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ABSTRACT

of the dissertation for the degree of Doctor of Science

SCIENTIFIC BASIS OF MECHANICAL-CHEMICAL SYNTHESIS OF DYNAMICALLY VULCANIZED NANOCOMPOSITES BASED ON POLYOLEFINS AND ELASTOPLASTICS WITH ELECTRICALLY CONDUCTIVE AND HIGH PHYSICAL-MECHANICAL PROPERTIES

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Applicant: **Khayala Vagif gizi Allahverdiyeva**

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The work was performed at “Mechanical-chemical modification and processing of polymers” laboratory of the Institute of Polymer Materials of Ministry of Science and Education Republic of Azerbaijan.

Scientific consultant: Doctor of chemical sciences, professor
Najaf Tofig oğlu Kakhramanov

Official opponents: Full member of ANAS,
doctor of chemical sciences, professor
Abel Mammadali oğlu Maharramov
Corresponding member of ANAS,
doctor of chemical sciences, professor
İslam İsrafil oğlu Mustafayev
Corresponding member of ANAS,
doctor of chemical sciences, professor
Mahammad Baba oğlu Babanlı
Corresponding member of ANAS,
doctor of chemical sciences, professor
Tofig Abbasali oğlu Aliyev

One-time dissertation council under registration number BED 1.28 on the base of dissertation council FD 1.28 of Supreme Attestation Commission under the President of the Republic of Azerbaijan operating at Institute of Polymer Materials of Ministry of Science and Education Republic of Azerbaijan

Chairman of the
Dissertation council:

Scientific secretary of the
Dissertation council:

Chairman of the
scientific seminar:

Corresponding member of ANAS,
doctor of chemical sciences, professor
Bakhtiyar Ajdar oğlu Mammadov

PhD in chemistry
Fatima Alimirza gizi Mustafayeva

doctor of chemical sciences,
associate professor
Nushaba İsmayil gizi Gurbanova

GENERAL DESCRIPTION OF WORK

The relevance and degree of development of the topic. The analysis of research conducted in the last 10 years in the world shows that nanofillers significantly contribute to the structure and quality properties of polymer nanocomposites¹. In this regard, scientific research aimed at creating nanocomposites with a set of necessary properties is of greater interest. The use of nanoparticles has given researchers a new look at the processes involved in developing structural materials for specific applications². The interest in the use of nanofillers is due to the fact that nanoparticles have an enhanced surface and can be compared to the globular structure of a polymer in an alloy, which gives a unique opportunity to obtain thermally stable heterogeneous "nucleation centers"³. Polyolefins (PO) are high-tonnage polymers characterized by their unique and relatively high physical-mechanical properties, which are easily processed by all known methods. As a result of modifying their structure and properties in various ways to replace products made of non-ferrous and ferrous metals with polymer composites, it leads to the opening of promising opportunities that can predict the purchase of construction materials. In the presented dissertation work, attention is focused on the preparation of multi-functional electrically conductive polymer materials with high physical-mechanical properties based on a wide range of PO. It is quite clear that the developed materials should be distinguished by

¹ Al-Sheheria, S.Z. The preparation of carbon nanofillers and their role on the performance of variable polymer nanocomposites / S.Z. Al-Sheheria, Z.M. Al-Amshanya, Q.A. Al Sulamia [et al.] // *Designed monomers and polymers*, – London: – 2019. Vol. 22, № 1, – p. 8-53.

² Müller, K. Review on the Processing and Properties of Polymer Nanocomposites and Nanocoatings and Their Applications in the Packaging. Automotive and Solar Energy Fields /K. Müller, E. Bugnicourt, M. Latorre [et al.] // *Nanomaterials*. – Basel: – 2017. Vol 7, Issue 4, – p. 74.

³ Wang, X. The Study of Crystallization Behavior, Microcellular Structure and Thermal Properties of Glass-Fiber/Polycarbonate Composites/ X. Wang, Y. Sun, J. Hu [et al.] // *Polymers*, – Basel: – 2023. Vol 15, – p. 1546.

a number of useful properties, which will create wide opportunities for their use in various fields of the electronic industry. As the research results show, it is possible to maintain the necessary fluidity of the alloy by using various technological additives to ensure the processability of polymer materials, except for electrical conductivity. Taking into account that electrically conductive polymer nanocomposites can be used for fastening to metal surfaces, there is a need to increase their adhesion strength. To prevent the conductor or semiconductor from overheating during long-term use, they must have good thermal conductivity. In other words, an electrically conductive polymer composite should have multifunctional properties, which is a difficult task to implement in one material, and at the same time, it is a rather interesting and important scientific-technical problem.

When evaluating the electrophysical properties of polymer materials in the literature, scientists have vague ideas about the mechanism of the electrical conductivity process and the results of the analysis of their properties^{4,5}.

In a number of cases, conflicting information is also given during the interpretation of the main theoretical approaches to the development of electrically conductive polymer materials, which does not allow the establishment of a unified whole theory of research results⁶. This situation is explained by the fact that many scientists did not pay due attention to the in-depth study of structural properties along with their properties during the evaluation of research results on the creation and electrical conductivity of polymer composites.

⁴ Борукаев Т.А., Физико-механические свойства композитов на основе полиэтилена высокой плотности и технического углерода / Т.А.Борукаев, Д. С. Гаев // Прикладная физика, – Москва: -2017. №5, -с.76-81.

⁵ Kim, J. Conductive polymers for nextgeneration energy storage systems: Recent progress and new functions / J. Kim, J. Lee, J. You [et al.] // Materials Horizons, – Cambs: – 2016. Vol. 3, – p. 517–535.

⁶ Cheng, X. Soft surface electrode based on PDMS-CB conductive polymer for electrocardiogram recordings / X. Cheng, C. Bao, X. Wang [et al.] // Applied Physics A, – Germany: – 2019. Vol. 125, – p. 876.

Unfortunately, it should be noted that until now there are no reliable criteria for evaluating the electrical conductivity of the polymer matrix and the structural properties that generally determine the quality of the prepared composite. A comprehensive analysis of the segregated structure and properties of only the initial polymer matrix and nanocomposites, depending on the type and amount of the filler, allows us to gain a more complete understanding of the chain mechanism of electrical conductivity. Finally, it seems possible to adjust the value of this indicator and get closer to obtaining construction materials with predetermined structures and properties. The lack of a systematic approach to the development of electrically conductive nanocomposites and the study of the technological features of their processing has become one of the important factors preventing their wider application in special fields of the electronic and electrical engineering industry.

Object and subject of the research. As the object of research, polyolefins- high-density polyethylene (HDPE), low-density polyethylene (LDPE), isotactic polypropylene (PP), random copolymer of polypropylene (RPP), ethylene-propylene block copolymer (EPBC), ethylene hexene-1 copolymer (EHC), ethylene butene-1 copolymer (EBC), compatibilizers- maleinated HDPE (PE-g-MA) and maleinated PP (PP-g-MA), elastomers- butadiene-styrene copolymer (SKS-30) and ethylene-propylene-diene copolymer (EPDE-40), fillers- technical nanocarbon, nanographite, aluminum and copper powder, calcium or zinc stearate for processing purposes, vulcanizing agents dicumyl peroxide and sulfur were selected. The subject of the dissertation work is to study the complex properties of nanocomposites by physical, physical-chemical and physical-mechanical methods of electrically conductive nanocomposites compatibilized with PE-g-MA and PP-g-MA based on the aforementioned POs, as well as POs containing nanocarbon, nanographite, aluminum and copper powder nanoparticles and elastoplastics based on EPDE or SKS, dynamically vulcanized nanocomposites and to determine the processing technology,

classification and possible application areas of the obtained multifunctional nanocomposites.

Aims and objectives of the research. The aim of the dissertation work is to develop the scientific basis of the mechanical-chemical synthesis of various types of multifunctional electrically conductive nanocomposites with high physical-mechanical properties and satisfactory processability based on a wide variety of PO and elastoplastics. The use of PO as a polymer matrix allows determining the role of nanoparticles and related ingredients in the formation of the mechanism of electrical conductivity and the main physical-mechanical properties of nanocomposites at the micro- and macro-structural level. Compatibilizers based on PE-g-MA or PP-g-MA were used to increase the compatibility of the mixed components and improve the properties of the nanocomposites, respectively. Elastoplastics have also been used as a polymer matrix in order to obtain electrically conductive nanocomposites distinguished by their flexibility and elasticity, which allows obtaining construction materials with rubbery properties in the process of mixing and dynamic vulcanization.

The following tasks have been solved to achieve the goal:

- ✓ To study the technological aspects of mixing in the process of developing nanocomposites, mechanical-chemical modification of a wide variety of PO and elastoplastics, and improving the compatibility of the components in the mixture;
- ✓ Study of the effect of a number of fillers separately and together on electrical conductivity and complex physical-mechanical properties of nanocomposites;
- ✓ Development of the scientific basis of the formation mechanism of electrically conductive chains in nanocomposites depending on the type of filler, a wide variety of PO, the characteristics of the segregated structure of elastoplastics and the crystallinity degree;
- ✓ To study the rheological properties of nanocomposites in a wide temperature range (170-230°C) and shear stress; to study the

dependence of shear rate on shear stress; to determine the dependence of viscosity on shear stress and temperature, the activation energy of viscous flow, and the effect of various amounts of fillers on the temperature-invariant characteristic of viscosity properties;

- ✓ To study the effect of the type and amount of fillers on the first and second phase transition, the temperature dependence of the free specific volume, and the mechanism of crystal growth during the crystallization process in nanocomposites obtained on the basis of different types of PO by the stepwise dilatometry method;
- ✓ To investigate the effect of adhesion strength of electrically conductive nanocomposites against the surface of various metals, depending on the type and amount of polymer matrix and filler;
- ✓ In connection with conducting thermal deformation studies of nanocomposites, to determine the temperature ranges of crystalline, highly elastic, glassy and viscous-flow states depending on the type of polymer matrix, filler and vulcanizing agent;
- ✓ To study the mechanism of dynamic vulcanization of nanocomposites to obtain electrically conductive nanocomposites with different flexibility;
- ✓ To develop the scientific basis of mechanical-chemical modification, research and dynamic vulcanization of electrically conductive nanocomposites based on PO and elastomers;
- ✓ To study the technological features of the processing of electrically conductive nanocomposites by extrusion and pressure casting;
- ✓ To classify electrically conductive nanocomposites in order to predict different practical applications depending on their composition.

Research methods. The following modern methods were used in order to confirm the accuracy of the scientific research conducted in the dissertation work: infrared spectroscopy, derivatographic

analysis, scanning electron microscope, electron microscopy, X-ray phase analysis, Vicat softening temperature, thermomechanics (Kanavets), rheology, stepwise dilatometry, tensile strength, elongation at break, ultimate strength in bending, thermal conductivity, frictional resistance, electrical conductivity, peel resistance (adhesion).

Conceptual issues presented for defence:

- The results of the study on the effect of the type and amount of filler on the formation mechanism of electrical conductivity in nanocomposites with a segregation structure based on a wide variety of PO and elastoplastics;
- The results of the systematic study of physical-mechanical properties of nanocomposites based on PO depending on the type of filler and polymer matrix, new scientific approaches to the formation of the structure-property relationship;
- The kinetic regularities of the crystallization process and crystal growth mechanism in electrically conductive nanocomposites based on a wide variety of PO and elastoplastics, theoretical principles of development, production and research of multifunctional electrically conductive nanocomposites;
- Results and discussion of the study of the physical-mechanical properties of electrically conductive nanocomposites obtained during dynamic vulcanization of PO and elastoplastics;
- Data obtained from the experimental study of the monotrem technology of processing nanocomposites by extrusion and pressure casting methods, their discussion and scientific conclusions;
- A multifunctional classification of electrically conductive nanocomposites developed to predict practical applications.

The scientific novelty of the research. For the first time during the dissertation work:

- Preparation of nanocomposites with a multi-functional segregation structure based on a wide variety of PO and elastoplastics, the separate and combined effects of technical carbon (TC), graphite (Gr) nanoparticles, compatibilizer,

aluminum and copper on electrical conductivity, physical-mechanical and thermal-physical properties were systematically studied;

- In a polymer matrix based on a wide variety of PO and elastoplastics, based on the results of IR-spectroscopy, X-ray phase analysis, DTA analysis, electrical conductivity, electron microscopy, SEM analysis, stepwise dilatometry, thermomechanics, rheology and complex physical-mechanical analyzes of the electrically conductive chain structure with the presence of nanoparticles in the interphase field theoretical propositions and results on the study of the formation mechanism were given. It has been shown that as crystallinity degree of the polymer matrix increases, the smaller amount of nanoparticles is required to obtain maximum electrical conductivity in nanocomposites;
- The rheological properties of nanocomposites were studied depending on the temperature, the applied load and the composition of the used components, and graphical data on temperature-invariant characteristics of viscosity properties of nanocomposites were obtained to predict their temperature regime according to real processing conditions;
- The peel resistance values of multifunctional nanocomposites from aluminum and copper foil surfaces were determined;
- According to the Kolmagorov-Avrami equation, a kinetic analysis of the crystallization process of electrically conductive nanocomposites with a segregated structure was performed depending on the amount of TC and graphite, and the mechanism of formation of the type of crystals was determined. It has been shown that as the amount of nanoparticles increases, as a rule, along with the continuous formation of crystallization centers, there is a change in the growth mechanism of crystals from 3D-sized spherulite to the formation of 2D-sized disc-shaped crystalline derivatives;
- The scientific bases of the preparation, research and processing of multifunctional electrically conductive nanocomposites with

segregated structures and dynamic vulcanized nanocomposites have been developed.

- Based on the results of thermomechanical studies of nanocomposites based on PO and elastoplastics, the temperature region of the first type of phase transition and the nature of deformation and phase inversion changes during the transition from one physical state to another were analyzed;
- The effect of temperature regime, pressure and other parameters of mechanical-chemical synthesis on the physical-mechanical properties of multifunctional and dynamic vulcanized nanocomposites based on PO was studied by reactive monotreme extrusion and pressure casting method;
- The classification of developed electrically conductive nanocomposites depending on the type of polymer matrix and the ratio of the used components is given and the most preferred areas of their practical use are indicated.

The theoretical and practical significance of the research:

- The new scientific approaches and results presented in the dissertation can be used by researchers, university professors, doctoral students, and graduate students as a necessary theoretical basis for studying the structure and properties of nanocomposites obtained on other polymers and fillers;
- It enriches and develops theoretical and practical knowledge and information in the direction of the scientific basis of purchase and research of electrically conductive nanocomposites, the results of theoretical and experimental analysis of their segregation structure, properties, chemistry and technology of composite materials;
- The results of experimental studies and the results of experimental studies carried out in the direction of obtaining multicomponent electrically conductive nanocomposites and a systematic scientific approach to the selection of optimal conditions for the study of processing can be useful for specialists working in extrusion and casting production enterprises based on composite construction materials;

- Dynamically vulcanized electrically conductive nanocomposites obtained in the process of mechanical-chemical synthesis based on elastoplastics are distinguished by their environmental cleanliness, high strength, and flexibility and elasticity characteristic of rubber, on the other hand. Unlike rubbers, the practical advantage of elastoplastics is also the possibility of using them in casting aggregates with high productivity in the production of products based on them;
- The prepared nanocomposites have undergone industrial tests at the "METAK" LLC plastic products processing plant. Positive test reports and laboratory regulations are presented in the appendices section.
- A classification of the developed multifunctional electrically conductive nanocomposites is given to predict practical applications such as electrical conductors, semiconductors and antistatics;

Personal contribution of the author. The personal participation of the author played a leading role in conducting experimental studies, analyzing and discussing the obtained results, developing and writing the strategy of the dissertation work, publishing articles and discussing report thesis materials at international conferences.

Approbation and application of research. The results of the dissertation were presented at the following international and national conferences: International Scientific Conference on "Actual problems of modern chemistry" dedicated to the 90th anniversary of the Institute of Petrochemical Processes named after Academician Y.N. Mammadaliyev of ANAS (Baku, 2-4 October 2019), International scientific conference on "Prospects of innovative development of chemical technology and engineering" (SDU, Sumgait, 28-29 November 2019), International United Academy of Sciences, General question of world science, collection of scientific papers on materials, IX International Scientific Conference, (Luxembourg, 30 November 2019); I International scientific-practical conference "Universities of Azerbaijan and Turkey: Education, science, technology" AMU,

(Baku, 18-20 December 2019); XVII International Conference of Young Scientists, National Academy of Sciences of Belarus, (Minsk, 22-25 September 2020); All-Russian scientific and practical conference "Perspective technologies and materials", (Sevastopol, 14-16 October 2020 and 6-8 October 2021); XV International Symposium on Fundamental and Applied Science Problems, MIAS, (Moscow, 30 November 2020); "Kirpichnikovsky Readings - XV International Conference of Young Scientists, Students and Graduate Students "Synthesis and Study of Properties, Modification and Processing of High-Molecular Compounds", (Kazan, 29 March-02 April 2021); MSF2022 Materialology of the future, RF (Nizhny Novgorod, 5-7 April 2022); International scientific and technical conference of young scientists "Innovative materials and technologies", (Minsk, 23-24 March 2022); International scientific conference dedicated to the 99th anniversary of the birth of National Leader Heydar Aliyev on the topic "Actual problems of modern natural and economic sciences" (Ganja, 5-6 May 2022); Republican scientific conference on "Catalysts, olefin-based oils" dedicated to academician N.M. Seyidov's 90th anniversary (IPCP, Baku, 19-20 May 2022); Republican scientific conference dedicated to the 110th anniversary of Academician A.M. Guliyev on the topic "Organic substances and composition materials of various purposes" (Baku, 30-31 May 2022); ANAS Ganja branch national conference on "Environmental protection, recycling of industrial and household waste" (Ganja, 24-25 November 2022), Conference dedicated to the 90th anniversary of Academician Sahib Museyib oglu Aliyev "Synthesis of petroleum chemistry, polyfunctional monomers, oligomers and polymers"(Baku, 22 June 2023).

Publications. The results of the dissertation work were published in 53 scientific works, 29 of them were scientific articles, 23 were thesis, in local and foreign journals, and one was an Azerbaijani patent. Articles in foreign journals have been published in "Heliyon" (IF-3.776, Q1, SCIE, England), "Journal of Elastomers & Plastics" (IF-2.215, Q3, SCIE, England), "Journal of the Chemical Society of Pakistan" (IF-0.68, Q4, SCIE), "Russian Journal of Applied Chemistry" (IF-0.869, Q4, SCIE), "Известия высших учебных

заведений. Химия и химическая технология” (Q4, WoS, Scopus), “Inorganic Materials: Applied Research” (WoS, Scopus), “Polymer Science, Series D” (Scopus), «Композиты и наноструктуры», «Материаловедение», «Пластические массы», «Все материалы. Энциклопедический справочник», «Перспективные материалы», in the local journals “Processes Petrochemistry and Oil-refining”, “New Materials, Compounds and Applications”, “Azərbaycan Kimya Jurnalı”, and “Kimya Problemləri Jurnalı”.

The name of the organization where the dissertation work was carried out. The dissertation work was carried out in accordance with the research plan at the "Mechanical-chemical modification and processing of polymers" laboratory at the Institute of Polymer Materials of Ministry of Science and Education Republic of Azerbaijan. In the fulfillment of the dissertation work, the author also participated in conducting joint scientific-research works on bilateral cooperation concluded with scientific organizations of Russian University of Chemistry and Technology named after D. I. Mendeleev (Moscow c.), Bashkortostan State University (Ufa c.), Institute of Radiation Problems of the Ministry of Science and Education Republic of Azerbaijan (Baku c.), Institute of Geology and Geophysics of the Ministry of Science and Education Republic of Azerbaijan (Baku c.), Azerbaijan State Oil and Industry University (Baku c.).

Scope and structure of the work. The thesis is 394 pages long (439020 characters) and consists of an introduction (21246 characters), 8 chapters (first chapter - 80952 characters, second chapter - 16754 characters, third chapter - 81693 characters, fourth chapter - 54586 characters, fifth chapter - 44214 characters, sixth chapter - 53916 characters, seventh chapter - 35057 characters, eighth chapter - 38671 characters), a summary (2389 characters) and main results (9542 characters), a 351-item references and appendices. The thesis includes 93 figures and 63 tables.

MAIN CONTENT OF THE WORK

In the introduction, the relevance of the problem is justified, the purpose and main issues, scientific novelty, theoretical and practical importance of the work are given, and the provisions presented for defense are explained.

In the first chapter, a literature review of the problem state of obtaining and researching nanocomposites, electrically conductive materials was presented. The main scientific provisions, goals and tasks of current research on the development and research of various types of composite materials, the problem of obtaining nanoparticles and their role in the formation of the structure and properties of the polymer matrix are given.

In the second chapter, the preliminary indicators of the components to be mixed, information on the methods of obtaining and researching composite materials, and the main methods of nanocomposite processing were reviewed.

The third chapter is devoted to the problem of analyzing the structure of metal-filled nanocomposites and studying their main physical-mechanical and physical-chemical properties.

In the fourth chapter, the results of research on the development and study of the structure, physical-mechanical and physical-chemical properties of nanocomposites based on a wide range of PO, TC and Gr were presented.

The fifth chapter is devoted to the study of the structure and complex of properties of multicomponent nanocomposites based on PO, compatibilizer, TC, Gr and metals. The effect of the amount of compatibilizer and TC on the change regularity of the adhesion strength of nanocomposites to the metal surface was considered.

The sixth chapter is devoted to the study of the mechanism of tunneling and electronic electrical conductivity of nanocomposites based on PO, TC and Gr and attention is paid to the formation mechanism of chain and cluster structure in nanocomposites. The main results, scientific propositions and new scientific approaches that allow to interpret the nature of the change of electrical conductivity of

nanocomposites in the interphase field depending on the type of PO and fillers are shown.

In the seventh chapter, for the first time, an analysis of the structure, complex physical-mechanical, physical-chemical properties and electrical conductivity of nanocomposites based on thermoplastic elastomers is given. Depending on the type of elastomer, thermoplastic PO used, the amount of TC, Gr, metal and compatibilizer, the regularity of change of electrical conductivity of thermoplastic elastomers were determined.

In the eighth chapter presents scientific approaches to the study of the technological features of the processing of electrically conductive multicomponent nanocomposites based on PO, TC and Gr by pressure casting and extrusion methods. The results of the classification of the prepared electrically conductive nanocomposites according to their composition and practical use in the electronic and electrical engineering industry are presented. A list of conclusions and literature references is provided at the end of this chapter.

Test reports and laboratory regulations for selecting optimal technological modes for processing by extrusion and pressure casting methods are presented **in the appendices**.

1. Crystallization regularities, rheological and physical-mechanical properties of metal filled nanocomposites based on polyolefins

First of all, it was important to study the selective effect of the type of polymer matrix and each of the components of the electrically conductive nanocomposite on their basic physical-mechanical, electrophysical, thermal deformation, rheological, technological and other properties in a consistent and step-by-step manner. Therefore, compatibilizers based on maleic anhydride-modified HDPE (PE-g-MA) and isotactic PP (PP-g-MA) were used to improve the compatibility between the components of the mixture. The use of compatibilizers had a positive effect on improving the quality characteristics of nanocomposites obtained on the basis of a wide range of POs.

In this chapter of the thesis, the separate influence of different types of metal nanoparticles on the main physical-mechanical and physical-chemical properties of nanocomposites based on PO is determined. Aluminum powder (AP) and Cu were used as metal filler, and HDPE, LDPE, PP were used as PO. The aim of the study was to determine the optimal amount of metal fillers to maintain the physical-mechanical properties of nanocomposites obtained on the basis of the specified PO at a complex level.

When comparing the results of studies on the physical-mechanical properties of composites based on PO and AP, it was determined that when the amount of AP increased from 0.5 wt % to 30 wt %, an indefinite change was observed in the values of the tensile strength and modulus of elasticity in bending. However, in all samples, a general trend in increasing the Vicat softening temperature and decreasing the elongation at break of the composites is observed. So, for example, unlike HDPE and PP, as a result of increasing the amount of AP in the content of LDPE, an increase in the value of the modulus of elasticity in bending and the tensile strength of the composites is observed, but the elongation at break decreases significantly.

It is known that both homogeneous and heterogeneous nuclear formation centers are present in the polymer matrix melt. Homogeneous centers are formed as a result of mutual orientation of macrochains in the alloy. Heterogeneous centers are formed during the orientation of macrochains on the developed surface of the filler. Naturally, as a result of such an orientation, the strength of adhesion bonds on the surface of AP particles will increase even more in the presence of PE-g-MA. Nucleation centers formed in the composite melt become crystallization centers during the cooling process.

The increase in melt viscosity of the filled composite can be attributed to the formation of a "transition monolayer" on the surface of the AP particles, which is further strengthened by the presence of PE-g-MA. A similar effect was observed us when determining the MFR in composites based on PP+AP. For example, if the MFR of the initial PP is 3.6 g/10 min, after adding 1.0, 3.0, 5.0, 10, 15, 20, 30 wt% AP, the value of this indicator changed in the following order: 3.6; 4.0;

3.1; 2.5; 1.3; 0.66; 0.12 g/10 min. As a result of sample cooling, linear macrochains of PO and compatibilizer that do not retain maleic anhydride (MA) are manifested by participation in the crystallization process and the formation of nuclei with a crystalline structure. It is clear that the macrochains holding the MA mangas are squeezed into the amorphous area between the spherulites in the process of crystal growth. During the introduction of metal nanoparticles, the process of growth of the crystalline structures of the polymer matrix is also accompanied by the compression of the fillers into the amorphous area between the spherulites, that is, the segregation process takes place. Thus, the amorphous area between the spherulites of the nanocomposites consists of a mixture of through chains of the polymer matrix, metal nanoparticles, and segments of macrochains holding MA mangas.

As can be seen from Figure 1 (a), AP particles in the non-compatibilized composite are unevenly distributed in the polymer matrix. However, after the introduction of the compatibilizer (PE-g-MA) (b), the AP particles are as if "knitted" and unevenly distributed in the polymer matrix, that is, they are not practically isolated. This situation, in turn, has a positive effect on the physical-mechanical properties of the composite.

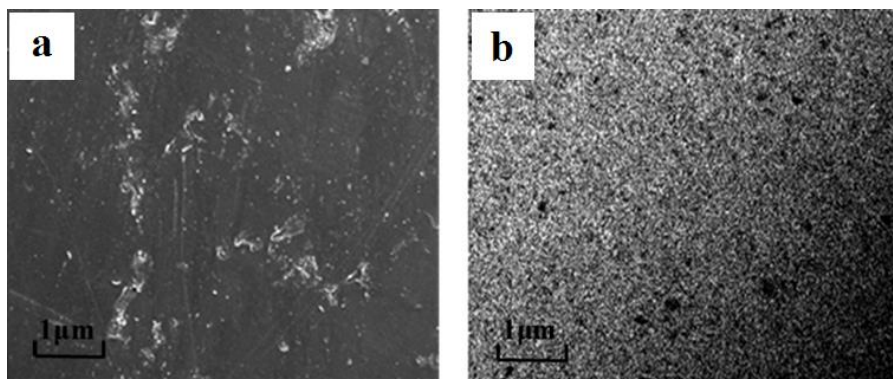


Figure 1(a, b). SEM images of filled composites: HDPE+20 wt % AP (a) and HDPE+ 20 wt % AP +2 wt % PE-g-MA (b)

In a similar manner, the influence of the inclusion of Cu powder on the main physical-mechanical and physical-chemical properties of PO was studied. It was determined that the properties of Cu-containing nanocomposites improve as a result of using PE-g-MA and PP-g-MA type compatibilizers. Temperature dependence of specific volume and specific free volume of metal-filled composites based on PO was studied by stepwise dilatometry method. The first type of phase transition and glass transition temperature were determined. Kinetic regularities of crystallization depending on the amount of metal filler in Kolmagorov-Avrami coordinates and growth mechanism of crystal nucleus formations of composites were studied. As a result of the continuous formation of nucleation centers when increasing the amount of Cu or AP from 0.5 wt% to 30 wt%, the mechanism of crystallization changes from spherical 3-D size to 2-D size of plate-type crystals.

The softening and first type phase transition temperature of metal-filled composites were determined by the method of thermomechanical studies. As the amount of filler increases, the softening temperature of the composite also increases. As a result of the vulcanization of the composites with dicumyl peroxide (DP), it was shown that along with the formation of a high elastic deformation area, it also transitions to a reticular state (Figure 2). As can be seen from the figure, with an increase in the amount of DP from 0.25 wt % to 2.0 wt %, quite noticeable changes are detected in the thermomechanical curves.

According to the obtained experimental data, the formation of three physical states - solid, highly elastic and viscous-flow - is possible when using DP in the amount of 0.25-0.5 wt%. When the amount of DP is 1.0-2.0 wt%, the density of the interchain structure reaches such a level that the polymer immediately changes from a highly elastic state to an irreversible reticular state (curves 4 and 5).

The adhesive-substrate adhesion mechanism in metal-filled composites was analyzed in detail depending on the amount of compatibilizer, Cu and AP. It was determined that the value of the adhesive (metal-filled composite) peel resistance from the surface of

the metal foil (substrate) depends on 4 main factors: the fluidity of the alloy, the polarity of the adhesive, the compatibility of the filler and the substrate, and the pressing temperature of the foiled nanocomposites. In this regard, the peel resistance of the obtained composites depending on the type and amount of polymer matrix, compatibilizer, and metal was studied and it was determined that the adhesion strength of compatibilized POs increases significantly. The best results are observed in composites containing 5.0-10 wt% metal and 2.0-3.0 wt% compatibilizer.

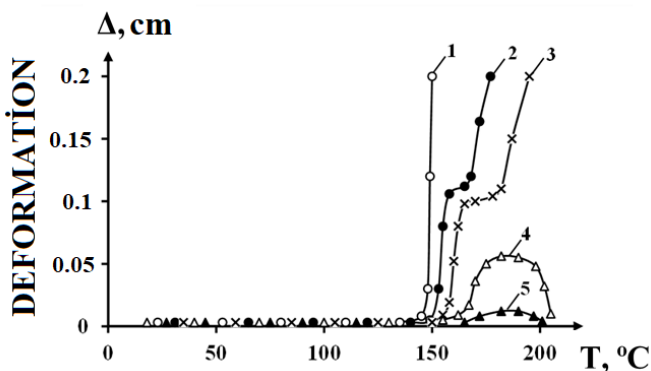


Figure 2. The influence of the amount of DP (wt %) on the regularity of changes of the thermomechanical curves of the composites based on HDPE+3.0 wt % PE-g-MA+5.0 wt % AP 1-(○); 2(●)-0.25 wt % DP; 3(x)- 0.5 wt % DP; 4(Δ)- 1.0 wt % DP and 5(▲)- 2.0 wt % DP.

2. Segregated structure and physical-mechanical properties of nanocomposites based on polyolefins, technical carbon and graphite

In this section, the results of the study of the effect of TC and graphite Gr on the main physical-mechanical, thermal physical and rheological properties of composite materials are discussed. Using the stepwise dilatometry method, unique results were obtained on the study of the kinetic regularities of crystallization and the mechanism

of the formation of crystalline derivatives depending on the amount of the above-mentioned fillers. Therefore, in this section, with the aim of obtaining electrically conductive composite materials based on HDPE, issues related to the justification of the selection of fillers for use in PO have been considered. Main attention was paid to the study of structure and complex physical-mechanical properties of nanocomposites based on TC and Gr selected from a number of fillers and a wide variety of PO.

In order to select effective conductive fillers, the effect of polymer composites on electrical conductivity and some physical-mechanical properties was first evaluated using HDPE and a number of TC and Gr samples as examples. In this regard, 9 types TC samples were used as research objects in Figure 3: 1- Printex XE 2B, 2- carbon black derived from acetylene (CB), 3- N-550; 4- П-514; 5- П-324; 6- П-803; 7- K-354; 8- П-234; 9- T-900.

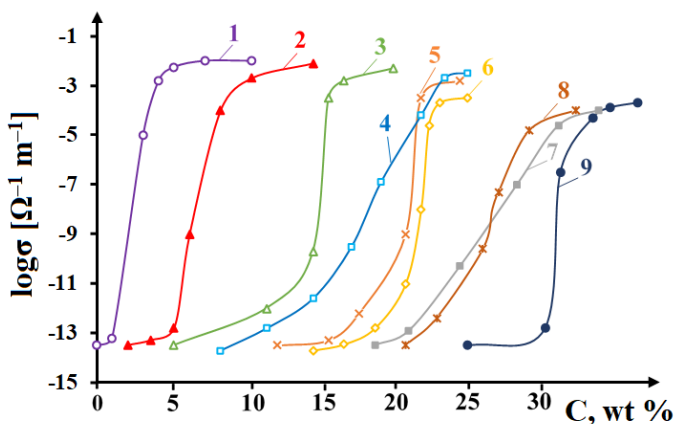


Figure 3. The effect of the amount of TC of different brands on the electrical conductivity of composites based on HDPE*: 1- Printex XE 2B; 2-CB; 3-N-550; 4- П-514; 5- П-324; 6- П-803; 7- K-354; 8- П-234; 9- T-900.

As can be seen from Figure 3, among the presented components, the Printex XE 2B (curve 1) and CB (curve 2) samples demonstrate higher efficiency. The advantage of these samples is that their maximum electrical conductivity was obtained with a relatively low amount of TC. The obtained data are of great importance, because they allow us to say that the difference in the effect of TC on the electrical conductivity is caused by many reasons. The most important of them is the enhanced surface area and dispersion of TC nanoparticles. For example, according to the given numbering, the sizes of TC nanoparticles vary in the following order: 1- (18-20 nm), 2-(30-40 nm), 3-(40-60 nm), 4-(120-140 nm), 5-(130-150 nm), 6-(130-150 nm), 7-(160-180 nm), 8-(160-170 nm), 9-(250-300 nm). As can be seen from the curves, as the particle size increases, the maximum electrical conductivity is achieved at higher amounts of TC.

However, the attempt to maintain electrical conductivity at a high amount of TC can lead to the deterioration of the complex physical-mechanical properties of composites based on HDPE*. This is also confirmed by the results given in table 1.

Table 1
Physical-mechanical properties of composite materials with different carbon content at the maximum value of electrical conductivity

№	HDPE contains various brands of TC wt %	Tensile strength, MPa	Elongation at break, %	Electrical conductivity, $\Omega^{-1} \text{ m}^{-1}$	MFR g/10 min
1	93HDPE*+7.0Printex XE 2B	33.0	50	$7.5 \cdot 10^{-2}$	2.2
2	88HDPE*+12 CB	31.8	30	$4.6 \cdot 10^{-2}$	1.3
3	85HDPE*+15 N550	25.4	20	$0.2 \cdot 10^{-2}$	0.2
4	77HDPE*+23 П-514	22.6	-	$5.6 \cdot 10^{-3}$	-
5	79HDPE*+21 П-324	19.7	-	$4.7 \cdot 10^{-3}$	-
6	78HDPE*+22 П-803	20.1	-	$4.6 \cdot 10^{-3}$	-
7	68HDPE*+32 K-354	17.8	-	$1.8 \cdot 10^{-3}$	-
8	69HDPE*+31 П-234	17.3	-	$8.5 \cdot 10^{-4}$	-
9	68HDPE*+32 T-900	17.9	-	$4.7 \cdot 10^{-3}$	-

HDPE - maleinated HDPE*

From the comparative analysis of the data in this table, it is clear that samples No. 1 and 2 have the best physical-mechanical properties, since the maximum effect on the electrical conductivity value was achieved at a relatively low amount of TC. The deterioration of the properties in samples 3-9 is due to the fact that a relatively higher amount of TC must be used to achieve a given maximum limit of electrical conductivity. However, as the amount of TC in the composite increases, the tensile strength, elongation at break, and MFR deteriorate.

Similar studies were carried out with Gr-filled HDPE*-based nanocomposites. Different brands of Gr were used as the research object (crystalline type ΓC-2 and ΓC-3, pencil type ΓK-1 and ΓK-2). The size of ΓC-2 and ΓC-3 nanoparticles is 25-45 nm, for ΓK-1 and ΓK-2 this indicator is in the range of 30-55 nm. Figure 4 shows the electrical conductivity properties of nanocomposites obtained on the basis of HDPE* with the above brands of Gr. As can be seen from the figure, the maximum value of electrical conductivity in these samples is obtained at 5.0-7.0 wt % ΓC-2 and ΓC-3 brands of Gr.

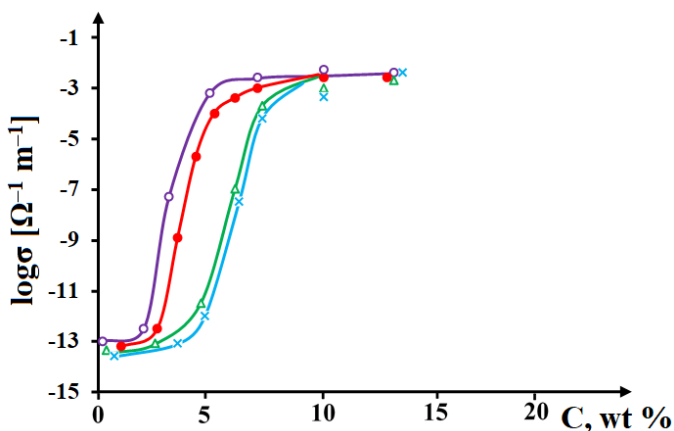


Figure 4. The effect of the amount of different brands of graphite on the electrical conductivity of nanocomposites based on HDPE*: **o** - ΓC-2; **•** - ΓC-3; **x** - ΓK-2; **Δ** - ΓK-1.

At this time, the best electrical conductivity indicator was obtained in HDPE samples with introduction of ΓC-2 brand Gr. Gr conducts electricity because it has weak Van der Waals interactions in each layer and consists of a system of polyconjugated bonds due to its π -electrons.

In addition, the physical-mechanical properties of nanocomposites containing 7.0 wt % of different brands of Gr in HDPE* were also studied. From the results of the study (table-2), it is clear that the physical and mechanical properties of Gr-filled nanocomposites are practically indistinguishable. The main difference is only the indicator of electrical conductivity.

In other words, Gr performs 3 main functions in nanocomposites: it provides electrical conductivity, imparts anti-friction properties and maintains the necessary level of MFR for processing products. Therefore, ΓC-2 brand of Gr was chosen as a filler to systematize research in the direction of obtaining and researching electrically conductive nanocomposites.

Table 2

Physical-mechanical properties of nanocomposites based on HDPE* incorporated with different brands of graphite

№	Composition of HDPE* with different brands of graphite, wt %	Tensile strength, MPa	Elongation at break, %	Electrical conductivity $\Omega^{-1} \text{ m}^{-1}$	MFR g/10 min	Friction in the first round, mg
1	HDPE*	31.3	435	$1.6 \cdot 10^{-13}$	5.6	139
2	93HDPE*+7.0 ΓC-2	31.5	40	$4.5 \cdot 10^{-2}$	6.2	92
3	93HDPE*+7.0 ΓC-3	30.8	35	$8.6 \cdot 10^{-3}$	6.1	91
4	93HDPE*+7.0 ΓK-1	31.0	40	$5.2 \cdot 10^{-4}$	6.0	88
5	93HDPE*+7.0 ΓK-2	31.2	40	$5.3 \cdot 10^{-5}$	5.9	90

2 carbon-containing fillers were selected in the studies of obtaining multifunctional electrically conductive nanocomposites based on a wide variety of PO, thermoplastic elastomers (TPE): **Printex XE 2B brand of TC and ΓC-2 brand of Gr.** Naturally, it

would be correct to clarify the process of segregation or separation in the structure of polymers beforehand. POs belong to semi-crystalline polymers, that is, they are considered as segregated materials. This is explained by the fact that in the process of crystallization of PO and the growth of crystals, their separation and formation into crystalline and amorphous phase takes place. After adding nanoparticles to PO, part of it participates in the formation of heterogeneous crystallization centers, and the rest is transferred to the amorphous area between the spherulites during the growth process of the crystalline derivatives of the polymer matrix. Thus, the process of segregation is the separation and redistribution of components of the same type in a polymer matrix. Therefore, it would be correct to consider PO and nanocomposites as materials with a segregated supramolecular structure. This feature of the segregation process provides unique opportunities to increase the physical-mechanical, thermal-physical and electrophysical properties of nanocomposites. Unfortunately, the results of studies on the effect of the amount of TC and Gr on the physical-mechanical properties of PO-based nanocomposites are not sufficiently substantiated in the scientific literature in this direction, and specific information is not given.

LDPE, PP, EHC, EBC, RPP and EPBC were used as a polymer matrix for the purpose of comprehensive analysis of the properties of nanocomposites based on PO. Conducting such studies resulted from the need to determine the effect of the degree of crystallinity and the structure of the PO macrochain on the main physical-mechanical properties of nanocomposites. It should be noted that in this section, the main attention is focused on the study of the effect of Gr, TC, AP, Cu on the physical-mechanical properties of nanocomposites based on the indicated polymers. It is also important to determine the combined effect of PO and the nature of the fillers on the regularity of the properties of multicomponent nanocomposites. The results of the effect of different types of fillers on the main physical-mechanical properties of nanocomposites obtained on the basis of HDPE* and at the same time, properties such as tensile yield

strength, tensile strength, elongation at break, Vicat softening temperature and flexural strength were determined.

For example, when analyzing the obtained results, it can be seen that the strength properties of the use of Gr from 1.0 to 5.0 wt % are practically little different from each other. The amount of 10 wt % of Gr causes a decrease in the strength properties of the sample. Samples containing 3.0-5.0 wt % Gr can be selected more suitable. In order to improve the compatibility of the components in the mixture, a compatibilizer (PE-g-MA) was used. YSPE* contains AP up to 5.0 wt.%. The inclusion of AP in the composition of HDPE* is accompanied by a certain increase in strength properties and a decrease in elongation at break. The inclusion of sulfur as a cross-linking agent led to a certain increase in strength indicators, elongation at break and Vicat softening temperature. Such a significant effect of sulfur on the properties of composites can be explained by the formation of a "rare mesh" structure, which has a positive effect on the change of its strength properties, as a result of the breaking of double bonds in the structure of the HDPE macrochain.

3. Theoretical basis of the influence of the segregation structure on the mechanism of electrical conductivity in polyolefin-based nanocomposites

In this section, the main focus is on obtaining polymer materials with high electrical conductivity properties. It is clear that the developed material should have complex useful properties that can be practically used in various fields of electronic industry. In this regard, in this chapter, not only the study of the electrical conductivity of nanocomposites obtained on the basis of PO, but also the results obtained from the study of the kinetic regularities of the crystallization and growth of crystals and their discussion are given. Thus, these processes are a determining factor in the formation of electrically conductive chains in TC and Gr in the interphase field in the segregated supramolecular structure of PO-based nanocomposites.

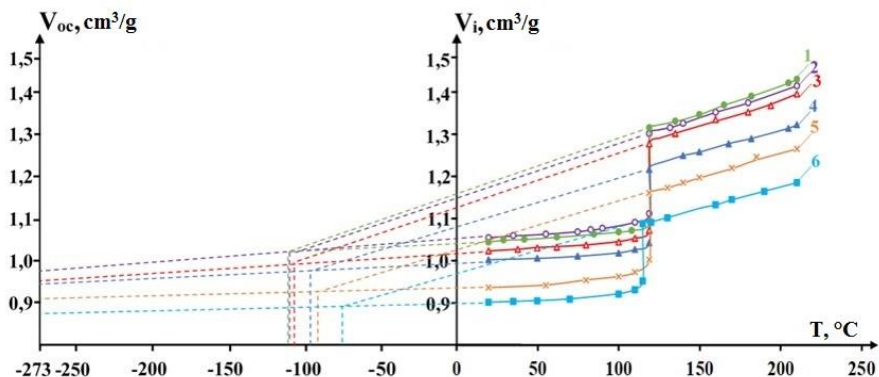


Figure 5. Dependence of specific volume on temperature for initial HDPE* (1) and its nanocomposites with different TC content: 2-1.0 wt % TC; 3-3.0 wt % TC; 4-5.0 wt % TC; 5-10 wt % TC; 6-20 wt % TC.

Figure 5 shows the results of the influence of the specific volume (V_i) of HDPE nanocomposites on the regularity of change depending on the temperature and the amount of TC determined by the stepwise dilatometry method. The amount of TC was varied in the range of 1.0-20 wt %. From the comparative analysis of the dilatometric curves shown in the figure, it can be seen that as the amount of TC increases, the dependency character of nanocomposites (V_i -T) changes with a certain regularity. During stepwise cooling from 210°C, there is a sharp jump in the specific volume change corresponding to the crystallization temperature, which is characterized as the first type of phase transition. A sharp change in the specific volume depending on the temperature indicates the transition of the nanocomposite alloy from an amorphous state to a semi-crystalline state, which is accompanied by a sharp change in the internal energy and other thermodynamic parameters of the polymer matrix. It is characteristic that in nanocomposites containing 1.0-10 wt % TC, the first type of phase transition occurs at 119°C, and in samples containing 20 wt % TC, the crystallization temperature corresponds to 115°C. Such a

decrease in the phase transition temperature for nanocomposites with relatively high TC is also consistent with the data of DTA analysis. As mentioned above, this is only possible only if steric hindrance by nanoparticles occurs. This is expressed in the creation of more perfect crystalline derivatives of macrochains, i.e., in their orientation and assembly, by the disruption of the long-range order. This situation is also explained by the fact that TC nanoparticles can exhibit structure forming properties and participate in the formation of heterogeneous nucleation centers. It can be considered that as a result of crystallization, a large number of spherulite derivatives are formed simultaneously from two centers that are different in nature, and they create steric barriers to each other during growth, leading to the formation of a small spherulite structure in nanocomposites. The dilatometric measurement method makes it possible to estimate the glass transition temperature of polymer materials. For example, if this indicator is -138°C for initial HDPE*, it is -135 , -131 , -116 , -93 and -84°C for samples containing 1.0, 3.0, 5.0, 10, 20 wt% TC. It is clear from the obtained data that the glass transition temperature exhibits a high sensitivity to the amount of TC. As its amount increases in the nanocomposite, the glass transition temperature tends to increase.

Figure 6 shows the results of the study of the kinetic regularity of the crystallization of HDPE* nanocomposites depending on the amount of TC at temperatures of 115 and 119°C in the double logarithmic coordinates of Kolmogorov-Avrami. The crystallization process in polymer composites occurs according to the Kolmogorov-Avrami empirical equation (1):

$$\varphi = e^{-K\tau^n} \quad (1)$$

Here, the φ - is the part of the polymer that has not yet turned into a crystalline phase; K - is the generalized nucleation and crystal growth constant; τ - it is time of crystallization.

n in this equation allows us to get some information about the mechanism of nucleation and crystal growth. The obtained dependences are linear, that is, they are well described by the Avrami

equation. As the content of TC in HDPE* increases, the angle of inclination to the abscissa axis in the indicated dependences slightly decreases, confirming the fact that the value of n decreases, which characterizes the type and mechanism of crystalline derivatives at the temperature of the first type of phase transition.

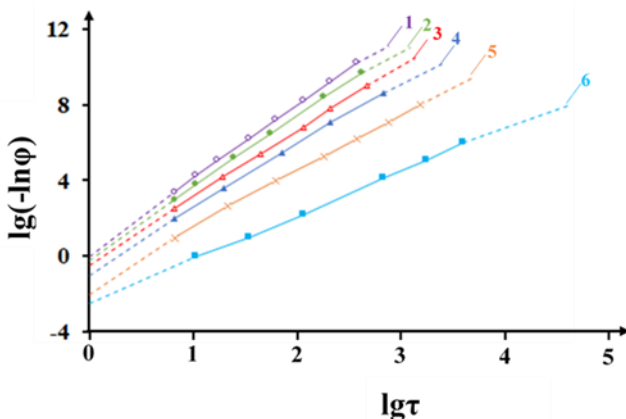


Figure 6. Kinetic regularities of crystallization of HDPE* nanocomposites in logarithmic Avrami coordinates in the field of the first type of phase transition with different TC amounts: 1-initial HDPE*, 2-1.0 wt % TC; 3-3.0 wt % TC; 4-5.0 wt % TC; 5-10 wt % TC; 6-20 wt % TC

Thus, for the initial functionalized HDPE*, the value of n is equal to 4.0, which indicates the growth of a 3D-dimensional spherulite structure from sporadic derivatives with continuous formation of homogeneous crystallization centers. In nanocomposites containing 3.0 wt% TC, the value of n decreases to 3.8, and in samples containing 5.0-10 wt% TC, it decreases to 3.6 and 3.3, respectively. When $n=3.3-3.8$ in nanocomposites, the condition of continuous formation of crystals from two nucleation centers mainly corresponds to homogeneous and heterogeneous 3D-dimensional spherulite growth type. In the content of HDPE*, in the amount of 20 wt % of TC, n is equal to 2.5, taking the lowest value. This value of n

corresponds to another mechanism of crystallization, which is characterized by the continuous formation of homogeneous and heterogeneous nucleation centers to form 2D-dimensional disk-shaped crystalline aggregates. Such a sharp decrease in the value of n in this composite once again confirms the above hypothesis that steric hindrances are formed, which significantly slow down the rate of crystallization and the growth of crystalline derivatives, the accumulation of nanoparticles in a large amount in the polymer matrix. Thus, with the help of the Kolmagorov-Avrami equation, it is possible obtain reliable information about the growth mechanism of crystalline derivatives in nanocomposites, thereby predicting possible changes in their supramolecular structure and, accordingly, properties.

By the same method, the regularities of the crystallization process of HDPE* nanocomposites containing Gr were studied, and the mechanism of crystal growth and formation was determined. It is clear that electrical conductivity is one of the physical properties that characterize the behavior of polymer materials in an electric field, their ability to conduct current depending on temperature and applied voltage. At the same time, it is necessary to determine how the electric charge is transferred from the polymer matrix (due to electron, tunnel or ion conductivity or by individual charged macromolecules). The problem becomes even more complicated when polymer based composites with different electrically conductive fillers or polar groups are used. At this time, other problems related to the formation of electrically conductive chains from filler particles in the interphase area come to the fore.

To carry out such studies, TC and Gr nanoparticles were used as fillers for PO, and information was obtained about the selective effect of TC and Gr on the electrical conductivity of nanocomposites.

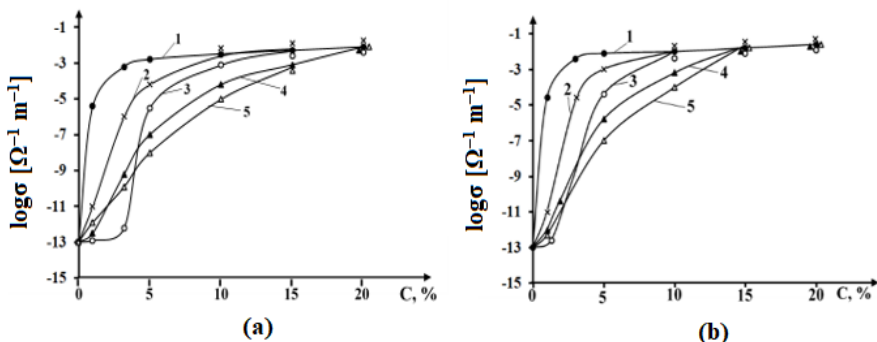


Figure 7. The effect of TC nanoparticles amount without compatibilizer (a) and with compatibilizer (b) on the electrical conductivity of nanocomposites obtained on the basis of 1-HDPE; 2-EHC; 3-LDPE; 4-PP; 5-RPP.

As can be seen from Figure 7, as the amount of TC increases, a general trend is observed in the increase of electrical conductivity of nanocomposites. According to the data obtained for some types of PO, the maximum value of electrical conductivity is obtained in the amount of 20 wt % of TC and is $10^{-2} \Omega^{-1} \text{ m}^{-1}$. From the comparative analysis of the curves in the figure, it is clear that the nanocomposites obtained on the basis of HDPE, which have the highest density and crystallinity degree among PO, show the greatest sensitivity to changes in electrical conductivity (figure 7, a, curve 1). Even with a minimal amount of TC (1.0-3.0 wt %), the electrical conductivity of the samples is characterized by relatively high results. According to the nature of the change of electrical conductivity depending on the amount of TC, the nanocomposites obtained on the basis of EHC are the closest to HDPE (Figure 7, a, curve 2). For composites containing 10 wt% TC, the value of electrical conductivity is high and is equal to $10^{-2} \Omega^{-1} \text{ m}^{-1}$. The subsequent increase of TC in the composition of HDPE and EHC does not cause noticeable changes in the value of this indicator. At the same time, it should be noted that the maximum value of electrical conductivity [$10^{-2} \Omega^{-1} \text{ m}^{-1}$] for LDPE nanocomposites is obtained in the amount of 15 wt % of TC. In the

nanocomposites obtained on the basis of PP and RPP, the maximum electrical conductivity is achieved in the amount of 20 wt % of TC. From the data presented in Figure 7 (a), it follows that the type of PO has a significant effect on the regularity of changes in electrical conductivity of nanocomposites depending on the amount of TC. At the same time, the maximum value of electrical conductivity for nanocomposites obtained on the basis of PO as a result does not exceed $10^{-2} \Omega^{-1} \text{ m}^{-1}$.

The influence of the compatibilizer on the change of electrical conductivity of nanocomposites obtained on the basis of PO was also determined. It is clear from the curves given in Figure 7 (b) that in this case the electrical conductivity changes with almost the same regularity. The main difference is that the maximum [$10^{-2} \Omega^{-1} \text{ m}^{-1}$] value of electrical conductivity in maleinated samples containing 10 wt% TC is obtained in HDPE*, EHC* and LDPE*. For nanocomposites based on PP* and RPP*, the maximum value of electrical conductivity was recorded at a relatively high amount of TC at 15 wt %. From the comparative analysis of the curves in Figure 7 (a) and 7 (b), it is clear that the maximum value of electrical conductivity for LDPE, PP and RPP composites in the presence of compatibilizer is obtained with a relatively small amount of TC.

It is quite important that the intermediate value of electrical conductivity (up to the maximum) in nanocomposites with compatibilizer is relatively high, because in the presence of a compatibilizer, a relatively small amount of TC is used to achieve the maximum effect. Using HDPE* as an example, it was determined that with an increase in the content of TC in the range of 5.0, 10, 20 wt %, the MFR of nanocomposites decreases in the following order: 2.4, 1.2, 0.5 g/10 min. The MFR for initial HDPE* is 5.6 g/10 min. In the technological aspect, these results are very beneficial for achieving high electrical conductivity with the minimum possible amount of TC in the nanocomposite.

During the study, the information about the effect of Gr nanoparticles on the electrical conductivity of nanocomposites was important for achieving high electrical conductivity of this type of

nanocomposites by keeping the MFR at a satisfactory level, reducing the value of frictional resistance observed in this case, and at the same time conducting a comparative analysis with nanocomposites containing TC.

Figure 8 (a, b) presents the results of the study of the effect of the amount of Gr on the electrical conductivity of nanocomposites based on PO such as HDPE, LDPE, EHC, RPP, PP and EPBC. It is clear from the figure that, as in TC, the value of electrical conductivity in Gr-containing nanocomposites compatibilized with PE-g-MA and PP-g-MA is relatively higher than non-compatibilized samples.

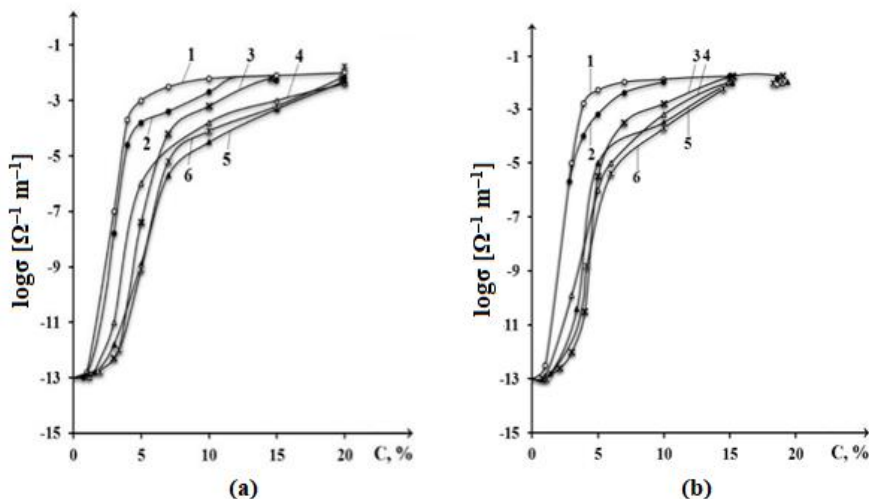


Figure 8. The effect of the amount of graphite on the electrical conductivity of PO without compatibilizer (a) and with compatibilizer (b): 1-HDPE; 2-EHC; 3-LDPE; 4-PP; 5-RPP; 6-EPBC

The maximum value of electrical conductivity in nanocomposites without a compatibilizer was obtained in the amount of 20 wt % for PP, RPP and EPBC samples, 15 wt % in LDPE, and 10 wt% of Gr in HDPE. The maximum electrical conductivity in

nanocomposites with a compatibilizer varies as follows depending on the amount of Gr: HDPE*-5 wt%; EHC*-10 wt %; 15 wt% in LDPE*, PP*, RPP* and EPBC*.

The results of DTA analysis once again confirm our arguments about the dependence of the properties of nanocomposites on the amount of filler. During the analysis of the properties of nanocomposites, it gives reason to think that a certain regularity is observed in their change. Thus, in samples with 20 wt % TC, a noticeable decrease in strength, elongation at break, crystallinity degree and melting temperature is observed.

In order to interpret the revealed regularities, the X-ray phase analysis results of the initial HDPE* and 20 wt % TC nanocomposite are presented (Figure 9). From the comparative analysis of diffractograms, it is clear that the displacements typical of HDPE* reflexes are observed in nanocomposites. With the introduction of TC, the initial HDPE* reflex is shifted, so it changes from $2\theta=21^{\circ}46'$ to $2\theta=21^{\circ}25'$, from $2\theta=24^{\circ}27'$ to $2\theta=23^{\circ}50'$. The shift in the location of the reflexes characteristic of HDPE* confirms that there is a physical interaction occurs between the TC nanoparticles and the polymer matrix. In addition, the intensity of characteristic reflexes in nanocomposites is slightly lower, which indicates a decrease in its crystallinity degree. It was found that the crystallinity degree of HDPE* decreases from 82% to 73% when only 20 wt% TC is included. A decrease in the crystallinity degree of highly filled nanocomposites or the increase in the degree of amorphousness of the material from 18 to 27%, confirms that the morphological changes occur in the molecular structure of the sample containing 20 wt% TC lead to a forced expansion of the interfacial area volume.

In other words, a 5.0 wt% increase in the amount of TC (i.e. from 15 to 20 wt%) is accompanied by a 9% increase in the volume of interfacial amorphous area. Based on the XRD results, if the degree of amorphousness of HDPE* is equal to 18%, the amount of TC nanoparticles in the interfacial area will be about 5.5 times higher than the total amount calculated for the polymer matrix.

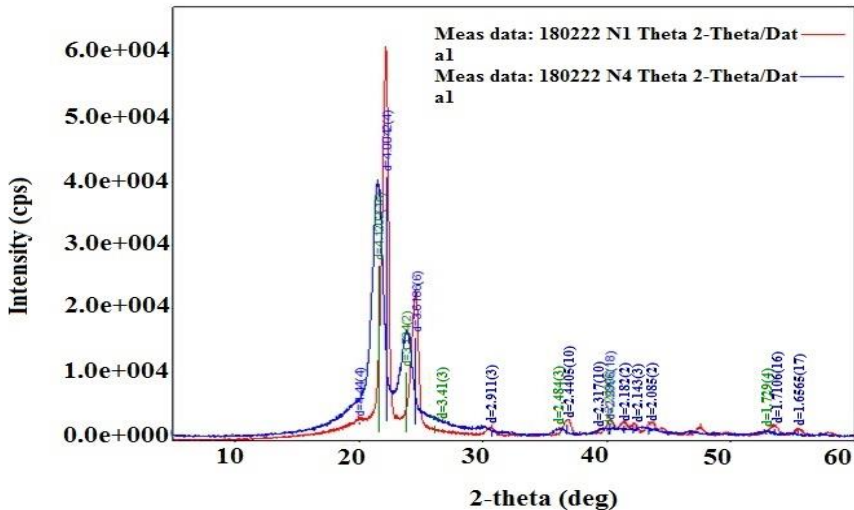


Figure 9. Diffractogram of initial HDPE* (red line) and HDPE*+20 wt% TC (blue line) nanocomposite

Accumulation of filler particles in the area between the spherulites leads to an increase in its density and, as a result, an increase in the probability of the formation of an electrically conductive chain structure (Figure 10, a, b). This is confirmed by the results of electron microscope analysis of composite samples. According to these results, in the amount of TC of 5.0 wt %, a chain electrically conductive structure is formed in the narrow area between the spherulites of HDPE* (Figure 10, a). With the increase of TC content up to 20 wt %, the density of the area between the spherulites increases so much that electrically conductive agglomerates-clusters (Figure 10, b) are formed, which keep the electron conductivity of the composites at a high level.

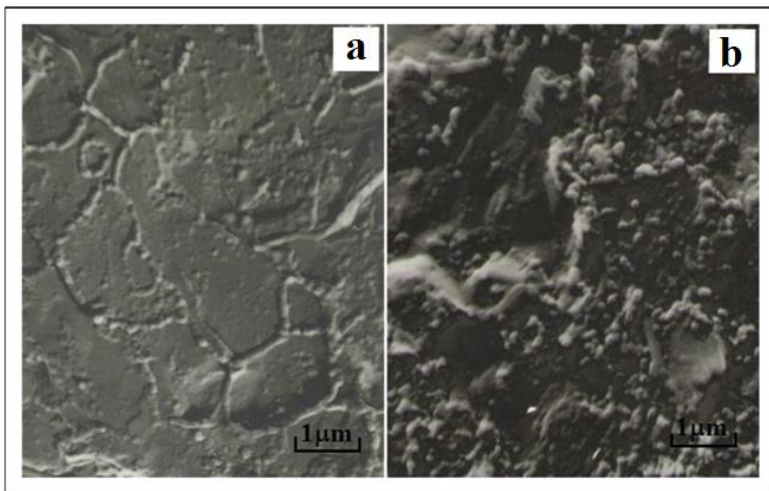


Figure 10. Electron microscope images of HDPE*+5.0 wt% TC (a) and HDPE*+20 wt% (b) nanocomposites. Magnified x15000.

In this case, TC particles are aggregates of particles interconnected by chemical bonds. Thus, henceforth, nanoparticles should be considered as **nanoaggregates**. SEM analysis of the structure of nanoparticles clearly proves this (Figure 11). It can be seen from the presented figure that the aggregates form agglomerates by grouping together. The difference is that the TC aggregate is a "cluster"-shaped discrete hard colloidal unit consisting of polydisperse particles connected by chemical (valent) bonds. They are primary aggregates, which are the smallest dispersible unit of TC, consisting of ten to a hundred particles, distinguished by their high strength.

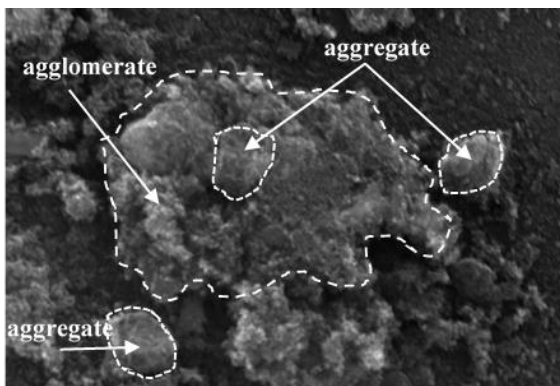


Figure 11. SEM image of TC nanoparticles (aggregates and agglomerates).

Figure 12 shows the SEM image of Gr nanoparticles. It can be seen from the figure that there are graphene layers in the Gr particles. It is known that carbon has four valence electrons. However, only three of the four electrons are involved in forming covalent bonds in graphite, so each carbon atom is bonded to only three carbons. The fourth electron in the unpaired state ensures the high electrical conductivity of graphite.

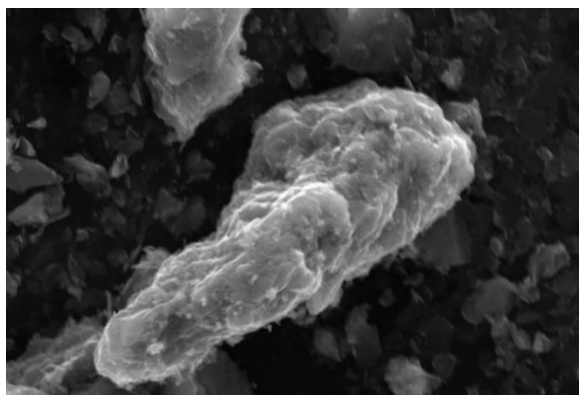


Figure 12. SEM image of graphite nanoparticles structure

According to the schematic diagram shown in Figure 13(a), the process of formation of an electrically conductive structure in the area between the spherulites of the nanocomposite begins even in the amount of 1.0-3.0 wt % of nanoparticles. At this time, a chain structure of TC(Gr) nanoparticles separated by a PO dielectric layer is formed in the interphase field. Such a structure mainly provides tunnel conductivity (Figure 13, a). Naturally, the thickness of the dielectric layer between the nanoparticles should decrease with the increase in the amount of TC(Gr) (Figure 13, b). Finally, a further increase in the amount of TC(Gr) increases the probability of direct contact of nanoparticles with each other and leads the formation of mixed-electron and tunnel conductivity (Figure 13, c). In this case, the probability of formation of a "chain structure" in which electronic conductivity is predominant increases. Even if there is a 0.5-1.5 nm layer between the nanoparticles, this should be considered as electronic conductivity. Most likely, when the content of TC(Gr) reaches 10 wt % in HDPE, EHC and LDPE (Figure 7 and 10), "chain clusters" are formed, thanks to which the maximum value of electrical conductivity in nanocomposites is obtained mainly due to electronic conductivity (Figure 13, d). The electrical conductivity remains unchanged during further increase of the amount of TC(Gr) up to practically 20 wt %. The scheme of formation of nanoparticle agglomerates shown in Figure 13(e) corresponds to this variant. In PP and RPP, a similar effect is achieved in the amount of 15 wt % of nanoparticles. The structure variant shown in Figure 13(d) corresponds to the maximum electrical conductivity. At this time, the whole structure of the chain clusters of nanoparticles is formed, which ensures the electronic conductivity of nanocomposites. This is proven by the fact that the electrical conductivity of nanocomposites does not change with the further increase of the amount of filler (above 10-15 wt %). In other words, on the basis of HDPE, EHC and LDPE, the degree of saturation of the conductive material is obtained in the amount of 10-15 wt % of TC and Gr.

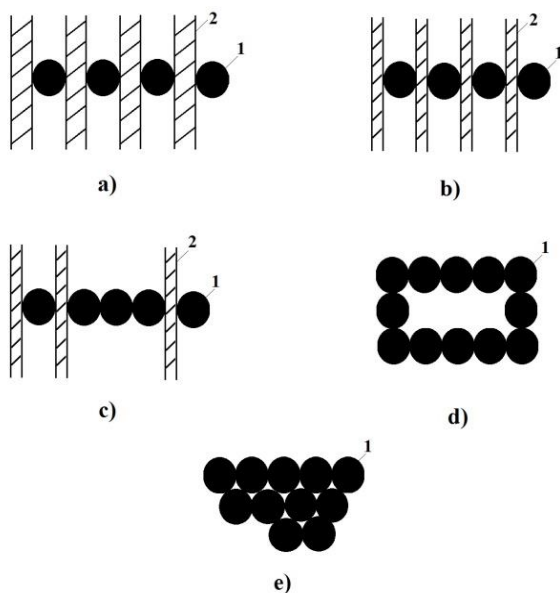


Figure 13. Schematic representation of the formation of electronic and tunneling conductivity in nanocomposites obtained on the basis of PO-TC(Gr): a) nanoparticles (1) separated by a thick dielectric layer (2); b) nanoparticles (1) separated by a thin dielectric layer (2); c) mixed-electron (1) and tunnel (2) conductivity; d) chain clusters; e) agglomerates of nanoparticles (1)

The effect of mechanical activation of TC and Gr nanoparticles on the change of electrical conductivity, values of tensile strength and elongation at break of nanocomposites based on a wide range of PO was considered. It was determined that as a result of mechanical activation of TC nanoparticles, their scattering density decreases by 12 times. The possibilities of improving the properties of nanocomposites by grinding the solid particles of the filler in a mill have been shown.

4. Segregated structure and properties of electrically conductive thermoplastic elastomers (elastoplastics) based on polyolefins, technical carbon, graphite

In this section, the structural properties, electrical conductivity, physical-mechanical and thermal deformation properties of nanocomposites obtained on the basis of thermoplastic POs HDPE, LDPE, PP, RPP, EHC, EBC and EPBC, as well as ethylene-propylene-diene rubber (EPDE-40) and butadiene-styrene copolymer (SKS-30) elastomers were discussed. TC, Gr and metal were used as fillers. Naturally, it was important to determine the optimal amount of mixing components that could maintain relatively good physical-mechanical properties along with high electrical conductivity properties of nanocomposites. Table 3 shows the physical-mechanical properties of elastoplastics obtained on the basis of compatibilized PO and SKS-30. It can be seen that the strength properties deteriorate as the amount of elastomer increases, regardless of the type of PO. It should also be noted that phase inversion occurs in a certain proportion of components in each composition.

It has been shown that when the value of the tensile strength in the "thermoplastic-elastomer" mixture is equal to the tensile yield strength, the transformation of the dispersed medium into the dispersed phase or vice versa, the moment of inversion occurs. This moment can be clearly determined during the study of the properties of the polymer mixture, such as tensile strength and tensile yield strength. It is at the moment of phase inversion that the polymer mixture in the system acquires the properties characteristic of rubber, at which time the differences in strength indicators practically disappear. However, depending on the type of PO, the formation of TPE occurs at different amounts of SKS. For example, in HDPE, EHC and EBC, this effect occurs in the amount of 30 wt % of SKS, in PP, RPP and EPBC, the properties of TPE are 40 wt % of SKS-30, and only in LDPE, the properties of SKS-30 it is obtained in the amount of 20 wt %.

Table 3

**Physical-mechanical properties of TPE obtained on the basis of
PO+SKS-30+PE-g-MA(PP-g-MA) mixture**

№	The amount of SKS-30 in the PO, wt%	Tensile strength, MPa	Tensile yield strength, MPa	Elongation at break, %	Bending strength limiti, MPa	MFR, g/10 min	Vicat softening temperature, °C	Jung's module, MPa
1	HDPE	31.3	29.4	435	34.4	5.6	139	1150
2	HDPE+10SKS+2C ₁	29.5	27.0	435	33.8	7.3	138	725
3	HDPE+20SKS+2C ₁	23.6	23.0	455	29.6	6.2	132	412
4	HDPE+30SKS+2C₁	18.6	18.6	505	25.7	5.5	128	166
5	HDPE+40SKS+2C ₁	14.2	14.2	410	20.5	4.1	119	83
6	HDPE+50SKS+2C ₁	11.7	11.7	350	16.6	3.0	110	-
7	LDPE	9.6	9.0	360	16.2	7.8	85	211
8	LDPE+10SKS+2C ₁	8.9	8.3	370	14.7	9.0	83	181
9	LDPE+20SKS+2C₁	6.4	6.4	370	11.2	7.8	75	104
10	LDPE+30SKS+2C ₁	5.2	5.2	355	8.5	6.2	69	52
11	LDPE+40SKS+2C ₁	4.0	4.0	330	6.6	4.6	65	-
12	LDPE+50SKS+2C ₁	3.3	3.3	265	4.2	3.3	61	-
13	PP	33.0	34.6	30	85.0	3.6	160	1424
14	PP+10SKS+2C ₂	31.1	32.8	45	86.2	5.4	157	1132
15	PP+20SKS+2C ₂	27.5	28.7	55	70.4	4.6	149	543
16	PP+30SKS+2C ₂	20.8	21.6	70	46.6	3.8	136	255
17	PP+40SKS+2C₂	16.7	16.7	55	38.4	3.0	129	188
18	PP+50SKS+2C ₂	12.8	12.8	55	32.3	2.5	123	-
19	EHC	37.4	35.8	810	42.2	5.1	115	1234
20	EHC+10SKS+2C ₁	34.2	32.9	810	40.3	7.4	113	825
21	EHC+20SKS+2C ₁	28.7	27.6	800	36.6	6.1	108	444
22	EHC+30SKS+2C₁	21.2	21.2	800	30.2	4.9	101	203
23	EHC+40SKS+2C ₁	17.5	17.5	560	24.5	3.7	96	95
24	EHC+50SKS+2C ₁	14.6	14.6	455	18.8	2.6	93	-
25	EBC	27.1	28.4	880	38.2	4.6	113	1115
26	EBC+10SKS+2C ₁	24.5	23.3	805	36.9	6.2	111	811
27	EBC+20SKS+2C ₁	19.3	18.5	715	33.5	7.0	105	405
28	EBC+30SKS+2C₁	15.7	15.7	710	26.6	4.5	101	176
29	EBC+40SKS+2C ₁	12.3	12.3	720	19.3	3.2	98	88
30	EBC+50SKS+2C ₁	9.6	9.6	680	14.9	2.8	90	-
31	RPP	28.5	26.4	600	30.4	1.8	131	802
32	RPP+10SKS+2C ₂	25.8	23.3	600	28.6	3.7	128	442
33	RPP+20SKS+2C ₂	22.4	21.5	650	24.2	3.2	122	198
34	RPP+30SKS+2C ₂	17.2	16.9	675	20.3	2.7	117	126

Continuation of Table 3

35	RPP+40SKS+2C₂	13.3	13.3	525	17.3	2.2	111	77
36	RPP+50SKS+2C₂	10.5	10.5	355	13.2	1.7	106	-
37	EPBC	25.6	26.9	200	26.5	2.9	148	712
38	EPBC+10SKS+2C₂	23.1	24.4	210	24.8	4.9	145	423
39	EPBC+20SKS+2C₂	19.8	21.2	235	21.2	4.2	141	219
40	EPBC+30SKS+2C₂	15.1	16.0	250	17.4	3.5	136	118
41	EPBC+40SKS+2C₂	11.7	11.7	210	13.5	2.8	130	79
42	EPBC+50SKS+2C₂	9.6	9.6	155	10.2	2.0	122	-

C₁ – PE-g-MA; C₂ – PP-g-MA

In confirmation of the above, the results of SEM analysis of polymer mixtures containing different amounts of SKS can be shown. For example, in Figure 14 (a, b, c, d), SEM images of polymer blends based on HDPE*+SKS are given. It can be seen from the images that the surface structure of the samples changes significantly when the amount of SKS increases from 10 to 40 wt %. These changes, as shown above, were observed in samples containing 30 wt % of SKS, characterized by the properties of TPE (Figure 14, c).

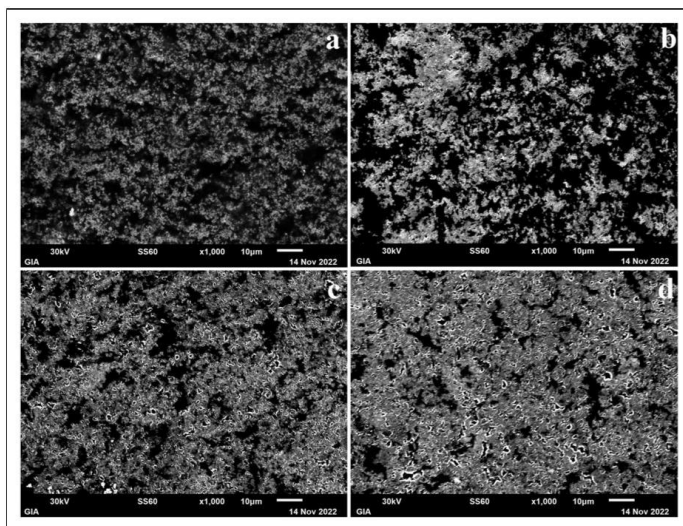


Figure 14. SEM image of TPE blend based on HDPE* with different amount of SKS: a-10 wt % SKS; b-20 wt % SKS; c-30 wt % SKS; d-40 wt % SKS

The emergence of rubber-like features in the mixture of 30 wt % and higher amounts of SKS from SEM images (Figure 14, c, d), in fact, confirms our arguments about the occurrence of phase inversion, that is, the transformation of HDPE* from a dispersed medium to a dispersed phase.

For comparison, the results of DSC analysis of HDPE* polymer mixtures containing different amounts of SKS are given in Figure 15. Based on the data obtained from the study, the melting enthalpy (ΔH), J/g, was calculated. As can be seen from the figure, the melting enthalpy decreases synchronously in the following order depending on the amount of SKS (10, 20, 30, 40 wt %): (initial HDPE*) \rightarrow 177.5; (10%) \rightarrow 148.9; (20%) \rightarrow 133.5; (30%) \rightarrow 112.1; (40%) \rightarrow 91.4; (50%) \rightarrow 68.3 (J/g).

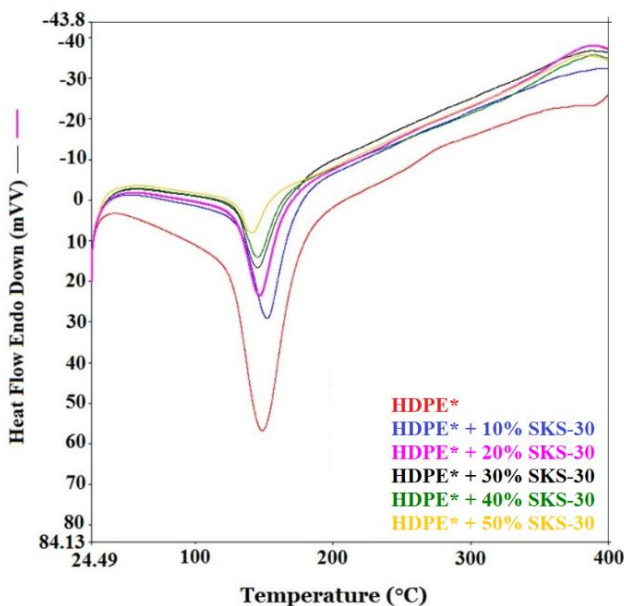


Figure 15. DSC curves of HDPE*+SKS-30 polymer blends

From the analysis of the curves in the figure, it is clear that as the amount of SKS increases, the height and enthalpy of the melting peak of the polymer mixture decrease, indicating the amorphization of the crystal structure of the composite. In addition, it was determined that when the amount of SKS increases in the range of 10, 20, 30, 50 wt %, the initial, peak and final temperature of melting changes. The culmination of the development process of TPEs is vulcanization of polymer mixtures with sulfur (table 4). In this case, we excluded the possibility of using vulcanization in the presence of peroxide, because the formation of a dense network structure in TPE has a significant effect on the sharp reduction of MFR, as a result of which their processing is practically impossible.

Table 4
Physical-mechanical properties of dynamically vulcanized TPEs
with sulfur based on PO + SKS-30 + compatibilizer + sulfur
compositions

№	The formulation of the composition, wt%	Tensile strength, MPa	Elongation at break, %	Bending strength limit ^{*,} MPa	MFR, g/10 min	Vicat softening temperature, C°
1	HDPE+30SKS+2C ₁ +3S	20.8	555	27.4	4.4	130
2	HDPE+30SKS+2C ₁ +5S	21.0	550	28.0	3.0	132
3	LDPE+20SKS+2C ₁ +3S	8.4	430	12.8	6.2	77
4	LDPE+20SKS+2C ₁ +5S	8.7	450	13.1	4.5	78
5	PP+40SKS+2C ₂ +3S	18.7	75	40.5	2.4	132
6	PP+40SKS+2C ₂ +5S	18.5	75	40.8	0.7	134
7	EHC+30SKS+2C ₁ +3S	23.7	800	32.6	3.8	104
8	EHC+30SKS+2C ₁ +5S	24.0	800	32.9	2.5	104
9	EBC+30SKS+2C ₁ +3S	18.4	810	28.3	2.2	102
10	EBC+30SKS+2C ₁ +5S	18.6	780	29.0	1.7	102
11	RPP+40SKS+2C ₂ +3S	15.8	600	20.2	1.6	114
12	RPP+40SKS+2C ₂ +5S	16.1	600	20.9	0.5	116
13	EPBC+40SKS+2C ₂ +3S	13.9	250	16.8	2.1	133
14	EPBC+40SKS+2C ₂ +5S	14.2	270	17.5	0.7	134

*S- sulfur, C₁ – PE-g-MA; C₂ –PP-g-MA

As can be seen from Table 4, their cross-linking with sulfur leads to a noticeable improvement in strength properties, elongation at break and Vicat softening temperature. The process of cross-linking with sulfur in polymer mixtures took place as a result of their preparation in a hot rolling mill. It should be noted that, ultimately, the main technological characteristic of TPE is MFR, which characterizes its ability to be processed by pressure casting or extrusion. Therefore, when choosing the recipe of vulcanizates, first of all, it is necessary to take into account the technological features of their processing.

Let's refer to Figure 16 (a, b) to get a complete idea about the role of the degree of crystallinity of the primary polymer matrix and the type of filler on the mechanism of the electrical conductivity of nanocomposites and the regularity of change.

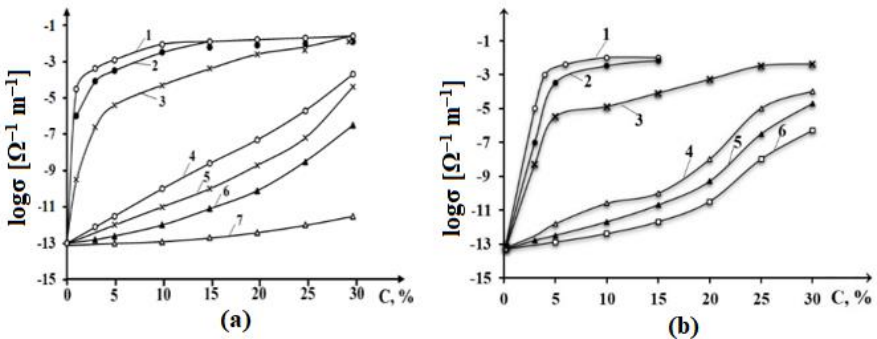


Figure 16. (a, b). The effect of the amount of TC (a) and Gr (b) on the electrical conductivity of HDPE*+EPDE compatibilized polymer blends: 1-HDPE*; 2-HDPE*+10 wt % EPDE; 3-HDPE*+30 wt % EPDE; 4- HDPE*+40 wt% EPDE; 5- HDPE*+50 wt% EPDE; 6- HDPE*+70 wt% EPDE; 7- EPDE

As can be seen from the figures, polymer mixtures based on compatibilized HDPE*+EPDE were used as the polymer matrix. The amount of TC and Gr for all samples was 30 wt %. The amount of

EPDE in HDPE* was 10, 30, 40, 50, 70 wt %. From the comparative analysis of the curves in Figure 16 (a, b), it can be seen that the electrical conductivity of nanocomposites decreases significantly with the increase in the amount of amorphous rubber component in the HDPE.

For clarity, schematic images of the processes occurring in the interphase area of TPE nanocomposites are presented in Figure 17.

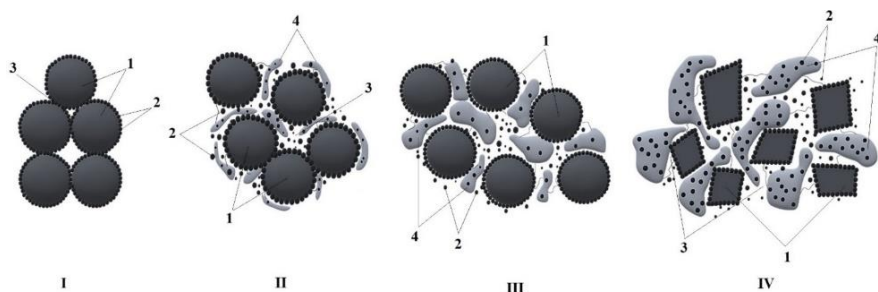


Figure 17. Schematic illustration of redistribution of nanoparticles (TC or Gr) and elastomeric component (EPDE or SKS) in the interspherulite area of TPE after vulcanization with sulfur. 1- crystal area; 2- nanoparticles; 3- interfacial area; 4- cross-linked elastomer. Amount of elastomeric component: I- elastomer-free polyolefin nanocomposite; II-TPE nanocomposite containing 10-20 wt% elastomer; III-TPE nanocomposite containing 30-40 wt% elastomer; IV-nanocomposite containing 50 wt % elastomer.

As can be seen from the figure, as the amount of elastomeric component in the TPE content increases, the interfacial area expands, which results in the partial disruption of the chain structure of the nanoparticles, leading to a decrease in the tunnel electrical conductivity of the nanocomposites. As mentioned above, during the sulfur vulcanization process of TPE nanocomposites, the cross-linking

takes place mainly in the elastomeric phase. For this reason, it can be assumed that cross-linking elastomers in TPE will appear as some kind of "islands" in the dispersed medium of PO. As a result of the cross-linking process in the elastomer phase, it is not excluded that a part of the nanoparticles is "occluded" in the vulcanizates.

For comparison, the effect of TC and Gr fillers on the electrical conductivity properties of HDPE*+SKS polymer blends was studied and it was determined that at this time they change according to approximately the same regularity as in nanocomposites obtained on the basis of HDPE*+EPDE-40 TPE. It can be considered that exactly 40 wt % amount of EPDE-40 or SKS-30 in HDPE* causes difficulty in tunneling. Predominance of relatively thickened dielectric layers leads to the disruption process in the formation of the chain structure of nanoparticles. At the same time, with an increase in the amount of the elastomeric component in the mixture, it is necessary to add a significantly larger amount of TC and Gr to maintain the tunnel permeability.

The influence of the amount of TC and Gr on the electrical conductivity of nanocomposites based on SKS-30+PO* or EPDE-40+PO* mixtures was studied. The use of different types of elastomer made it possible to evaluate the role of polar and non-polar components in the change properties of electrical conductivity of nanocomposites.

Vulcanized TPEs containing a minimum amount of sulfur (3.0 wt %) were chosen as the object of research, which makes their processing convenient and can keep the viscosity of the alloy at a relatively low level. From the analysis of the data in Table 5, it is clear that regardless of the type of primary PO, the introduction of fillers (TC, Gr, AP and CS) leads to an increase in the strength properties of TPE nanocomposites. The latter case suggests that one of the functions of the considered fillers is to enhance the strength properties of TPE. As can be seen from the table, the electrical conductivity properties are also maintained at a high level during vulcanization with sulfur.

Table 5

Physical-mechanical and physical-chemical properties of dynamically vulcanized multifunctional TPE nanocomposites based on maleinated PO, SKS, TC, Gr, AP and CS with sulfur

Composition of nanoelastoplastic , wt%	Electrical conductivity, $\Omega^{-1} \text{ m}^{-1}$	Thermal conductivity coefficient, W/m·K	Peel resistance, g/cm	Tensile strength, MPa	Bending strength limiti., MPa	MFR, g/10 min
LDPE+30SKS+2C ₁ +20TC+1AP+1CS+3S	$4.6 \cdot 10^{-3}$	3.5	52	27.8	31.8	2.2
LDPE+20SKS+2C ₁ +20TC+1AP+1CS+3S	$7.9 \cdot 10^{-4}$	3.3	48	13.5	15.9	3.5
PP+40SKS+2C ₂ +20TC+1AP+1CS+3S	$2.9 \cdot 10^{-3}$	3.4	53	26.4	45.5	1.4
EHC+30SKS+2C ₁ +20TC+1AP+1CS+3S	$7.3 \cdot 10^{-3}$	3.6	51	30.8	40.6	2.1
RPP+40SKS+2C ₂ +20TC+1AP+1CS+3S	$2.8 \cdot 10^{-3}$	3.3	49	24.7	29.8	0.9
EPBC+40SKS+2C ₂ +20TC+1AP+1CS+3S	$2.3 \cdot 10^{-3}$	3.1	52	21.5	21.3	1.3
HDPE+30SKS+2C ₁ +20Gr+1AP+1CS+3S	$6.6 \cdot 10^{-3}$	3.3	30	25.5	29.6	3.2
LDPE+20SKS+2C ₁ +20Gr+1AP+1CS+3S	$4.3 \cdot 10^{-3}$	3.5	31	11.6	14.5	5.4
PP+40SKS+2C ₂ +20Gr+1AP+1CS+3S	$3.3 \cdot 10^{-3}$	3.2	31	24.5	41.8	3.9
EHC+30SKS+2C ₁ +20Gr+1AP+1CS+3S	$8.3 \cdot 10^{-3}$	3.3	29	29.5	38.7	4.4
RPP+40SKS+2C ₂ +20Gr+1AP+1CS+3S	$1.7 \cdot 10^{-3}$	3.5	31	23.8	26.5	1.8
EPBC+40SKS+2C ₂ +20Gr+1AP+1CS+3S	$0.8 \cdot 10^{-3}$	3.5	32	20.8	20.0	2.2

C₁- PE-g-MA; C₂- PP-g-MA; TC- technical carbon, Gr- graphite; AP- aluminum powder; CS- calcium stearate; S-sulfur.

In addition, the MFR is considered an important technological indicator of nanocomposites and is distinguished in high value in nanocomposites containing Gr. This is explained by the fact that Gr has a layered structure and as a result of the thermomechanical effect in the alloy, it is broken into smaller particles for the improvement of the MFR index of nanocomposites.

Another important indicator is the thermal conductivity of the cross-linked TPE nanocomposites, which is significantly increased by the application of AP nanoparticles. It is enough to note that if the thermal conductivity of the initial PO varies in the range of 0.25-0.48 W/m·K, according to the data in table 5, the thermal conductivity of TPE nanocomposites with the inclusion of AP in the composition of the nanocomposite increases by 20 times on average, to 3.1-3.6 W/m·K increases significantly. At the same time, AP nanoparticles play the biggest role in increasing the thermal conductivity, because its thermal conductivity is 230 W/m·K, while in dry TC this indicator is only 0.07-0.12 W/m·K. Analyzing Table 5, it can be determined that compared to nanocomposites containing TC, samples containing Gr are characterized by relatively low values for peel resistance. This situation is explained by the fact that Gr-containing composites belong to the category of friction-resistant materials due to their nature. There is a certain analogy: the higher the friction resistance of the polymer composite, the lower the adhesion forces in the adhesive-substrate system.

5. Technological characteristics of the mechanical-chemical synthesis of multicomponent electrically conductive nanocomposites based on polyolefins

Technological modes of pressure casting process for a number of nanocomposites based on HDPE, LDPE, RPP and EPBC were developed. It has been shown that with the increase of the casting pressure, the temperature regime of the material cylinder, and the time of storage under pressure, an increase in the strength properties and elongation at break of nanocomposites is observed. As a filler, TC, AP were used. The influence of the location of the sprue relative to the

samples on the formed part of the press mold was taken into account. It was determined that the samples located longitudinally in the formed part of the mold are characterized by relatively better physical-mechanical properties.

The influence of the temperature regime of the material cylinder, the screw speed, the effect of the extrusion pressure, the values of the tensile strength, elongation at break and the MFR of the nanocomposites were obtained.

During the dynamic vulcanized reaction "monotrem" extrusion process, depending on the amount of sulfur and peroxide, the possibility of obtaining new types of polymer materials with isotropic properties of nanocomposites has been shown.

The effect of vulcanization agent (sulfur) on reactive extrusion process of nanocomposites based on RPP and EPBC was considered. Relatively high values of the properties of vulcanized nanocomposites were obtained, and the optimal amount of sulfur was determined. Studying the tensile strength and elongation at break of the sheet material along and across of the extrusion made it possible to determine that the vulcanization of the nanocomposites contributes to the isotropy of the properties.

For the first time, the classification of multifunctional electrically conductive nanocomposites obtained by mechanical-chemical synthesis as a result of the conducted research was given. It has been shown that, depending on the composition of nanocomposites and the amount of components, the obtained nanocomposites can be used in various fields of electronics as conducting, semiconducting and antistatic materials. Thus, it can be noted that the nanocomposites prepared by us can be divided into 3 classes according to their electrical conductivity properties as follows: conductors - $\sigma=10^{-2}-10^{-3} \Omega^{-1} \text{ m}^{-1}$, semiconductors - $\sigma=10^{-4}-10^{-6} \Omega^{-1} \text{ m}^{-1}$, antistatics - $\sigma = 10^{-6}-10^{-9} \Omega^{-1} \text{ m}^{-1}$.

MAIN RESULTS

1. Selective effect of metals (aluminum and copper) on the complex physical-mechanical, rheological, adhesion and thermal-physical properties of nanocomposites based on a wide range of compatibilized HDPE*, LDPE*, PP*, EHC*, EBC*, RPP*, EPBC* (PO) was studied. The main possibilities for increasing the strength properties, thermal conductivity and peel resistance of metal-filled systems from copper and aluminum substrate surfaces are shown and the mechanism of adhesive-substrate adhesion contact is analyzed [2-21, 24, 29-31, 33, 34, 40, 42, 44, 45].
2. Among the different types of TC and graphite, the most effective conductive fillers were determined: Printex XE 2B branded TC and GC-2 brand of graphite. For the first time, it has been shown that the amount of PE-g-MA, PP-g-MA and TK has a decisive influence on the formation of the segregated structure of a wide variety of PO-based nanocomposites. The regularities of changes in tensile yield strength, tensile strength, elongation at break, flexural strength limit, MFR and Vicat softening temperature of electrically conductive nanocomposites were systematically analyzed. It was determined that the best strength properties were obtained by adding 5.0 wt % TC to the composition of HDPE*, PP*, RPP*, EHC*, EBC*, EPBC. In composites based on LDPE*, this effect is achieved in the amount of 10-15 wt % of TC. Thermomechanical properties of nanocomposites based on HDPE and TC were studied. It is shown that the temperature of the first type of phase transition increases from 138 to 142°C with the increase of TC content from 1.0 wt% to 20 wt%. Derivatographic analysis method showed that when the amount of TC increases from 1.0 wt % to 20 wt % the value of the melting temperature of nanocomposites changes in the following order: 148, 146, 149, 143°C [41, 47, 51, 52].

3. The influence of the amount of graphite on the formation of the segregation structure of nanocomposites was studied, and a systematic analysis of the physical-mechanical properties of nanocomposites compatibilized on the basis of PO was carried out. It has been shown that the best strength properties in nanocomposites based on POs are obtained in the amount of 3.0-5.0 wt % of graphite. In this case, the maximum effect in nanocomposites based on LDPE* is recorded at 15 wt % of graphite [47, 51, 52].
4. The combined effect of TC and graphite, AP and copper content on the physical-mechanical properties of multicomponent nanocomposites based on compatibilized POs, such as tensile yield strength, tensile strength, elongation at break, Vicat softening temperature, and bending strength limit, was studied. It has been shown that the maximum values of strength properties of multicomponent nanocomposites are regulated by changing the ratio of components included in the composition of the used PO. It was found that the use of PE-g-MA and PP-g-MA compatibilizer in multicomponent filled systems based on PO allows to maintaining the strength properties at a relatively high level. At the same time, the introduction of calcium stearate or zinc stearate in the amount of up to 1.0 wt % improves the technological properties of multicomponent nanocomposites and maintains a sufficiently good level of MFR. In addition, it was determined that the use of vulcanization process in the presence of sulfur (3.0-7.0 wt %) and peroxide (up to 0.5 %) in multicomponent nanocomposites significantly improves their strength properties, elongation at break and Vicat softening temperature [36, 49].
5. The effect of the amount of TC on the peel resistance of AP-containing composites based on PO was studied and the maximum value of adhesion strength is achieved in the range of 5.0-12 wt % TC. The use of compatibilizer significantly increases the peel resistance of composites. The influence of the pressing temperature regime on the type of adhesion

breakdown of nanocomposites based on PO on aluminum and copper foil surfaces was determined. It is shown that, regardless of the type of PO used, the simultaneous use of TC and Al or TC and Cu as fillers leads to a significant increase in the cohesion breakdown type [32, 37, 39, 40, 42].

6. New scientific approaches for studying of the formation mechanism of electrical conductivity in the segregated structure of nanocomposites for the PO class have been presented. Using a wide variety of PO and compatibilizers as examples (PE-g-MA and PP-g-MA), the selective effect of the amount of TC and Gr on the electrical conductivity of nanocomposites was determined for the first time. The results of XRD, electron microscopy, and SEM analysis of the structure of nanocomposites allow us to get a fairly clear idea of the mechanism of formation of tunneling or electron conduction in them. Based on the received data, 3 classes of nanocomposites with different electrical conductivity were differentiated: conductors $\sigma=10^{-2}-10^{-3} \Omega^{-1} \text{ m}^{-1}$, semiconductors $\sigma = 10^{-4}-10^{-6} \Omega^{-1} \text{ m}^{-1}$ and antistatics $\sigma = 10^{-7}-10^{-9} \Omega^{-1} \text{ m}^{-1}$ [1, 23, 38, 46, 47, 52, 53].
7. Using HDPE as an example, it was shown by the stepwise dilatometry method that the amount of TC and Gr has a significant effect on the first-type of phase transition and the kinetic regularities of the crystallization of electrically conductive nanocomposites. According to the Kolmagorov-Avrami equation, the crystal growth mechanism in nanocomposites was studied depending on the amount of TC and Gr and clarified that with an increase in the amount of nanoparticles from 1.0 to 20 wt %, the glass transition temperature (second type phase transition) increases from -110 to -15°C, and the crystal growth mechanism is 3D (three-dimensional) with the continuous formation of crystallization centers ranging from spherulite to 2D (two-dimensional) disc-like [1, 46, 48, 50].

- 8.** The study of thermal-physical properties of nanocomposites by DTA method showed that their melting and thermal decomposition temperatures increase with the increase of TC and graphite content in HDPE* from 1.0 to 15 wt %. However, in the amount of TC and graphite in the amount of 20 wt %, the opposite process is observed, accompanied by a simultaneous decrease in the melting and thermal destruction temperatures of nanocomposites. The effect of mechanical activation of TC and graphite nanoparticles on the change of electrical conductivity, tensile strength and elongation at break of PO based nanocomposites was studied, and a 12-fold decrease in the scattering density of TC nanoparticles was recorded. The main principle possibilities of improving the properties of nanocomposites by means of mechanical activation of solid filler particles in a planetary mill have been shown [35, 41, 46, 48, 50, 52].
- 9.** It was determined that the inclusion of SKS-30 or EPDE-40 elastomeric component in the composition of PO leads to the preparation of TPE, characterized by a region of high elastic deformation characteristic of rubber. A regular decrease in the crystallinity degree, strength, rheological and thermal-physical properties was observed with the increase of the amount of SKS in TPE. The results of thermomechanical studies showed that at certain ratios of PO and elastomer, phase inversion occurs with the simultaneous formation of a highly elastic state region characteristic of rubber. Construction of the stress-strain diagram for the composite based on HDPE+SKS confirmed that 30 wt % and more of the elastomer component leads to the formation of an S-type dependence characteristic of rubber [43, 47, 49, 51, 52].
- 10.** Vulcanization of filled and unfilled TPE in the presence of sulfur significantly improves of their strength and thermo-physical properties. The combined use of TC or graphite nanoparticles in vulcanized TPE, as well as AP and CS, allowed to obtain multifunctional dynamic vulcanized conductive

- nanocomposites, distinguished by high strength properties, thermal conductivity, resistance to heat and friction [43, 48, 50].
11. It has been shown that the electrical conductivity of TPE nanocomposites with different thermoplastic-elastomer ratios is affected by the amount of TC or graphite, and with an increase in the elastomer component, a significant decrease in the electrical conductivity of TPE nanocomposites is observed. Therefore, to maintain the electrical conductivity of TPE nanocomposites at a high level, a relatively large amount of TC or graphite is required. The results of electron microscopy, DTA, stepwise dilatometry, XRD, SEM, physical-mechanical and thermal deformation studies results, have allowed the development of new scientific approaches for the interpretation of the variable segregation structure of the interfacial area of TPE depending on the ratio of mixing components [46, 49].
 12. The technological modes of pressure casting process for a number of electrically conductive nanocomposites based on HDPE*, LDPE* and EHC* have been developed. It has been shown that when TC, AP and compatibilizer are used as fillers, the strength properties and elongation at break of nanocomposites increase to a certain extent with the increase of casting pressure, material cylinder temperature regime, mold temperature and storage time under pressure [26, 28].
 13. A new type of dynamically vulcanized polymer materials with isotropic properties were obtained as a result of the effect of the technological mode of extrusion process of electrically conductive nanocomposites based on RPP*, EPBC* and TC in a certain amount of vulcanizing agents such as sulfur and peroxide. The optimal temperature regime of the material cylinder, screw speed and the optimal values of the extrusion pressure were determined, which provide relatively high indicators of the tensile strength and elongation at break of the nanocomposites. According to the mutual cooperation agreement, industrial tests were carried out at "METAK" LLC, one of the leading producers of plastic products in Azerbaijan.

Positive test reports and laboratory regulations on the purchase of electrically conductive nanocomposites have been presented [22, 26, 36].

14. Classification of multifunctional electrically conductive nanocomposites based on PO and TPE, as well as recommendations for their use, are presented. According to the presented classification, the first place in terms of electrical conductivity is occupied by compatibilized PO nanocomposites containing 5.0-10 wt% TC or graphite, for which the electrical conductivity is equal to $10^{-2} \Omega^{-1} \text{ m}^{-1}$. Prepared electrically conductive nanocomposites belong to the category of structural materials for electronic and radio engineering industry, machine building, aviation, military and space technology. The prepared multifunctional conductive TPE nanocomposites can be recommended for use as anti-radar coatings, photoelectric generators, batteries, organic solar cells, flexible and transparent displays, electromagnetic screens, and antistatic materials [1, 46, 47, 50, 51].

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