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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

OBTAINING AND STUDY OF NANOCOMPOSITES BASED ON METAL OXIDES (Cu_2O, ZnO) STABILIZED BY A MATRIX OF MALEINIZED HIGH-PRESSURE POLYETHYLENE

Speciality:2318.01-Chemistry and technology of composite materials

Field of science: chemistry

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Sumgait – 2022

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GENERAL OVERVIEW OF THE WORK

Relevance and degree of topic elaboration. One of the priority areas of modern engineering and technology is the development of new polymer composite materials. Combining the properties of polymer matrices with such valuable properties as film formation, mechanical strength, chemical resistance and corrosion resistance in one composite makes the resulting material even more promising. At present, such materials can be used in aviation, instrument making, medicine, and other high-tech areas.

The study of the properties of systems containing nanoscale particles is very interesting and important both from the point of view of fundamental science and from the point of view of practical application of such systems in a number of new technologies. Nanoobjects occupy an intermediate position between bulk materials and atoms and exhibit new physical and chemical properties characteristic only of the nanoscale state of matter. The study of the technology of obtaining and properties of new nanocomposites is an urgent problem, which has led to an increased interest of researchers in nanomaterials.

Among the directions of intensive development of nanomaterials, the main one is the production of nanocomposite materials based on organic polymer matrices and nanoparticles of various compounds. Of particular interest is the use of metal nanoparticles or metal oxides. It has to do with the fact that due to the dimensionality effect of metal nanoparticles and the specific properties of stabilizing organic compounds, the resulting nanocomposites have complex physicochemical and biological properties. The inclusion of metal nanoparticles or metal oxides in polymers as additives makes it possible to obtain polymer nanocomposites with higher physical-chemical, operational and other properties than traditionally filled polymer materials.

The development of the science of nanoscale and cluster metal particles in polymer matrices stimulates the growing interest in this problem in many fields of chemistry, physics and materials science. The development of such research has led to the creation of metal-containing nanocomposite materials with special physical, mechanical and operational properties (for example, high thermal and electrical conductivity, high magnetic sensitivity, shielding of ionizing radiation, etc.).

The main requirement for obtaining composite materials is not only to improve their properties, but also to save resources. Among composite materials for various purposes, special attention is paid to polyolefin-based materials, since they are technologically advanced, have low density and are cheap. The development of highly efficient composite materials based on polyolefins containing nanoparticles of various metals is an urgent scientific and technical task.

Currently, various methods for obtaining of metal-containing polymer nanocomposite materials have been developed. Among them, methods are widely used in the synthesis of nanocomposites, where dispersed metal-containing nanoparticles are directly distributed in a polymer matrix or the simultaneous formation of nanoparticles and a stabilizing polymer matrix occurs during polymerization.

However, at present, when obtaining polymeric nanomaterials, a uniform distribution of nanoparticles in the polymer bulk, the preparation of firm nanocomposites, the control of the composite properties through the composition, size and number of nanoparticles remain topical issues.

Although there are many theoretical and experimental works on the obtaining and study of the physico-mechanical properties of such nanocomposites, the properties of the obtained composites are still insufficiently studied, and the number of metallic and metal-containing compounds that can be used as nanofillers is limited. Therefore, there is a need to create new metal-containing polymer nanocomposite materials and to study them more widely.

The relevance of the dissertation topic lies in the fact that it is proposed to obtain metal oxide nanoparticles stabilized by a matrix of maleinized high-pressure polyethylene using a mechanochemical method and use them as nanofillers for industrial polymers, since this method opens up wide opportunities for obtaining new types of nanocomposites, which, in principle, have improved properties and can be used in various fields of technology.

Object and subject of research. The object of research of the dissertation work is the production of metal oxide nanoparticles stabilized by a matrix of maleinized high-pressure polyethylene, the study of their structure, properties and applications. The subject of the research is the obtaining and study of the properties of nanocomposites based on various industrial polymers using the obtained nanoparticles as fillers.

Research goals and objectives - The purpose of the research - obtaining nanoparticles of metal oxides stabilized with a matrix of maleinized high-pressure polyethylene (MPE) and using them as nanofillers for various industrial polymers: high-pressure polyethylene (LDPE), isotactic polypropylene (iPP), mixtures of isotactic polypropylene and high pressure polyethylene (iPP/LDPE), isotactic polypropylene/ butadiene-nitrile rubber (iPP/NBR), epoxy resin (ED-20), study of the structure and properties of the obtained nanocomposites and determination of possible areas of their practical use.

To achieve this goal, the following issues were resolved:

- obtaining nanoparticles of copper(I) oxide and zinc oxide stabilized by a matrix of maleinized high-pressure polyethylene by a mechanochemical method, studying their structure and properties;
- obtaining nanocomposites based on high-pressure polyethylene by mechanical means using Cu_2O and ZnO nanoparticles stabilized by a matrix of maleinized high-pressure polyethylene, studying their properties;
- obtaining nanocomposites based on isotactic polypropylene by mechanical means using Cu_2O and ZnO nanoparticles stabilized by a matrix of maleinized high-pressure polyethylene, studying their properties;
- obtaining nanocomposites based on a mixture of isotactic polypropylene and high-pressure polyethylene by mechanical means using Cu_2O and ZnO nanoparticles stabilized by a matrix of maleinized high-pressure polyethylene, and studying their properties;

- obtaining nanocomposites based on a mixture of isotactic polypropylene and butadiene- nitrile rubber by mechanical means using Cu_2O and ZnO nanoparticles stabilized by a matrix of maleinized high-pressure polyethylene, studying their properties;

- obtaining composites based on epoxy resins using Cu_2O and ZnO nanoparticles as a filler, stabilized by a matrix of maleinized high-pressure polyethylene; studying their properties;

Research methods. Metal-containing nanoparticles stabilized with an MPE matrix were obtained by a mechanochemical method in a polymer melt without the use of solvents in a Brabender microextruder in a nitrogen atmosphere. The structure and composition of polymer nanocomposites were determined by infrared spectroscopy (IR), scanning electron microscopy (SEM), and derivatographic thermal analysis (TGA, DTA). The phase composition and microstructure of the nanocomposite samples were studied by X-ray phase analysis (XRD).

The physical -mechanical properties of the samples were studied on an RMI-250 instrument, the alloy flow index on an IIRT instrument, and the Vicat heat resistance was determined on a Vicat instrument. The thermal stability of the studied samples of nanocomposites was determined on a Q-1500D derivatograph (MOM, Hungary), and SEM analysis was carried out on a JEOL scanning electron microscope (Japan).

The main provisions for defense:

- obtaining by mechanochemical method copper (I) oxide nanoparticles stabilized by a matrix of maleinized high-pressure polyethylene, determination of their structure and composition;

- obtaining zinc oxide nanoparticles stabilized by a matrix of maleinized high-pressure polyethylene by a mechanochemical method, determining their structure and composition;

- obtaining and studying the properties of nanocomposites based on high-pressure polyethylene using Cu_2O and ZnO nanoparticles stabilized by a matrix of maleinized high-pressure polyethylene;

- obtaining and studying the properties of nanocomposites based on isotactic polypropylene using Cu_2O and ZnO nanoparticles stabilized by a matrix of maleinized high-pressure polyethylene;

- preparation and study of nanocomposites based on a mixture of isotactic polypropylene and high-pressure polyethylene using Cu_2O and ZnO nanoparticles stabilized by a matrix of maleinized high-pressure polyethylene;

- preparation and study of composites based on a mixture of isotactic polypropylene and butadiene- nitrile rubber using Cu_2O and ZnO nanoparticles stabilized by a matrix of maleinized high-pressure polyethylene;

- obtaining and study of composites based on epoxy resin ED-20 using Cu_2O and ZnO nanoparticles stabilized by a matrix of maleinized high-pressure polyethylene;

- Influence of the type and amount of nanofiller on the structure, physical-mechanical and thermal properties of the resulting nanocomposites.

Scientific novelty of the research. Nanoparticles of metal oxides (Cu_2O, ZnO) stabilized by a matrix of maleinized high-pressure polyethylene were obtained by a mechanochemical method without the use of organic solvents. By using them as modifying agents, new composites based on industrial polymers were obtained.

It is shown that the interaction of metal oxide nanoparticles located in the interfacial layer of nanocomposites, interacting with the maleic groups of maleinized polyethylene, creates a synergistic effect, resulting in improved properties of the composites.

In the obtained nanocomposites, metal oxide nanoparticles, playing the role of structure buiders, contribute to the formation of a relatively fine spherulite structure.

The effect of the nature of nanoparticles and the microstructure of the initial polymer on the properties of the resulting nanocomposites has been studied.

Theoretical and practical significance of the research. The work proposes a theoretical justification for the use of metal oxide nanoparticles stabilized by a matrix of maleinized high-pressure polyethylene as a filler in the preparation of polymer nanocomposite materials. The practical significance of the presented work lies in the fact that an environmentally friendly mechanochemical method was used to obtain nanoparticles of metal oxides stabilized with a matrix of maleinized high-pressure polyethylene used as a filler in the preparation of nanocomposites, and the optimal technological parameters of the nanoparticle synthesis process were studied. It was found that it is possible to control the properties of nanocomposites by changing the chemical composition of nanoparticles and their amount in the matrix. Thus, the obtained new nanocomposites can find application in various fields of modern science and technology, for example, in microelectronics, aerospace, and other fields due to their high thermal stability and improved physical and mechanical properties.

Approbation and implementation of the study. The author has 22 scientific papers on the topic of the dissertation in journals listed by the AAC, of which 10 articles (2 in mono-authorship), 1 patent (Azerbaijani patent) and abstracts at 11 international and republican conferences.

The main results of the dissertation were presented at the following international and national conferences: PolyChar 26 World Forum on Advanced Materials (Tbilisi, 2018); Abstracts of the Reports of the International scientific-practical conference "Innovative prospects for the development of oil refining and petrochemistry" dedicated to the 100th anniversary of Academician Aliyev V.S. (Baku, 2018); Beynəlxalq Elmi Konfrans, Müasir təbiət və iqtisad elmlərinin aktual problemləri (Gəncə, 2018); 6th International Caucasian Symposium on Polymers and Advanced Materials Batumi (Georgia, 2019); Materials of the International Scientific and Technical Con. "Polycomtribe. – 2019" (Gomel, Belarus, 2019); The International Scientific Conference "Actual Problems of Modern Chemistry" (Baku, 2019); Organic and hybrid nanomaterials VI All-Russian school-conference of young scientists (Russia, Ivanovo, 2019); Abstracts and materials of the International Scientific Conference "Prospects for innovative development of chemical technology and engineering" on November 28-29, 2019 dedicated to the 70th anniversary of the city of Sumqayit (Sumqayıt, 2019); Second International Scientific Conference of Young Scientists and Specialists Multidisciplinary approaches in solving modern problems of fundamental applied sciences. Dedicated to the 75th anniversary of Azerbaijan National Academy of Sciences (Baku, 2020); Second International Caucasian Symposium on Polymers and Advanced Materials (Georgia, Tbilisi 2021); 7th International conference: Modern Trends in Physics, MTP-2021 (Azerbaijan Baku, 2021).

The name of the organization where the dissertation was completed. Institute of Polymer Materials of Azerbaijan National Academy of Sciences. The dissertation work was carried out in accordance with the research plan.

Personal participation of the author: The author has a leading role in the performance of the work, including the formulation of the problem, the main ideas and directions of the study, the conduct of experiments, the interpretation of the results and the formulation of conclusions.

The structure and scope of the dissertation. The dissertation work consists of an introduction, three chapters, conclusions, a list of cited literature of 164 titles, an appendix, contains 27 figures, 17 tables, includes a total of 174065 characters: introduction 13753, chapter I 48492, chapter II 18303, chapter III 90530, conclusions 2557.

The introduction substantiates the relevance of choosing a dissertation topic, formulates the purpose and objectives of the study, scientific novelty and practical significance of the dissertation work. **The first chapter** is of a review nature and is devoted to nanoparticles, the main methods for obtaining nanoparticles and new nanomaterials. Here, the information of modern scientific literature on nanoparticles and composites based on them is summarized. The main attention is paid to methods for obtaining metal nanoparticles stabilized by organopolymer matrices, as well as to obtaining nanocomposites and studying their properties. **The second chapter** presents the physical-chemical and technical characteristics of the raw materials and instruments used in the experimental work. This chapter describes: the methodology for the mechanochemical production of metal nanoparticles stabilized by a matrix of maleinized high pressure polyethylene, as well as the main physical-chemical and mechanical methods for studying their composition, structure and properties. **The third** chapter is devoted to the mechanochemical method of obtaining nanoparticles of metal oxides (Cu_2O and ZnO), stabilized by a matrix of maleinized high-pressure polyethylene, the study of their structure and properties, as well as their use as nanofillers for various industrial polymers in order to obtain nanocomposites based on them, to study their properties and discussion of the obtained results. Nanocomposites based on basic industrial polymers with nanofillers in various ratios have been obtained and studied, and their optimal amounts have been determined. The influence of the type of nanofillers and the microstructure of base polymers on the properties of the resulting nanocomposites was revealed. At the end of the dissertation work, conclusions are given, a list of references, in the appendix - a list of conditional abbreviations and an act on the conduct of studies confirming the bactericidal properties - biological resistance against fungi of samples of Cu_2O and ZnO nanoparticles stabilized with an MPE matrix.

THE MAIN CONTENT OF THE WORK

1. Obtaining nanoparticles of metal oxides stabilized by a matrix of maleinized high-pressure polyethylene, study of their composition and structure.

1.1. Obtaining of metal oxide nanoparticles stabilized with a matrix of maleinized high-pressure polyethylene

Nanoparticles of copper(I) oxide and zinc oxide stabilized with a matrix of maleinized high-pressure polyethylene were obtained using a mechanochemical method by thermal decomposition of metal acetates in a polymer melt under conditions of high shear deformation. Binary mixtures of maleinized polyethylene (MPE) and precursor metal acetates were preliminarily prepared by mixing on laboratory rollers. The resulting binary mixtures were subjected to thermolysis in an inert atmosphere (nitrogen) in a laboratory extruder at a temperature of 240°C for 20 minutes under fast shear strain conditions. The amount of the precursor was calculated so that the amount of metal oxide nanoparticles obtained after decomposition was 5% of the mass of maleinized polyethylene.

During extrusion, as a result of rapid mixing of the precursor-polymer melt in an inert medium at high temperatures, the precursor undergoes thermal decomposition, and the metal oxide nanoparticles obtained as a result of decomposition are stabilized in the polymer melt. Obtaining stabilized nanoparticles of metal oxides in a polymer matrix occurs at a temperature of the polymer melt, which coincides with the decomposition temperature of the metal precursor.

The technical advantage of this method for obtaining metal nanoparticles stabilized by a polymer matrix is that the processes of obtaining dispersed metal oxides nanoparticles and stabilization of the resulting nanoparticles are carried out simultaneously by the method in situ. In addition, the uniform distribution of metal-containing nanoparticles in the polymer melt can be controlled by the size of the phases and the degree of dispersion.

1.2. Study of the composition and structure of metal oxide nanoparticles stabilized by a matrix of maleinized high-pressure polyethylene

The phase composition and microstructure of metal oxide nanoparticles stabilized by a matrix of maleinized high-pressure polyethylene have been studied by X-ray phase analysis (XRD) and infrared spectroscopy (IR). Figures 1, 2 and 3 show XRD diffractograms of the initial maleinized high-pressure polyethylene, as well as maleinized high-pressure polyethylene containing copper (I) oxide and zinc oxide nanoparticles. Phase identification was carried out using the ASTM file for interplanar distances. As can be seen from fig.1, in the case of the diffraction pattern of the original MPE, only reflections related to polyethylene are observed.

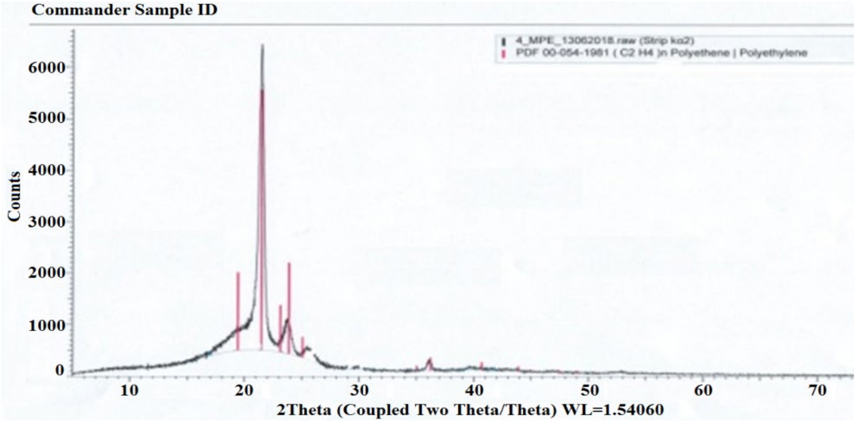

Fig. 1. X-ray diffraction patterns of the initial maleinized high-pressure polyethylene

In the XRD pattern of the Cu_2O sample stabilized with the MPE matrix, along with polyethylene reflections, other reflections are observed. Phase identification was carried out according to the data on interplanar distances, using the ASTM card file. It is shown that reflections from the planes of the crystal lattice of metals were observed in the studied nanocomposites, corresponding to the d_{hkl} series of copper oxide I (Cu_2O) according to the ASTM card file.

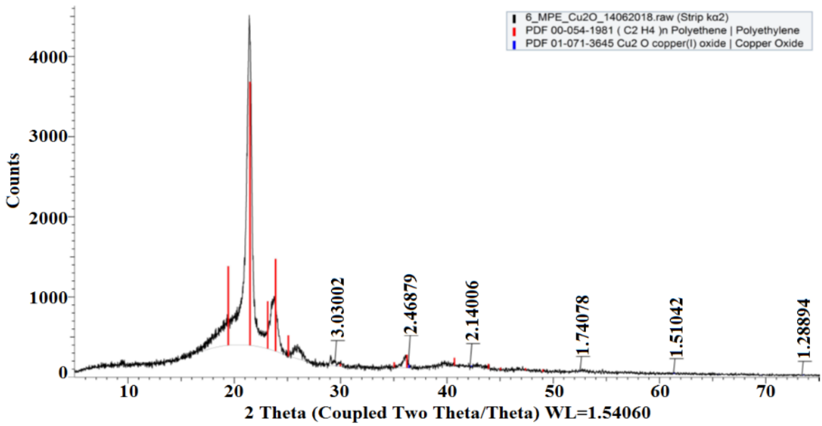

Fig. 2. X-ray diffraction pattern of maleinized high-pressure polyethylene containing copper (I) oxide nanoparticles

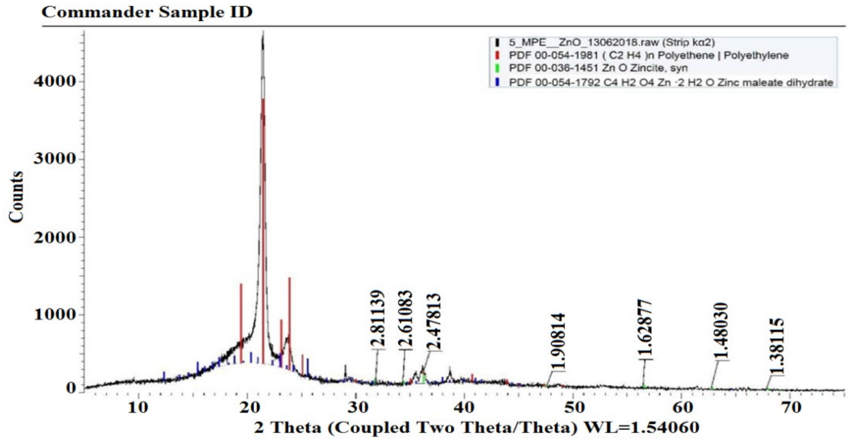

Fig. 3. X-ray diffraction pattern of MPE containing zinc oxide nanoparticles

Analysis of the diffractogram in fig. 3 showed that in the studied nanocomposites, reflections from the planes of the crystal lattice of metals were observed, corresponding to the $d_{hk}l$ series of zinc oxide (ZnO) according to the ASTM card file, and there is also a structure corresponding to ($C_4H_2O_4Zn \cdot 2H_2O$) -zinc maleate dehydrate.

The structure and composition of nanostructured polymer composites were determined by IR spectroscopy in the wavenumber range 400–4000 cm^{-1}.

The data of X-ray phase analysis are also confirmed by the absorption bands in the IR spectra of these samples.

Fig. 4, 5, and 6 show the corresponding IR spectra of the initial MPE, as well as MPE containing nanoparticles of copper and zinc oxides.

The IR spectrogram of the initial maleinized high-pressure polyethylene contains the following absorption bands characterizing the maleic group, where the absorption bands at 1717, 1738 cm^{-1} characterize the C=O carbonyl group, the absorption bands at 1017, 1046, 1163 cm^{-1} characterize the C-O- C, and the absorption bands at 2848 and 2915 cm^{-1} characterize the CH-CH_2 bonds and CH3 groups, respectively.

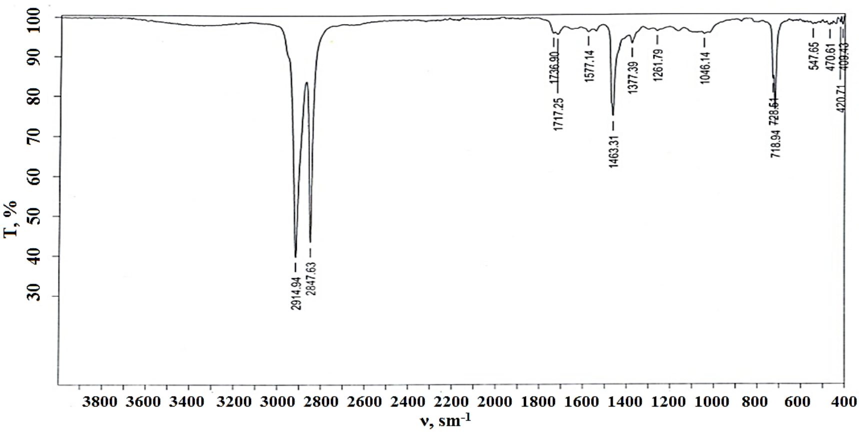

Fig. 4. IR spectrum of the original MPE

Comparing the IR spectrum of the initially maleinized high-pressure polyethylene with the IR spectrum of the nanocomposite sample with copper (I) oxide, it can be seen that such a fundamental change does not occur in the IR spectrum of the copper-containing sample. This is explained by the fact that the device cannot detect Cu_2O, since the absorption bands corresponding to it are beyond the wave numbers of 400 cm^{-1}.

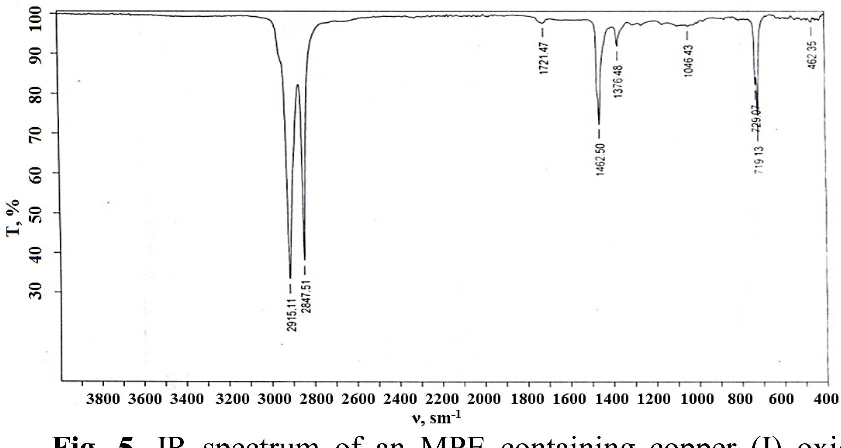

Fig. 5. IR spectrum of an MPE containing copper (I) oxide nanoparticles

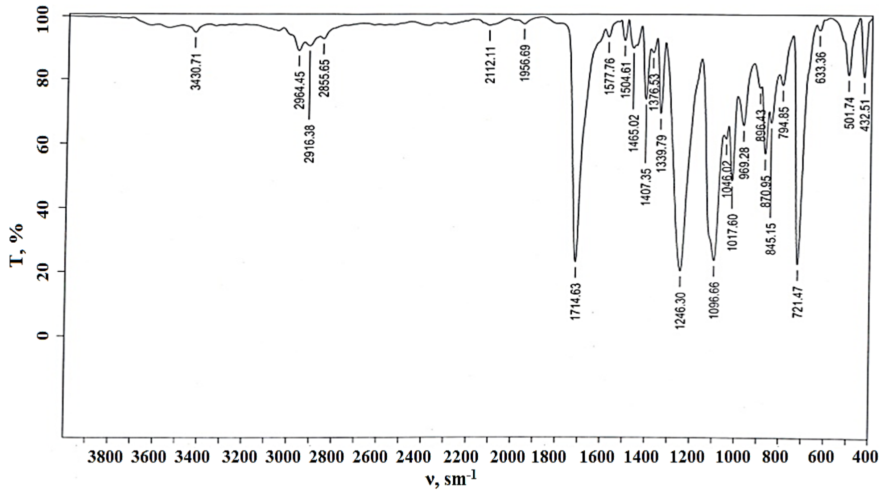

Fig. 6. IR spectrum of MPE containing nanoparticles, zinc oxide

Comparing the IR spectrum of the initial maleated high-pressure polyethylene in Fig. 4 with the IR spectrum of the nanocomposite with zinc-containing nanoparticles (Fig. 6), it becomes clear that the absorption bands of maleic groups in the IR spectrum of the synthesized maleated polyethylene with ZnO nanoparticles do not participate, but appears an absorption band at 1577 cm^{-1} characteristic of the COO group, as well as new absorption bands at 432, 501, 633, 721 cm^{-1}, corresponding to stretching vibrations of the Me-O bond.

Thus, when analyzing the XRD and IR spectroscopy data of the obtained nanocomposites, it can be concluded that copper (I) oxide nanoparticles do not interact with the polymer matrix, but are introduced into its interspherolitic regions. In the case of zinc-containing nanoparticles, the bonds in the maleic group of the polymer are broken with the preservation of the C=O group and the formation of a COO- bond. Apparently, zinc-containing nanoparticles are partially preserved in the polymer structure in the form of ZnO, and are partially added at the COO- group to form COO-Zn-OOC.

It was found that zinc oxide and copper (I) oxide nanoparticles stabilized by a matrix of maleinized high-pressure polyethylene have dimensions (25 ± 1.0) nm, determined by the Create Area method according to the program (DIFFRAC.EVA.V3.2). The number of nanoparticles is equal to 5 wt. %, the degree of crystallinity is 35÷45%

Bactericidal properties. Biological stability against fungi of samples of Cu_2O and ZnO nanoparticles stabilized with an MPE matrix was studied in the laboratory of microbiological biotechnology of the Institute of Microbiology of ANAS. Studies have shown that the tested samples are resistant to such fungi as Aspergillus Niger, A. Ochraseus, Penicillium Cuclopium, Cladosporium Herbarium, Fusarium Moniliforme və F. Oxysporium. The results are reflected in the act (photos and test report are attached to the dissertation).

Thus, a method has been developed for the mechanochemical production of copper and zinc oxide nanoparticles stabilized by a matrix of maleinized high-pressure polyethylene by the *in situ* method in a polymer melt. Their phase composition and structure were studied by XRD and IR spectroscopy, and it was found that nanoparticles have bactericidal properties.

2. Obtaining and study of nanocomposites based on industrial polymers with metal oxide nanoparticles stabilized by a matrix of maleinized high-pressure polyethylene

New nanocomposites were obtained mechanically by including metal oxide (Cu_2O and ZnO) nanoparticles stabilized with an MPE matrix into industrial polymers as a filler, and their properties were studied. Of the industrial polymers, the following were used: thermoplastics - high-pressure polyethylene (LDPE), isotactic polypropylene (iPP) and their mixtures (iPP / LDPE); from thermoplastic elastomers - a mixture of isotactic polypropylene and butadiene-nitrile rubber (iPP / NBR); from thermosets - epoxy resin (ED-20).

2.1 Study of the effect of metal oxide nanoparticles and the microstructure of initial polymers on the properties of obtained nanocomposites

The effect of metal oxide nanoparticles on the physical and mechanical properties and thermostability of nanocomposites based on various polymers was studied. The thermostability of the samples was evaluated for decomposition temperatures, half-life ($\tau_{1/2}$) and activation energy of thermooxidation destruction (Ea) corresponding to mass loss of 10%, 20% and 50% ($T_{10\%}$, $T_{20\%}$, $T_{50\%}$).

First, let us consider the results of studying the process of modifying high-pressure polyethylene with metal oxide nanoparticle fillers stabilized by an MPE matrix. Due to the fact that LDPE and MPE are well combined at the level of microstructures, the properties of the resulting modified nanocomposites are affected by the structure of the metal nanoparticles themselves. A comparative analysis of the effect of a nanofiller additives (NF) containing nanoparticles of copper(I) and zinc oxides showed that the improvement in the physical-mechanical and thermal-oxidative properties of the obtained nanocomposites with the participation of copper oxide nanoparticles is higher compared to the indicators obtained with the participation of zinc oxide nanoparticles . Below are some physical-mechanical (tab.1) and thermal (tab.2) indicators of composites based on LDPE containing nanoparticles of copper (I) and zinc oxides.

Table 1.

Physical -mechanical properties of nanocomposites based on LDPE

Nanocomposition proportions (wt.%)	**σ_q, MPa**	**ε_q, %**	**T_{vicat}, °C**	**MFI, g/10min**
LDPE	11.39	400	130	9.9
LDPE/Cu_2O MPE (100/0.3)	12.78	720	143	10.5
LDPE/Cu_2O MPE (100/0.5)	14.06	780	135	13.7
LDPE/Cu_2O MPE (100/1.0)	11.97	660	132	20.5
LDPE/ZnO MPE (100/0.3)	12.07	720	140	10.8
LDPE/ZnO MPE (100/0.5)	13.56	660	135	11.7
LDPE/ZnO MPE (100/1.0)	11.67	637	128	15.4

Table 2.

Thermal properties of nanocomposites based on LDPE

Nanocomposition proportions (wt.%)	**$T_{10\%}$, °C**	**$T_{20\%}$, °C**	**$T_{50\%}$, °C**	**$\tau_{1/2}$, min**	**E_a, kJ/mol**
LDPE	325	350	400	72	120.4
LDPE/Cu_2OMPE(100/0.3)	350	365	425	75	125.8
LDPE/Cu_2OMPE(100/0.5)	360	370	450	79	131.9
LDPE/Cu_2OMPE(100/1.0)	337	360	440	74	123.7
LDPE/ZnO MPE (100/0.3)	340	365	425	75	130.8
LDPE/ZnO MPE (100/0.5)	360	370	450	81	135.9
LDPE/ZnO MPE (100/1.0)	337	360	420	74	132.7

As can be seen from Tables 1 and 2, in all cases, the physical-mechanical and thermal parameters of composites with Cu_2O nanoparticles are higher than those of composites with ZnO. For example, the degree of improvement of performance for LDPE modified with Cu_2O and ZnO nanoparticles stabilized by an MPE matrix relative to the initial LDPE is as follows, for Cu_2O NPs: tensile strength (σ_q) increases by 1.23 times, specific elongation (ε_q) by 1.95 times, heat resistance (T_{vicat}) by 1.11 times, the melt yield strength (MFI) by 1.38 times, the activation energy of thermal-oxidative degradation (E_a) increases from 120.45 to 131.92 kJ•mol^{-1}; for ZnO NPs, the corresponding indicators increase by 1.19; 1.1; 1.07; 1.18 times, E_a from 120.45 to 135.97 kJ·mol^{-1}. This is apparently due to the fact that copper(I) oxide nanoparticles actively interact with MPE maleic groups to form a synergistic effect; there is an increase in all parameters of the LDPE/Cu_2OMPE nanocomposite. In the case of ZnO NPs in the nanostructured composite, along with ZnO NPs, there is also the “zinc maleate dihydrate” $C_4H_2O_4Zn \cdot 2H_2O$ structure, which, apparently, contributes to insufficiently complete interaction of ZnO NPs with MPE maleic groups, and the indicators of modified LDPE samples are somewhat lower, however, and in this case, a synergistic effect also occurs, i.e. strengthening of all physical-mechanical and thermal-oxidative parameters of the LDPE/ZnOMPE nanocomposite.

Let us consider the modification of isotactic polypropylene with nanofillers containing nanoparticles of copper and zinc oxides stabilized by an MPE matrix. Compatibility of iPP with MPE at the level of their microstructure is low, however, the presence of maleic groups in the MPE structure has a positive effect on their compatibility.

Let us consider the influence of the structures of the metal oxide nanoparticles themselves on the properties of the obtained modified nanocomposites based on iPP. Cu_2O nanoparticles are very rigid, and when they are included into the iPP matrix, the obtained samples of nanocomposites were fragile and brittle. Therefore, their physical-mechanical and thermal properties have not been studied.

ZnO nanoparticles are relatively soft and the additional content of "zinc maleate dihydrate" promotes better interaction between the nanostructured composite and the iPP polymer matrix. The obtained modified samples of iPP with ZnOMPE have high physical-mechanical and thermal-oxidative properties, which is due to the synergistic effect of interaction between ZnOMPE and iPP. Some physical and mechanical properties of nanocomposites based on polypropylene are presented in table 3, thermal - in table 4.

Table 3

Physical - mechanical properties of nanocomposites based on iPP

Nanocomposition proportions (wt.%)	**σ_p, MPa**	**ε_p, %**	**T_{vicat}, °C**	**MFI, g/10min**
iPP	21.6	46	165	9.5
iPP/ZnO MPE (100/0.5)	28.1	22	165	15.1
iPP/ZnO MPE (100/1)	32.7	80	165	19.7
iPP/ZnO MPE (100/3)	11.8	32	165	41.2

According to Table 3 and 4, when using ZnO nanoparticles as nanomodifiers, the increase in the main characteristics of the composition with the optimal amount of nanoparticles (1.0 wt.%) is: tensile strength by 1.51 times, specific elongation by 1.74 times, the heat resistance remains at the level of 165°C, the melt yield strength increases by 2.07 times, the activation energy of thermal oxidative destruction (E_a) increases from 122.49 to 175.53 kJ·mol^{-1}.

Table 4

Thermal properties of nanocomposites based on i-PP

Nanocomposition proportions (wt.%)	**$T_{10\%}$, °C**	**$T_{20\%}$, °C**	**$T_{50\%}$, °C**	**$\tau_{1/2}$, min.**	**E_a , kJ/mol**
i-PP	245	285	340	64	122.49
i-PP/ZnOMPE(100/0.5)	260	300	360	68	170.23
i-PP/ZnOMPE (100/1)	275	310	370	71	175.53
i-PP/ZnOMPE (100/3)	285	325	360	69	173.12

According to Table 3 and 4, when using ZnO nanoparticles as nanomodifiers, the increase in the main characteristics of the composition with the optimal amount of nanoparticles (1.0 wt.%) is: tensile strength by 1.51 times, specific elongation by 1.74 times, the heat resistance remains at the level of 165°C, the melt yield strength increases by 2.07 times, the activation energy of thermal oxidative destruction (E_a) increases from 122.49 to 175.53 kJ·mol^{-1}.

Let us consider the modification of a mixture of isotactic polypropylene with LDPE with nanofillers containing nanoparticles of copper and zinc oxides stabilized by an MPE matrix. It is known that iPP is not compatible with any polymer. To improve the compatibility of iPP, a modification method is widely used, which consists in creating polymer-polymer compositions. iPP and LDPE are immiscible polyolefins. To improve their compatibility, it is necessary to have functional groups in their composition or the introduction of nanofillers into the composition, which are an interfacial additive that improves both the compatibility of the components and the operational properties of the obtained materials.

As it was shown earlier, LDPE and MPE are well combined at the level of microstructures, and the compatibility of iPP with MPE at the level of their microstructure is small. The presence of metal nanoparticles in the structure of MPE has a positive effect on their compatibility. Thus, the properties of the obtained modified iPP/LDPE nanocomposites are mainly influenced by metal nanoparticles.

As was found earlier, Cu_2O nanoparticles are very rigid and, when they are introduced into the iPP matrix, the resulting nanocomposite samples are fragile and brittle. The introduction of Cu_2O NPs (optimal content 0.5 wt %) into the composition of the iPP/LDPE mixture promotes the production of a nanocomposite based on it with improved physical-mechanical and thermal-oxidative properties, which is due to the synergistic effect of interaction between Cu_2OMPE NPs and i-PP. Physical and mechanical properties of nanocomposites based on iPP/LDPE are presented in Table 5, and thermal properties - in Table 6.

Table 5

Physical -mechanical properties of nanocomposites based on iPP/LDPE

Nanocomposition proportions (wt.%)	**σ_q, MPa**	**ε_q, %**	**T_{vicat}, °C**	**MFI, g/10min**
iPP/LDPE (50/50)	13.15	20	160	9.5
iPP/LDPE/Cu_2O MPE (50/50/0.3)	14.76	28	160	18.4
iPP/LDPE/Cu_2O MPE (50/50/0.5)	13.97	24	160	24.1
iPP/LDPE/Cu_2O MPE (50/50/1.0)	13.35	20	160	41.9
iPP/LDPE/ZnO MPE (50/50/0.3)	14.57	24	160	16.8
iPP/LDPE/ZnO MPE (50/50/0.5)	13.96	24	160	20.5
iPP/LDPE/ZnO MPE (50/50/1.0)	13.53	22	160	40.4

A comparative analysis of the effect of additives of nanofillers (NF) containing nanoparticles of copper and zinc oxides showed that the improvement in the physical-mechanical and thermal-oxidative properties of the obtained iPP/LDPE nanocomposites with the participation of copper oxide nanoparticles is higher compared to the indicators obtained with the participation of zinc oxides nanoparticles.

Table 6

Thermal properties of nanocomposites based on i-PP/LDPE mixture

Nanocomposition proportions (wt.%)	**$T_{10\%}$, °C**	**$T_{20\%}$, °C**	**$T_{50\%}$, °C**	**$\tau_{1/2}$, min.**	**E_a, kJ/mol**
iPP/LDPE (50/50)	200	275	325	55.6	191.48
iPP/LDPE/Cu_2O MPE (50/50/0.3)	300	330	370	68.4	215.1
iPP/LDPE/Cu_2O MPE (50/50/0.5)	320	345	390	71.2	233.75
iPP/LDPE/Cu_2O MPE (50/50/1.0)	290	320	360	66.7	213.2
iPP/LDPE/ZnOMPE (50/50/0.3)	280	320	355	65	212.8
iPP/LDPE/ZnOMPE (50/50/0.5)	300	335	380	70	229.5
iPP/LDPE/ZnOMPE (50/50/1.0)	270	310	350	63	210.7

Thus, for Cu_2O NPs, the tensile strength at break increases by a factor of 1.12, relative elongation by a factor of 1.4, heat resistance remains at the level of 160°C, melt yield strength by a factor of 1.93, activation energy of thermal oxidative destruction (Ea) increases from 191.48 to 233.75 kJ·mol^{-1}. Thus, for ZnO NPs: the tensile strength at break increases by 1.1 times, relative elongation - by 1.2 times, heat resistance remains at the level of 160°C, melt yield strength - by 1.76 times, activation energy of thermal oxidative degradation (E_a) increases from 191.48 to 229.53 kJ ·mol^{-1}.

Let us consider the modification of a mixture of isotactic polypropylene and nitrile butadiene rubber with nanofillers containing nanoparticles of copper and zinc oxides stabilized by an MPE matrix obtained by a mechanochemical method (Table 7; Table 8).

A comparative analysis of the effect of additives of nanofillers (NF) containing nanoparticles of copper and zinc oxides showed that the improvement in the physical-mechanical and thermal-oxidative parameters of the obtained nanocomposites with the participation of zinc oxide nanoparticles is higher compared to the indicators obtained with the participation of copper oxide nanoparticles.

Table 7

Physical-mechanical properties of nanocomposites based on iPP/NBR

Nanocomposition proportions (wt.%)	**σ_q, MPa**	**ε_q, %**	**T_{vicat}, °C**	**MFI, g/10min**
iPP/NBR (50/50)	5.04	16	87	0.089
iPP/NBR/Cu_2O MPE (50/50/0.3)	5.55	20	115	0.114
iPP/NBR/Cu_2O MPE (50/50/0.5)	6.15	24	110	0.123
iPP/NBR/Cu_2O MPE (50/50/1.0)	5.51	21	105	0.169
iPP/NBR/ZnO MPE (50/50/0.3)	6.65	36	120	0.127
iPP/NBR/ZnO MPE (50/50/0.5)	6.94	40	127	0.155
iPP/NBR/ZnO MPE (50/50/1.0)	6.51	32	115	0.287

Table 8

Thermal properties of nanocomposites based on i-PP/NBR

Nanocomposition proportions (wt.%)	**$T_{10\%}$, °C**	**$T_{20\%}$, °C**	**$T_{50\%}$, °C**	**$\tau_{1/2}$, min.**	**E_a, kJ/mol**
i-PP/NBR (50/50)	225	250	300	62	124.48
i-PP/NBR/Cu_2OMPE (50/50/0.3)	280	310	370	66	165.32
i-PP/NBR/Cu_2OMPE (50/50/0.5)	290	320	375	72	176.47
i-PP/NBR/Cu_2OMPE (50/50/1.0)	270	300	360	65	163.51
i-PP/NBR/ZnOMPE (50/50/0.3)	260	305	370	75	186.32
i-PP/NBR/ZnOMPE (50/50/0.5)	270	315	380	80	204.77
i-PP/NBR/ZnOMPE (50/50/1.0)	255	300	365	72	172.45

Thus, the tensile strength at break increases by a factor of 1.38 for ZnO NPs, relative elongation by a factor of 2.5, heat resistance by a factor of 1.45, melt yield strength by a factor of 1.74, and the activation energy of thermal oxidative degradation (E_a) increases from 124.48 to 204.77 kJ·mol^{-1}; for Cu_2O NPs: the corresponding figures increase by factors of 1.22, 1.5, 1.32, 1.38, E_a from 124.48 to 176.49 kJ·mol^{-1}.

This is due to the fact that in the nanostructured composite, along with zinc oxide nanoparticles, there are structures of zinc maleate dihydrate, which, being located in the interfacial layer of the structural elements of polypropylene, nitrile rubber, and maleinized high-pressure polyethylene, form heterogeneous nucleation centers in the composition melt, which in the process of step cooling of the nanocomposite contributes to the creation of additional centers of crystallization, leading in general to an improvement in the crystallization process and the formation of a relatively fine spherulitic structure.

Figure 7 shows SEM microphotographs of the i-PP / BNK mixture (Figure 7a) and the metal oxide-based nanocomposite (Figure 7b) based on this mixture.

a) b)

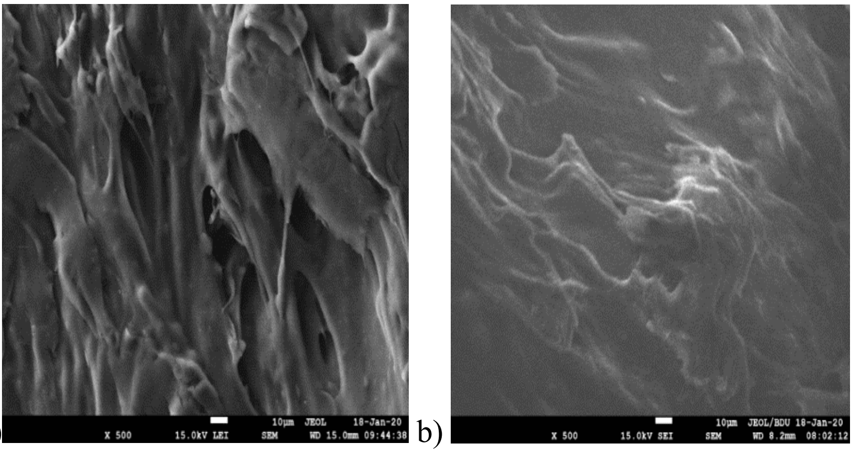

Fig. 7. Micrographs of PP/NBR (a) and PP/NBR/NF (b) samples.

As can be seen from the SEM micrographs, the structure of the PP/NBR composite is shapeless, i.d. heterogeneous. This is explained by the fact that iPP and NBR are polymers that do not correspond to each other at the molecular level. When nanoparticles are added to PP/NBR, metal oxide nanoparticles form a relative consistency in the interfacial regions of the polymer and a smooth surface with a fine spherulitic structure is formed, which increases the fluidity of the nanocomposite.

SEM analysis shows that a small amount of nanoparticles (0.3–0.5 wt.%) plays the role of a structure builder - a crystal builder, which leads to the formation of a relatively small spherulitic layered structure in the polymer. As a result, the physical-mechanical and thermal properties of the obtained nanocomposites are significantly improved.

Let us consider a modification of a thermoset - epoxy resin ED-20 with nanofillers containing nanoparticles of copper and zinc oxides stabilized by an MPE matrix. It has been shown that the inclusion of a nanofiller containing copper oxide nanoparticles in the composition based on ED-20 significantly reduces the temperature of the "cold" and "hot" curing reactions, increases the thermal stability, and acts as a catalyst for the curing reaction. This is evidenced by the shift of the peak on the curing curve in the direction of decreasing temperature.

At the same time, zinc oxide nanoparticles practically do not affect the thermal properties of nanocomposites based on ED-20.

3. Influence of the interfacial layer on the properties of the obtained modified nanocomposites

The research work carried out is devoted to the production of metal oxides stabilized with a matrix of maleinized high-pressure polyethylene (MPE), their use as modifying agents of various industrial polymers, such as: high-pressure polyethylene (LDPE), isotactic polypropylene (iPP), mixtures of iPP/LDPE and iPP/NBR, as well as epoxy resin ED-20 in order to obtain nanocomposites with an improved set of properties. The structure and properties of the obtained nanocomposites are determined. An improvement in the physical-mechanical and thermal-oxidative properties of nanocomposites was revealed, which is associated with a synergistic effect due to the interaction of copper (I) and zinc oxide nanoparticles with maleic groups in the matrix of maleinized polyethylene.

The study of the dispersion properties of nanoparticles can be considered one of the important points of our research, which controls their size and homogeneous distribution within the matrix.

Most of the published materials on nanocomposites in the scientific literature are devoted to the obtaining and properties of composites based on polyethylene and polypropylene, which are typical polyolefins. Interest in such composites is due to the fact that they are inexpensive and have a higher set of properties than the initial polyolefins. At the same time, it should be noted that the most important factor for composites based on large-tonnage polymers is that products based on them are characterized by high mechanical properties that ensure long-term performance under operating conditions. The main purpose of the dissertation is to develop composites based on existing polymers with new improved properties.

Metal-containing nanofillers provide ample opportunities to control the properties of substances without a significant change in their composition due to the manifestation of size effects that affect the electronic, thermal, mechanical, electrical, and other properties of the filler and affect the properties of materials.

Metal-containing nanofillers have a high capacity for elastic deformation, which increases the tensile strength of composites with fillers based on them. They impart rigidity to polymers and increase the specific characteristics of the strength and stiffness values of composites. A noticeable improvement in properties is achieved by a smaller amount of included substances compared to other fillers. The inclusion of small amounts of nanofiller significantly affects the degree of crystallinity, which is due to the fact that a part of the polymer transferred to the adsorption layer near the surface of the metal-containing nanofiller participates in crystallization. Obviously, this is also due to the fact that metal-containing nanofillers are centers of nucleation. In addition, apparently, the nature of the interaction at the phase boundary changes while maintaining sufficient segmental mobility of polymer molecules.

In modern polymer materials science, polymer mixtures are used to manufacture composite materials with a complex of improved functional properties, as well as to expand their range and areas of application. Due to the mixing of polymers, many properties of the resulting composites can be improved, primarily mechanical strength. The complex of technological and operational properties of the obtained materials is determined by the structure of polymer mixtures. At the same time, the uniqueness of polymer mixtures lies in the fact that they not only retain the properties of the initial components of the mixture, but can also acquire new properties that none of the initial components possesses.

However, even in the absence of synergy, i.e., the excess of the obtained properties in comparison with their additive values, the mixture of polymers in one composite combines the main properties of both components.

A characteristic feature of most polymer blends is their thermodynamic incompatibility and inability to form single-phase blends. Thermodynamic compatibility and, accordingly, the mutual solubility of one polymer in another is determined by the change in the thermodynamic potential G of the system when polymers are mixed.

However, from a practical point of view, there is no need to achieve an increase in thermodynamic compatibility for polymer mixtures, since a significant increase in their properties can be successfully achieved by enhancing the adhesive interaction between the components of the mixture. For this purpose, various compatibilizers are most often introduced into the polymer mixture - substances that have a chemical affinity with both components of the mixture.

Thus, an increase in interfacial interaction at the interface of components is a key condition for obtaining polymer composites with improved properties. The nanoparticles of copper (1) and zinc oxides obtained in this work, stabilized by the MPE matrix, have high adhesive strength, their introduction into the composition of polymer mixtures of iPP /LDPE and iPP/NBR, contributes to an increase in adhesion between the components of the mixture and thereby some of their compatibility. This is evidenced by the high values of the physical-mechanical and thermal-oxidative properties of the obtained nanocomposites.

In an "ideal" polymer mixture, intermolecular interaction occurs at the interface of two chemically pure components, and, as stated above, it can be assumed that there is a vacuum with a dielectric constant equal to one between both surfaces, and the gap width is equal to the average interatomic distance. In a real polymer mixture, a layer of a third substance may be present on the surfaces of the components – either a specially included modifier, a compatibilizer, etc., or formed due to low-molecular and oligomeric products released from the polymer, as well as thermal oxidation products. The thickness of such a layer can vary significantly – from monomolecular layers to tens of micrometers.

Numerous experimental data on the mechanical, strength, relaxation, and other properties of polymer-polymer and polymer-filler mixtures are explained in terms of the presence of an interfacial layer.

The properties of polymer composites are significantly affected by the supramolecular structure of the polymer (the size of spherulites, the degree of crystallinity, the presence of C=O groups and various branches, etc.) and interfacial interaction at the interface.

The metal-containing nanoparticles used in the work, located at the boundary of the interfacial layer of structural elements iPP, LDPE, iPP/LDPE, iPP/NBR and MPE, contribute to the formation of heterogeneous nucleation centers in the melt composition, which in the process of step cooling of the nanocomposite contribute to an increase in crystallization centers, resulting in an overall improvement in the crystallization process and the formation of a relatively small-spherulite structure.

The obtained results indicate that small amounts of nanofillers (0.3 – 0.5 wt. %), included into the polymer, obviously play the role of structure-forming agents - artificial nuclei of crystallization, which contributes to the appearance of a fine-spherulite structure in the polymer, characterized by improved physico-mechanical and thermal properties of the resulting nanocomposite.

CONCLUSIONS

1. Nanoparticles of copper (I) oxide and zinc oxide stabilized by a matrix of maleinized high-pressure polyethylene were obtained by the mechanochemical method in the polymer melt. Their phase composition and structure were determined by X-ray and IR spectroscopy methods, and their bactericidal properties were revealed [1, 3, 5, 6].

2. The resulting metal oxide nanoparticles stabilized by a matrix of maleinized high pressure polyethylene were used as modifying agents to improve the performance of industrial polymers (LDPE, iPP, iPP/LDPE, iPP/NBR, ED-20). It has been found that the obtained modified nanocomposites have high operational, deformation, strength, thermal-physical properties and can be processed by pressing, injection molding and extrusion methods, which further expands the scope of their application [1, 2, 4, 7-22].

3. It is been found that the physical-mechanical properties of the obtained nanocomposites increase compared to the initial polymer matrix - the heat resistance increases by 1.5-2 times, and the melt yield strength increases by 1.3-1.8 times, which is associated with a synergistic effect due to the interaction of copper (I) and zinc oxide nanoparticles with maleic groups in the matrix of maleinized polyethylene [13-22].

4. SEM analysis has shown that metal oxide nanoparticles stabilized by an MPE matrix form a relatively fine spherulitic structure in modified nanocomposites, resulting in improved physical-mechanical, thermal, and operational properties of the resulting nanocomposites [17, 21, 22].

5. The study of the thermal-physical and thermal properties of the obtained nanocomposites showed that when a metal-containing nanofiller is added to the composition in an amount of 0.3 - 0.5 wt. %, the activation energy of the process of thermal-oxidative destruction increases by 1.4-1.7 times, which expands the temperature range of their operation [8-22].

6. It is shown that when copper (I) oxide nanoparticles are included as filler into a composition based on ED-20, the "cold" curing temperature decreases from 90 °C to 75 ° C, and the "hot" curing temperature decreases from 120 °C to 100 °C. The thermal stability of the composition increases, which is confirmed by an increase in the activation energy of thermal-oxidative destruction (Eact.) from 210 to 225 kJ/mol. It has been found that copper (I) oxide nanoparticles act as a catalyst for the curing reaction. Under the same conditions, zinc oxide nanoparticles practically do not affect the curing rate of nanocomposites [12].

7. The obtained nanocomposites with improved properties can be recommended for use as a promising material in mechanical engineering, electrical engineering, medicine, food industry, petrochemistry, construction, etc. [20, 21].

The main results of the dissertation are presented in the following publications:

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The defense will be held on _13_ _september_ _2022_ at the meeting of the Dissertation council ED 1.28 of Supreme Attestation Commission under the President of the Republic of Azerbaijan operating at the Institute of Polymer Materials of Azerbaijan National Academy of Sciences

Address: S.Vurgun str., 124, AZ5004 Sumgait

Dissertation is accessible at the Institute of Polymer Materials of Azerbaijan National Academy of Sciences Library

Electronic versions of dissertation and its abstract are available on the official website of the Institute of Polymer Materials of Azerbaijan National Academy of Sciences

Abstract was sent to the required addresses on
_18___ _june_ 2022.

Signed for print: 13.06.2022

Paper format: A5

Volume: 38515

Number of hard copies: 20