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ELECTRET COMPOSITE MATERIALS BASED ON HIGH PRESSURE POLYETHYLENE

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Abstract. For the first time, electret polymer composite materials have been developed with a significantly small amount of Tellurium (Te) additives, which has scientific and practical interest. It was established that the introduction of a small amount of the specified additive into HPPE increases the surface charge density σ of the electret, reaches a maximum at a mass fraction of Te of 0,5 wt%, and then decreases. An increase in σ is observed for the HPPE + 0,5 wt % Te compared to HPPE and other contents. Using thermally stimulated depolarization, an increase in the stability of the electret state of the polymer was established, which can be explained by a change in the polymer structure, the appearance of new electrically active traps, and an increase in the polymer conductivity when Te was added to the polymer matrix. Good results are observed in the study of the dependence of the specific volume electrical resistance (ρV), the dielectric loss tangent ($tg\delta$) and the dielectric constant ε on the tellurium (Te) content at room temperature. At room temperature, the values of dielectric permittivity (ε) for HPPE and HPPE + 0,5 wt Te decrease during electretization. With increasing temperature, ε increases when the polymer macromolecules acquire increased mobility. A relationship between the type of supramolecular structure and the properties of the polymer products was established. The microstructures of HDPE modified with tellurium were studied using scanning electron microscopy. A relationship was established between the type of supramolecular structure and the properties of polymer products.

Keywords: HPPE, tellurium, electret properties, thermally stimulated depolarization, dielectric constant.
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1 Introduction

The rapid development of science and technology requires the use of new materials and study of their physical properties (Turnhiut, 2005; Nuriyev et al., 2014; Aliev, 2013; Elinson et al., 2021). The application of electrets (dielectrics that retain electric charges of different or the same sign for a long time) has been steadily expanding in recent decades. Electric converters of thermal, mechanical, optical, and other signals, generators, anti-corrosion coatings etc. are devices and structures in which electrets are successfully used. Therefore, expanding the range of knowledge in the field of technology and regulating the properties of such materials is relevant (Mehrabova et al., 2025; Gojayev et al., 2009; Magerramov et al., 2010).

Recently, there has been an increasing trend towards replacing difficult-to-process inorganic materials with organic polymers for creating electrets (Kravtsov & Brunig, 2000; Galikhanov et al., 2024). Particular attention has been paid to the search for new electret materials, which

involves obtaining polymer composite materials and structures based on them. For example, the advantages of polymer composite materials include the possibility of relatively easy control of their properties by modification with fillers of various natures, such as plasticizers, dyes, and other additives.

Depending on the nature of the polymer matrix and the type of filler, different methods are used to obtain an electroactive state, and various dielectric and electrophysical methods are used to study the features of charge stabilization. Numerous experimental data convincingly show that the electroactive properties of a heterogeneous polymer-filler system are mainly determined by the charge state of the phases, the features of the structure and interfacial interaction, and the distribution of the polarized field in the composite (Gojayev et al., 2013).

In (Galikhanov et al., 2017; Chen, 2003), the influence of the organic additive from dewaxing sludge (DS) on the surface charge accumulation density, electrical durability of high-pressure polyethylene (HPPE) films of various grades, and the change in charge density depending on storage duration was studied. It was found that the addition of just 2% (by mass) of DS to HPPE leads to a significant reduction in the accumulated surface charge density, which contributes to a noticeable increase in the electrical durability of the developed polymer material.

The cheapness and prevalence of a number of large-tonnage polymers (polyethylene, polypropylene, polystyrene, etc.) make them very promising materials for obtaining cornelectrets. It is no coincidence that a large number of works on the study of the electret effect in dielectrics are devoted to these polymers.

The choice of high pressure polyethylene (HPPE) is based on the well-studied electrophysical properties of this material and the fact that polyethylene (PE) in the oriented state exhibits stability of the effect with an effective surface charge density of $10^{-5} \div 10^{-4} Kl/m^2$.

The aim of this work was to develop electret composite materials with a significantly small amount of additives based on high pressure polyethylene (HPPE) with high and stable characteristics.

2 Experimental Methodology

High pressure polyethylene (HPPE) grade 10803-020 was chosen as the object of study, and the chemical element tellurium (chemical formula Te), which is brittle but becomes plastic when heated, was used as an additive. It is used in the production of alloys, semiconductors, rubbers, and other applications. It disperses well in melts. Additives in amounts of 0,1-1% by mass were introduced into the initial HPPE raw material by mechanical mixing in the melt.

Before introducing the additive into the HPPE, it was dispersed using sieve analysis on a unit to determine the grain size distribution. The sieve analysis unit consisted of a vibratory drive and set of sieves. A set of sieves was selected to cover the entire range of grain sizes of the tested mass of the bulk material (5-7 sieves). At the bottom part, there is a sieve plate for collecting the final sifted material. The vibratory drive imparts a vibration and rocking motion to the sieves installed and fixed on it. The duration of sieving for the coarse-grained material was 10 min, for the fine-grained material for approximately 20 min. The time and force of the vibration were regulated by a timer on the vibratory drive. To produce films by hot pressing from blanks obtained in a casting machine, a manual electrically heated hydraulic press of the PG-60 type was used.

The calculated amount of tellurium (Te) was loaded into a polished flat mold within a gasket 0,1 mm thick gasket. A fluoroplastic film (foil) was used to prevent adhesion. The mold was heated to $140-150°C$. Upon reaching the required temperature, the mold was compressed under a pressure of 150 atm. The films were extracted after cooling the molds.

Then, samples in the form of a disk with a diameter of 4 m were cut out from the films. The thickness of the samples was 50 μm.

The uniformity of the film was determined using an optical thickness gauge (N3B-2) and a

micrometer. The sample thickness value represents the arithmetic mean of the 10 measurements. Before the experiment, the samples were carefully degreased. Photographs of the developed samples (Figure 1).

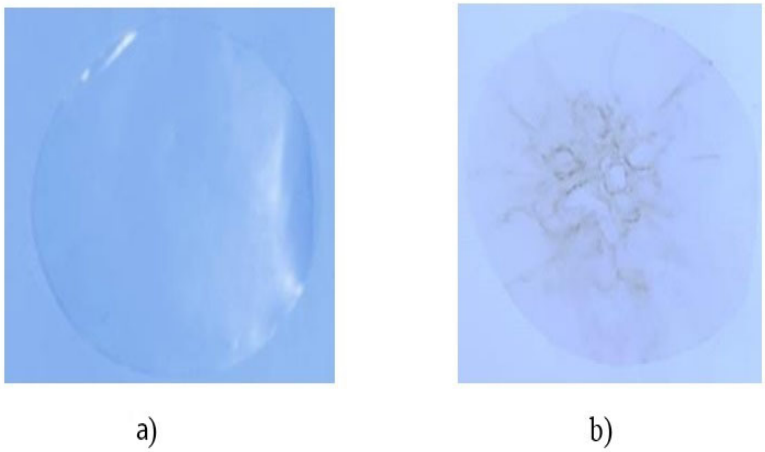

Figure 1: (a) HPPE (without additive), (b) HPPE + 0,5 wt% Te

The dielectric permittivity ε was measured using an automatic bridge R-589 at a frequency of 1 kHz in the range of 293-368 K. The specific volumetric electrical resistivity (ρ_V) of the samples was determined using a teraohmmeter E6-13A. It should be noted that the instrument measures only the resistance. To calculate the specific volumetric resistivity (ρ_V) , it is necessary to perform additional calculations based on the measured resistance, taking into account the applied voltage and the sample's physical dimensions, namely its thickness and the electrode area.

The dielectric loss tangent (tgδ) was measured by using an AC bridge (P589) at a frequency of 1 kHz.

The samples were charged from the non-metallized surface side by a negative corona at a voltage of 6 kV using a system of metal needles located vertically at a distance of 1 cm from the sample surface (Figure 2). It should be noted that samples charged by negative corona, when discharged, produce a significantly higher current than when charged by positive corona.

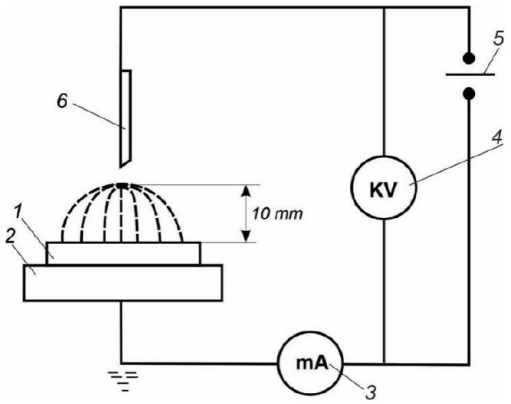

Figure 2: Installation for obtaining electrets in a corona discharge: 1-polymer film, 2-grounded electrode, 3-milliammeter, 4-kilovoltmeter, 5-high-voltage source AIM-70, 6-upper needle - electrode.

Figure 3 shows the schematic diagram of the surface charge density measurement using the vibrating electrode method (a non-contact induction method). Here: V - voltmeter, UIP-1 - DC

voltage source, C1-55 - oscilloscope.

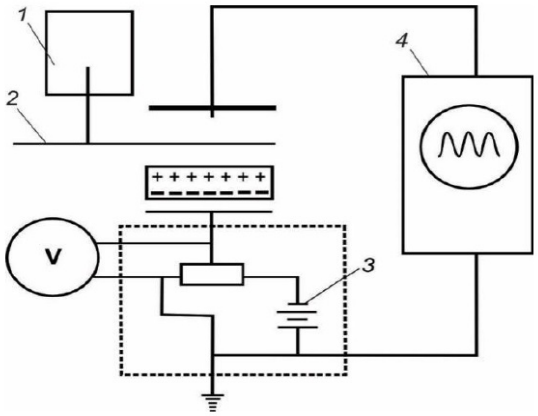

Figure 3: Scheme for measuring surface charge density: 1-electric motor, 2-impeller with four blades, 3-DC voltage source, 4-Oscilloscope.

The polarization time of the samples was 600 sec. The electret potential difference was determined immediately after polarization using the compensation method, and the surface charge density (σ) was calculated using the following formula:

$$\sigma_\varepsilon = \frac{\varepsilon\varepsilon_0 U_U}{d}$$

where, ε - is the dielectric ermittivity of the polymer, ε_0 - is the electric constant, U_k- is the magnitude of the compensation voltage, and δ - is the sample thickness.

To measure the currents of thermally stimulated depolarization (TSD) of the tested samples, they were placed between two electrodes in a heated measuring cell. The electrodes were connected to an electrometer amplifier U5-11, the output of which was connected to two coordinate recorders "Endim 620.02", on which the change in current depending on the temperature was recorded in the X-Y coordinate. The recording of thermostimulated currents in the range of 293-368 K was carried out with a linear temperature increase at a rate of 0,025 deg/sec (Figure 4).

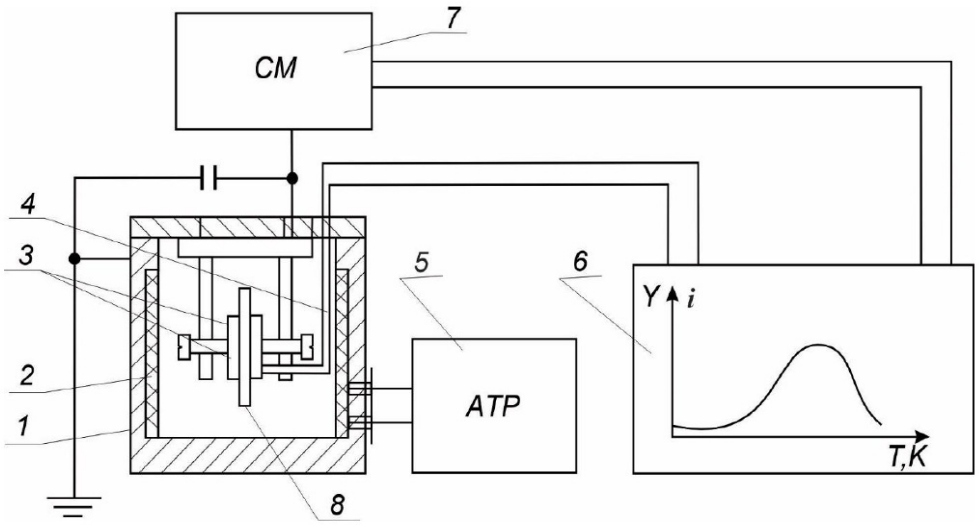

Figure 4: 1 - body of the thermal chamber; 2 - heating unit of the thermal chamber; 3 -electrodes; 4 - thermocouple; 5 - automatic temperature programmer; 6 - two-coordinate self-recording device (XY recorder); 7 - current meter; 8 -electret.

A qualitative assessment of structural changes in the HPPE film, following the introduction of small nanocarbon additives, was conducted by examining the morphology of supramolecular formations using scanning electron microscopy (SEM). Surface micrographs were obtained using a JEOL JSM-6610LV-x-Max scanning electron microscope.

Figure 5: Scanning electron microscope (SEM) model JEOL JSM-6610LV-x-Max for research of HPPE+MWCNT composites.

3 Results and Discussion

Note that corona electrets are obtained in fields of lower intensity than other electrets. For this, pointed electrodes, located at certain distance from the surface of the dielectric being charged are used. A corona discharge occurs between the point and the surface, resulting in air ionization and the movement of charge carriers (electrons and ions) to the electret surface. Charge carriers remained mainly in the surface layer of the dielectric. The field strength of the polymer is low and below the breakdown strength of the polymer.

Figure 6 shows the results of the change in the surface charge density (σ_K) of an electret made of high-pressure polyethylene (HPPE) and its compositions over time.

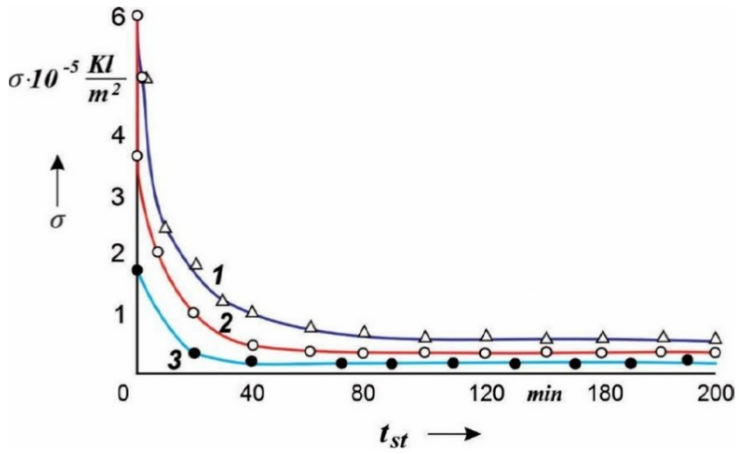

Figure 6: Dependence of charge density on the storage duration of electret from HPPE films and its modifications: 1-HPPE + 0,5 wt% Te, 2- HPPE (without additive), 3-HPPE + 1 wt% Te.

From these results, the nature of the changes $\sigma = f\,(t_{storage})$ for the composition HPPE + 0,5 wt% Te differs significantly from the other samples. The contribution of the added additive in the optimal amount to the process of charge accumulation at various depths of HPPE after

treatment with a negative corona is clearly visible from the data on the dependence of the surface charge density on the storage duration (Figure 6).

From the obtained data, it follows that after the polarization of HPPE films and its optimal modification, the process of depolarization begins, which is associated with the destruction and release of trapped charge carriers from traps located at various depths. At the same time, the initial value of the surface charge density (σ) for the optimally modified HPPE film (curve 1) becomes significant compared to that of the HPPE film without additive. After the complete destruction of homocharges, the surface charge density of the HPPE films and its optimal modification, reaching a certain value, did not change depending on the storage duration.

However, in this case, the numerical value of the optimally modified film turned out to be greater than that of HPPE films without additives and with other additive contents.

If at the initial stage of electron storage the release of charges occurs in shallow traps, then with relatively long-term storage the release of charges from deep traps takes place.

The dependence of σ on the amount of the additive is shown in Figure 7. The optimal value of the electret charge was observed at a filler content of 0,5 wt% Te.

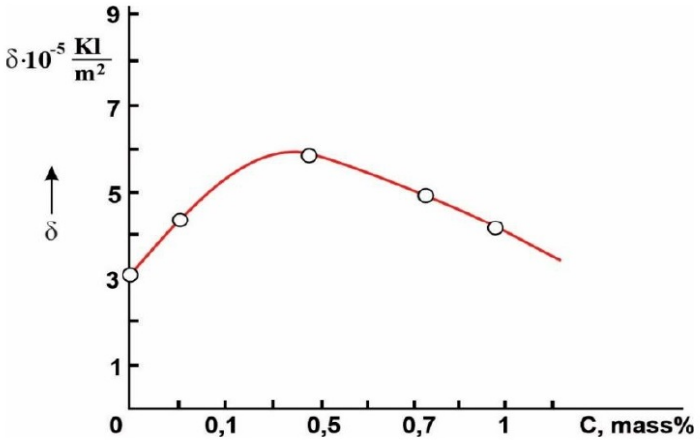

Figure 7: Dependence of the charge density of corona electrets of HPPE films on the content of Te additive.

These results show that the surface charge density (σ_K) of this composition is 1-2 times greater than that of pure HPPE. In addition, from Figures 6 and 7, it can be seen that for the polymer composition HPPE + 0,5 wt% Te and HPPE without additive charged under the same conditions, the charge of the electret of the optimal polymer composition is more stable.

The enhancement of the electret properties of polymer composites with the introduction of a small amount (up to 0.5 wt%) of additive can be explained by several factors.Firstly, when polymers are filled with solid dispersed fillers, new structural deviations arise in the polymer matrix, which can act as charge carrier traps. These include the phase boundary and the loosened adsorption layer of the polymer near the surface of the filler. The filling of polymers leads to changes in the characteristics of supramolecular structure formation and in the packing density.The decrease in the electret properties of the composite upon the introduction of a large amount of additive (curve 3) imparts antistatic and electrically conductive properties to the polymers. The electrical conductivity of the composite has a decisive influence on the rate of electret charge decay.

The obtained results can be explained by Yovcheva et al. (2004); Mehrabova et al. (2024) the fact that if the charges injected from the discharge zone (with a negative coronating electrode) - electrons - are captured by traps caused by structural defects in the surface layer of the polymer: then, probably, the optimal composition HPPE + 0,5 wt% Te leads to an increase in the number

of traps, as a result of destruction and oxidation occurring under the action of mechanical stress and charged particles in the surface layer of the polymer.

The above explanations are supported by thermally stimulated depolarization (TSD) data of polymer and composite electrets.

Figure 8 shows the experimental TSD results for composite corona electrets based on high-pressure polyethylene with Te additives.

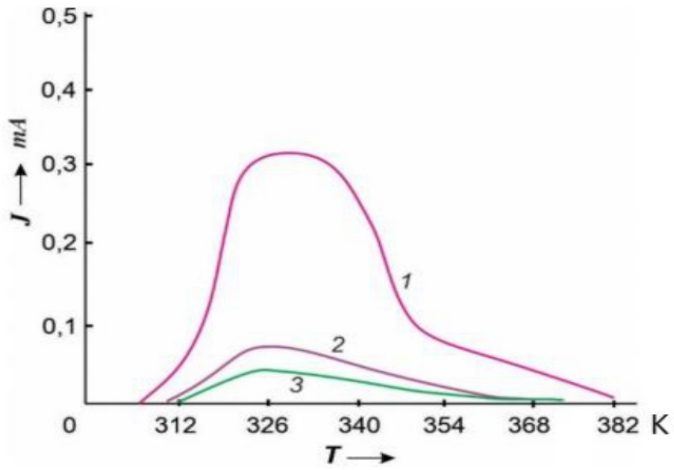

Figure 8: Thermogram of TSD current of HPPE films and its modifications charged by negative corona: 1-HPPE+0,5 wt% Te, 2- HPPE (without additive), 3-HPPE + 1 wt% Te.

Analyzing the TSD spectra, it can be noted that the introduction of 0,5 wt% Te filler into the polymer led to the appearance of deeper charge carrier trapping centers. Simultaneously, the number of traps for injected charges during coronating increases (increase in the intensity and area of the corresponding maximum) and their depth increases (the temperature position shifts to the high-temperature region). The nature of the appearance of the observed inversion peak at 326 K against the background of a large main peak in the region of 312 ÷ 368 K can be explained as follows: during the process of corona discharge action, a volume charge is formed, and interfacial polarization occurs at the boundaries of Te particles and the polymer (Abbasov et al., 2000; Godzhaev et al., 2008).

The direction of this polarization is opposite to that of the volume charges. During depolarization, inversion currents due to interfacial polarization were observed in the TSD spectra. Our proposed explanation is somewhat consistent with the Maxwell-Wagner effect, where the accumulation of charges in heterogeneous materials (in our case, composites) is attributed to the difference in conductivity in the amorphous and crystalline phases. When such a material is electrified, carriers will either collect near the given interfacial boundary or, move away from it, depending on which of the two conductivity currents is greater.

In general, in polymer electrets with homocharge, charge relaxation can occur due to the intrinsic conductivity of the dielectric. The experimental data obtained indicate that conductivity in the materials under study plays a decisive role in the relaxation process of the electronic state.

In Kestelman et al. (2000); Tyutnev et al. (2004); Balakina et al. (2010), it was shown that the structural properties and phase transitions of polymers and electrets based on them are reflected in the dielectric properties, because the dielectric permittivity of polymers is due to dipole, electronic, and resonance polarizations.

Figure 9 presents the results of studying the dielectric permittivity,- (ε) of the initial HPPE and its compositions based on the temperature before and after the negative corona treatment.

Table 1: Electrophysical properties and composition of HPPE.

No	Materials	Properties		
		tgδ f=1kHz, t=20^0 C	ρ_V,(Om·m) t=20^0 C	ε
	HPPE -108	$4{,}5{\cdot}10^{-4}$	$1{\cdot}10^{15}$	2,4
	HPPE + 0,5 wt % Te	$3{\cdot}10^{-4}$	$1{\cdot}10^{9}$	2,3
	HPPE + 0,7 wt % TE	$4{\cdot}10^{-4}$	$1{\cdot}10^{7}$	2,3
	HPPE + 1 wt % Te	$4{\cdot}10^{-4}$	$1{\cdot}10^{7}$	2,4

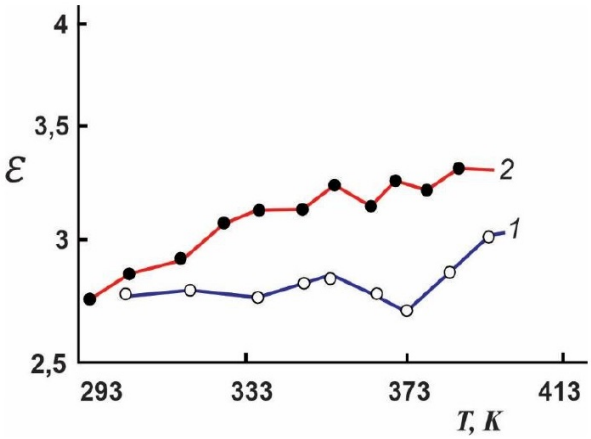

Figure 9: Temperature dependence of (1) dielectric permittivity of high -pressure polyethylene and (2) electret based on it.

It can be seen from Figure 9, that at room temperature, the values of dielectric permittivity for both HPPE without additive (curve 1) and HPPE + 0,5 wt% Te (curve 2) decrease during electretization. This is most likely due to the presence of injected charge carriers in corona electrets. With increasing temperature the dielectric permittivity (ε) increases when the polymer macromolecules acquire increased mobility.

This indicates that the conclusions drawn from the thermally stimulated depolarization data (Figure 7) are confirmed by the dielectric permittivity data, where additional orientation of the polymer dipole groups occurs (Yovcheva et al., 2007; Goldade et al., 1998; Godzhaev et al., 2020).

A good correlation was observed between the specific volume electrical resistivity (ρ_V), dielectric loss tangent (tgd) and dielectric permittivity (ε) and the tellurium (Te) content at room temperature, as shown in Table 1.

From the table, it can be seen that tg δ, ρ_V and ε of HPPE are also sensitive to the introduction of the Te additive, with a content of 0,5 wt% Te being optimal here as well, as it ensures high and stable electret properties compared to both the initial HPPE and HPPE with other additive contents (Nuriyev et al., 2017; Godzhaev et al., 2010).

In addition, the introduction of organic Tellurium compound into the polymer (HPPE) leads to an increase in molecular mobility and a significant decrease in the specific volumetric electrical resistance ρ_V .

As is well known, the stability of the electret state in polymer dielectrics is largely determined by localized surface states (traps). Due to the physicochemical characteristics of the surface structure of these materials, surface traps often turn out to be energetically deeper than bulk traps. As a result, the homocharge formed during the preparation of electrets accumulates in the surface traps and is effectively retained there.Studies of the electret properties

of polyethylene with the addition of tellurium (Te) have shown that the presence of Te in an optimal amount influences the manifestation of the electret effect in polyethylene, despite the decrease in conductivity ρ_V.

It is evident that the introduction of Te in an optimal concentration leads to an increase in the density and degree of crystallinity of the material and, consequently, to a reduction in moisture permeability, which results in a decrease in conductivity

Regulation of the supramolecular structure of amorphous-crystalline polymers essentially means obtaining polymer materials with the desired properties. During the investigations of the microstructure of HPPE modified with Te using electron microscopy, we obtained interesting data.

Figures 10 and 11 show microphotographs of the initial HPPE and HPPE containing an optimal amount of Te.

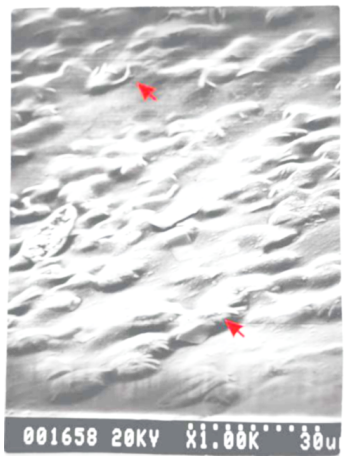

Figure 10: Microphotography of the HPPE film (initial)

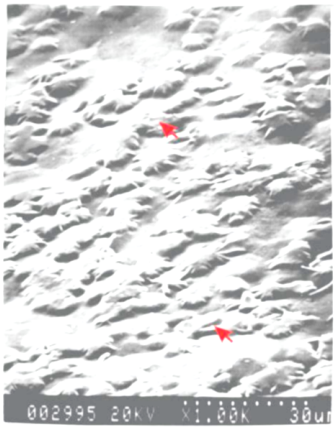

Figure 11: Microphotography of the HPPE film +0,5 mass% Te.

From Figures 10 and 11, it follows that Te does not tend to agglomerate, since the size of the micro-additive does not change, and its distribution in the polymer matrix is almost uniform both statistically and in the matrix mixture (Kovalenko & Goldade, 2021; Novozhilova et al., 2021; Mehrabova et al., 2025). In the accumulated (Te) LDPE, a slight increase in the size of supramolecular elements and the appearance of numerous voids are observed, which contribute to an increase in TSD. As can be seen from the electron micrographs, in the polymer composition obtained by hydraulic pressing, ordered regions and voids appear that are not observed in the unfilled state, which leads to an increase in TSD values when tellurium (Te) is present in the optimal amount.

Thus, in these composites, three distinct physical effects can be proposed as responsible for the relaxation of the electret charge: the relaxation of injected homocharge, the relaxation of heterocharge (dipole orientation), and the relaxation of Maxwell–Wagner polarization (at the phase interface).

Based on the microphotography data and the comparative structural analysis, we are entitled to assert that trace additions of tellurium have the ability to slow down the process of supramolecular surface structure formation, primarily by integrating into the amorphous phase of the polymer and creating centers of structural organization. In other words, an increase in tellurium content up to an optimal amount contributes to a reduction in the degree of crystallinity. The introduction of Te up to 0.5 wt % (by weight), which is technologically compatible with HPPE, simultaneously promotes charge accumulation in the final polymer material when used at its optimal concentration (Balasubramanian et al., 2018).

Thus, the observed improvement in the electret properties and the stability of the composition based on HPPE can be attributed to the structuring features of the specified additive.

4 Conclusion

The composition and amount of organic additive modifying the electret properties of high-pressure polyethylene grade 10803-020 were determined. The developed modification of HPPE is distinguished by the significantly small amount of tellurium (Te) additives and their technological compatibility. It was established that with a small amount of the specified additive in HPPE, the surface charge density σ of the electret increases and reaches a maximum at a Te content of approximately 0,5 wt %, and then begins to decrease.

Analysis of the thermally stimulated depolarization data established an increase in the stability of the electret state of the polymer compositions, which means that using additives in optimal amounts forms a stable HPPE structure.

It was established that at room temperature, the values of ε for both HPPE (initial) and HPPE + 0,5 wt % decrease during electretization, and increase with increasing temperature.

The investigation of HPPE using scanning electron microscopy allowed the establishment of the relationship between the structure and properties of polymer products, and conducting a targeted search for the most effective doping additives capable of improving the physicomechanical, electret, and technological properties of the polymer.

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