing the deterioration of the polymer's physical and mechanical properties and suppressing destructive processes under various external influences. Additionally, the aim is to establish a correlation between the effects of these structure-forming additives on the supramolecular structure and the dynamics of phase transitions and crystallization kinetics in polymer composites.

2 Experimental Methodology

The object of the research was high pressure polyethylene (HPPE) grade 10803-020, and the organic additives used were phthalic anhydride (PhAn) (chemical formula $C_6H_4(CO_2)$) and phthalic acid (PhAc) (chemical formula $C_6H_4(CO_2H_2)$). The additives were introduced into the raw HPPE by mechanical mixing in the melt, a common method due to its simplicity and ease of industrial application, in quantities ranging from 0,01% to 0,1% by mass. Prior to introducing the additives into HPPE, they were dispersed using sieve analysis on a device designed to determine particle size distribution. The particle size was less than $50\mu m$.

The sieve analysis apparatus consists of a vibration drive and a set of sieves. The set of sieves is selected to cover the entire grain size range of the bulk material being tested (5-6 sieves). At the bottom is a sieve tray that captures the final sifted material. The vibration drive imparts vibrating and rolling motion to the sieves installed and secured on it. The sieving duration is about 10 minutes for coarse materials and around 20 minutes for fine materials. The time and vibration intensity are regulated by a timer on the vibration drive. For producing films, a manual electrically heated hydraulic press (model PG-60) was used to hot-press the samples obtained from the injection machine. The calculated amount of micro-additives was loaded into a pre-prepared flat mold with a gasket thickness of 0,1 mm. To prevent adhesion, fluoroplastic film (foil) was used. The mold was heated to 140-150°S. Upon reaching the required temperature, the mold was compressed under a pressure of 150 atm. After cooling the mold, the films were extracted. Samples with a thickness of 50 – 60 μm were produced from these films for measuring



Figure 1: Scanning electron microscope (SEM) model JEOL JSM-6610LV-x-Max for research of HPPE+MWCNT composites.

physical, mechanical, electrical, and thermal properties.

The homogeneity of the film was assessed by measuring its thickness across the entire surface area. The film thickness was determined using an N3B-2 optical thickness gauge as well as a micrometer. The reported thickness value represents the arithmetic mean of 10 measurements.

Repeated experimental studies of the mechanical, dielectric, and optical properties, as well as the structural features of the developed composition, confirm the uniform dispersion of PhAn + PhAc within the LDPE matrix.

The measurement of the time to electrical breakdown (lifetime) of the investigated polymer films under an electric field with a frequency of 50 Hz was carried out using a breakdown test setup equipped with a specially designed cell comprising two steel disc electrodes with diameters of 30 mm and 10 mm, respectively.

The measurement of the lifetime (time to breakdown) of the studied polymer films under the influence of an electric field with a frequency of 50 Hz was carried out according to the methodology described in (Zeynalov et al., 2023). The specific volume electrical resistivity (ρ_V) of the samples was measured using a teraohmmeter E6-13A in the temperature range of 300-500 K, with a linear heating rate of 3 degrees per minute. The measurement of the dielectric loss tangent was conducted using an alternating current bridge model P589 at a frequency of 1 kHz, also in the temperature range of 300-500 K.

A qualitative assessment of changes in the structure of HPPE films with the introduction of small additives was performed by determining the morphology of supramolecular formations using scanning electron microscopy. Electron microscope images of the surface were taken using the JEOL JSM-6610LV-x-Max scanning microscope (Fig. 1).

Dilatometrical measurements were carried out using the IIRT-1 instrument under a load of 5.3 kgf in the temperature range from 180°C to room temperature, with a measurement accuracy of $\pm 0.5\%$.

The melt flow index (MFI) of the samples was determined using a capillary rheometer of the INSTR brand (Italy), model MELT FLOW TESTER CEAST MFSO, at a temperature of 180 °C under a load of 5 kgf. The average relative experimental error did not exceed 5 %.

3 Experimental Results and Discussion

The results of experiments investigating the effect of small amounts of organic additives PhAn + PhAc on the lifetime τ_E and electrical properties of high pressure polyethylene (HPPE) films are presented. It is shown that the dependence of $\lg \tau_E = f(E)$ for all modified HPPE samples generally exhibits a linear character and is described by a well-known formula

$$\tau_E = B_{\text{exp}}(-\beta E),$$

where the parameters β and B depend on the nature of the polymer and the testing temperature (Nuriyev et al., 2018). Experimental results (Tanaka et al., 2004) showed that the introduction

of the optimal content of the PhAn + PhAc additive into HPPE leads to an increase in its electrical strength from $14 \cdot 10^7$ to $25 \cdot 10^7$ V/m, which represents an approximate 50% increase.

It has been determined that the content of 0,05 mass% PhAn + PhAc is optimal, as it provides the highest stability of electrical properties compared to both the original HPPE and HPPE with other additive concentrations. For example, the introduction of the optimal PhAn + PhAc content into HPPE leads to an increase in specific volume electrical resistivity (ρ_V). Moreover, these additives contribute to maintaining an elevated level of specific volume electrical resistivity across the entire temperature range (20-110°S) during testing. Additionally, the developed HPPE modification exhibits a relatively stable value of the dielectric loss tangent ($\text{tg}\delta$).

The results of experimental studies on the electrical properties of HPPE and its compositions are presented in the Table 1.

Table 1: Electrical characteristics of HPPE film and its developed compositions.

Polymer Materials	Characteristics			
	$E \cdot 10^{-7} \text{ V} \cdot \text{m}^{-1}$	$\text{tg}\delta, f = 1\text{kHz}$	$\rho_V, \text{Om} \cdot \text{m}$	ε
HPPE (original)	18	$5 \cdot 10^{-4}$	$1 \cdot 10^{15}$	2,3
HPPE +0,03 mass % PhAn +0,03 mass % PhAc	17	$4,5 \cdot 10^{-4}$	$1 \cdot 10^{15}$	2,3
HPPE +0,03 mass % PhAn +0,05 mass % PhAc	25	$2,7 \cdot 10^{-4}$	$1 \cdot 10^{17}$	2,2
HPPE +0,07 mass % PhAn +0,07 mass % PhAc	17	$4,5 \cdot 10^{-4}$	$1 \cdot 10^{15}$	2,4
HPPE +0,1 mass % PhAn +0,1 mass % PhAc	15	$5,2 \cdot 10^{-4}$	$1 \cdot 10^{14}$	2,4

Thus, the observed increases in electrical strength and the stability of the modified HPPE can be attributed to the structuring characteristics of the specified additive, which ensures dense packing of macromolecules during film formation.

The study of HPPE and its composition using scanning electron microscopy provided a deeper understanding of the supramolecular structure of the samples under investigation. Figures 2 and 3 present micrographs of the original HPPE and HPPE containing 0,05% by mass of PhAn + PhAc.

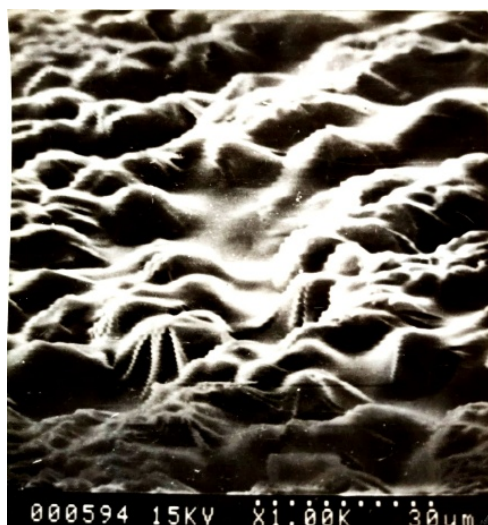


Figure 2: Micrographs of the film HPPE (original)

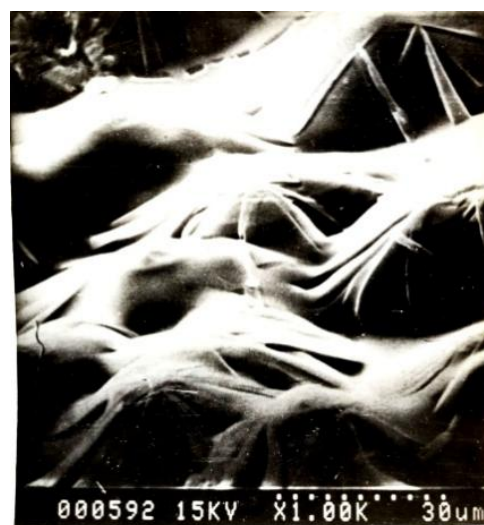


Figure 3: Micrographs of the film HPPE + 0,05 mass % PhAn +0,05 mass % PhAc

It can be seen that with the introduction of 0,05 mass% PhAn + 0,05 mass% PhAc into HPPE, the growth of larger structural formations is observed, with a clearly defined higher continuity or organization of packing.

This is also confirmed by the experimental results obtained from X-ray diffraction studies, which are presented in Table 2.

Table 2: X-ray diffraction results of LDPE and its optimal modification

Material	Crystallite size, Å	Degree of crystallinity, %
LDPE 10803-020 (initial)	126	40
LDPE + 0.05 wt% PI + 0.05 wt% PC	136	41

It is easy to notice that the resulting structural elements take the form of lamellae, interwoven within a specific microvolume of the polymer, while at the same time merging into neighboring structural elements of similar construction. Based on the micrographs and a comparative analysis of the structure, we are justified in asserting that the micro-additives PhAn + PhAc have the ability to accelerate the process of structure formation on the supramolecular surface. These additives, by embedding primarily into the amorphous phase of the polymer, create centers of structure formation, whose elements, intertwined with polymer crystallites, form a single ordered structure with a high degree of packing (Zaharescu et al., 2020; Ray et al., 2003; Pandey et al., 2020; Hedir et al., 2019).

Thus, the observed increase in electrical strength and the stability of the modified HPPE can be attributed to the structuring characteristics of the specified additives, which ensure dense macromolecule packing during film formation. Therefore, when studying the influence of modifying additives on the polymer, it was necessary to determine not only the nature of the additive's effect on HPPE but also the mechanism behind this phenomenon. In this regard, the influence of small amounts of PhAn + PhAc on the rate and mechanism of polyethylene crystallization is of particular interest.

The most comprehensive information about certain physical states of polymers can be obtained through dilatometric measurements, i.e., by examining the dependence of specific volume on temperature. Dilatometric studies of macromolecules allow not only the determination of the influence of structural changes on the nature of phase transitions but also the identification of potential differences in the crystallization mechanism itself.

Figure 4 shows the curves of the dependence of the specific volume of HPPE samples and its modification on temperature.

As seen in Figure 4, the introduction of 0,05 mass% of the structure-forming additives significantly narrows the melting temperature range and reduces the specific volume at a given temperature. Unlike low-molecular-weight crystals, polymer crystals do not melt at a specific point but within a certain temperature range, with the midpoint being considered the experimental melting temperature.

Comparing the curves for the original HPPE and HPPE + 0,05 mass% PhAn + 0,05 mass% PhAc, crystallization and the first-order phase transition occur at the same temperature range of 88-90°S. It is evident that the particles of the investigated compounds influence the orientation of HPPE macrochains, leading to the formation of heterogeneous crystallization centers. Indeed, at the crystallization temperature of HPPE containing the structure-forming additives, the first-order phase transition occurs within a narrow temperature range. As the polymer transitions into a solid state, i.e., at temperatures below its crystallization point, the specific volume (V_y) of HPPE + 0,05 mass% PhAn + 0,05 mass% PhAc decreases sharply.

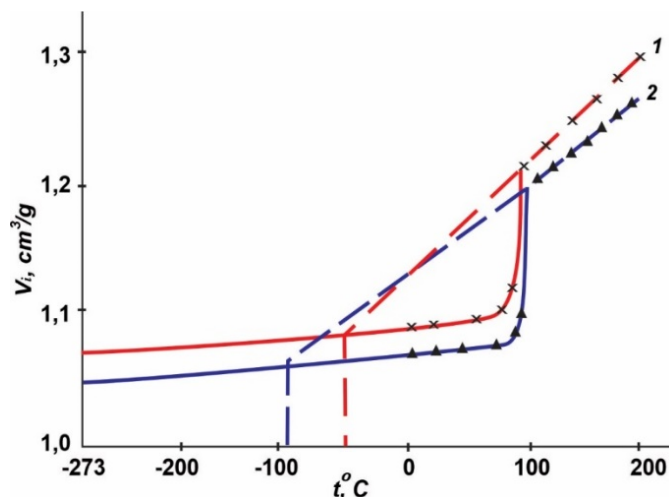


Figure 4: Dependence of specific volume on temperature; 1- HPPE (original); 2- HPPE +0,05 mass % PhAn+0,05 mass % PhAc.

The structural characteristics of various polymers, potential phase transitions, and the entire set of relaxation properties are largely determined by the “free” volume and its dependence on temperature.

In Figure 2, an attempt is shown to extrapolate the dependence of $V_i=f(T)$ towards absolute zero (-273°S). It can be assumed that as the temperature approaches absolute zero, the “free” volume will become zero. This processing of dilatometric data allows for the estimation of the specific volume at 0 K, i.e., the “occupied specific volume” (V_z). Knowing the difference between the specific volume at any temperature (V_i) and at absolute zero V_z , one can determine the value of the free specific volume (V_c) as the difference $V_i - V_z$.

Figure 5 shows the change in “free” volume of HPPE and its modified samples, with the curves representing their dependence on temperature.

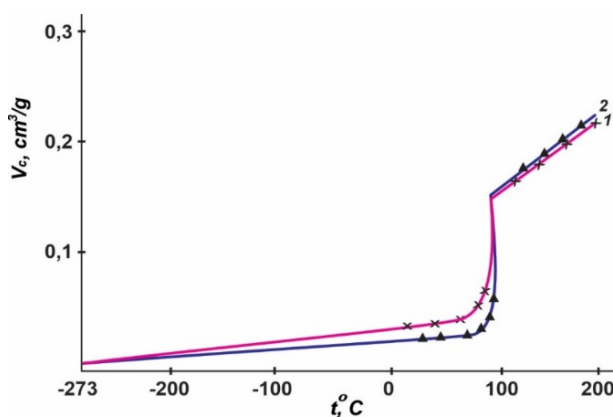


Figure 5: Dependence of free specific volume on temperature; 1- HPPE (original); 2- HPPE +0,05 mass % PhAn +0,05 mass % PhAc.

From the data presented in this figure, it is clear that as the temperature decreases, the free volume in the samples decreases according to a certain pattern. A sharp change in the free volume (V_c) is observed in the region of the first-order phase transition. Analyzing the curves in Figure 5, it can be concluded that the samples of HPPE containing 0,05 mass% PhAn + 0,05 mass% PhAc have the lowest free volume.

This phenomenon is likely explained by the fact that the introduction of the structure-forming additive increases the total number of nucleation centers forming simultaneously. As a result, the growth of spherulites stops once they reach a certain size, leading to the formation of

a fine-spherulitic supramolecular structure in the polymer with fewer defects in the crystalline regions (Cail et al., 2001; Ivanchev et al., 2006; Halasa et al., 2005), which accounts for the noticeable reduction in V_c .

It is known (Wong et al., 2019) that glass transition corresponds to a state of the polymer in which its structure becomes “frozen”.

The glass transition temperature T_g of HPPE was determined by extrapolating the dependence of $V_i=f(T)$ from the temperature range where the polymer is in a liquid state to the solid-state region.

For the original HPPE $T_g = 50^\circ\text{C}$, samples HPPE +0,1 mass% PhAn+0, mass% PhAc. HPPE +0,1 mass% PhAn+0,1 mass% PhAc. $T_g = 80^\circ\text{C}$, samples with HPPE +0,05 mass% PhAn+0,05 mass % PhAc reaches 90°S .

To evaluate the cooling rate of products in a mold during injection molding or extrusion, it is necessary to understand the kinetics of isothermal crystallization.

The kinetic crystallization data were quantitatively analyzed using the Avrami equation (Avrami, 2000; Ermakov, et al., 2007; Cesur et al., 2014; Hwang et al., 2012; Mehrabova et al., 2025b).

$$\varphi = 1 - \chi\theta\exp^{-Kr^n} \quad (1)$$

where φ – represents the portion of the polymer that has not yet transformed into the crystalline phase; χ - is the fraction of crystallized material; θ – is the fraction of uncrystallized material; K – is the generalized constant for nucleation and growth of crystalline formations; n - provides information about the crystallization mechanism (this value ranges from 1 to 4 and depends on the shape of the growing crystals).

To determine the Avrami exponent from the experiment, the formula (1) is subjected to double logarithmic transformation,

$$\lg(-\ln\varphi) = \lg K + n\lg\tau \quad (2)$$

It is easy to verify that in the coordinates $\lg(-\ln\varphi) - \lg\tau$ formula (2) represents a linear equation, allowing for the determination of n and τ at different temperatures. The data processing for the crystallization kinetics of HPPE and its modified samples is shown in Figure 6.

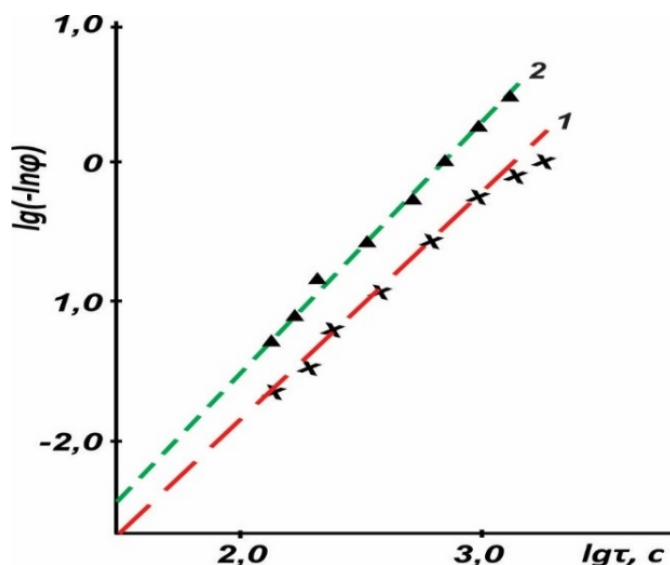


Figure 6: Crystallization kinetic curves of HPPE in Avrami coordinates. 1- HPPE +0,05 mass% PhAn+0,05 mass% PhAc; 2- HPPE (original).

It has been determined that for HPPE $n=1,7$ and for its modified samples $n=2$. The increase in the slope of the linear dependence $\lg(-\ln\varphi) = \lg K + n\lg\tau$ (Fig. 6) indicates an increase in

the crystallization rate of HPPE in the presence of the structure-forming additive, leading to the formation of plate-like crystals with continuous nucleation of crystallization centers.

4 Conclusion

The study experimentally established the process of obtaining polymer compositions for electrical insulation purposes based on high-density polyethylene grade 10803-020 with the addition of phthalic anhydride (PhAn) and phthalic acid (PhAc). The optimal composition of the polymer mixture for enhancing performance characteristics was experimentally determined. The most important dielectric parameters of the developed polymer composition, HPPE + 0,05 mass% PhAn + 0,05 mass% PhAc, were studied, including specific volume electrical resistivity (ρ_V), dielectric loss tangent ($tg\delta$) and dielectric permittivity (ϵ). The experimental results confirmed the contribution of the modifying organic additives, phthalic anhydride (PhAn) and phthalic acid (PhAc), to the improvement and stability of the electrophysical properties of HPPE during operation. In studying the effect of the modifying additives on the polymer, it was important to determine not only the nature of the additives' influence on HPPE but also the mechanism behind this effect. Dilatometric methods were used to investigate the influence of small amounts of PhAn + PhAc on the crystallization rate and mechanism of HPPE. It was found that the introduction of 0,05 mass% PhAn + PhAc into HPPE increases the polymer's crystallization rate. The increase in the crystallization rate of HPPE with the introduction of structure-forming agents allows for reduced cooling time of the product in molds during extrusion and injection molding processes. This results in enhanced production efficiency for equipment processing polymers based on modified HPPE.

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