

DEVELOPMENT OF COMPOSITE MATERIALS BASED ON SOME METAL PHOSPHATES

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ABSTRACT

Despite the small share of nanoparticles in the chemical industry, the demand for modifiers is growing every day. This is due to their high physical, mechanical and operational properties, resistance to aggressive media and their ability to be used over a wide temperature range. Therefore, it is more important to produce polymer materials on the basis of metal-containing compounds and apply them in practice. According to the results of this study, the introduction of metal phosphates into the polymer improves the physical and mechanical properties of polyolefins. The physico-mechanical properties of the obtained polymer composite materials were improved up to 2.0 times compared to the initial polyethylene, polypropylene samples without filling. As a result of the addition of fillers, the elasticity, impact and fire resistance of polymers increased up to 2 times. The aim of this work was to study the effect of complex improvement of the physical and mechanical properties of polyolefins, in particular, on the heat resistance and flammability of composites through the introduction of metal phosphates into the polymer. For this purpose, scanning electron microscopy (SEM), shape and morphology of nanoparticles, X-ray diffraction. Thermogravimetric Analysis (TGA) and similar characterization methods were used. Analyzes have shown that the addition of fillers helps to increase the fire resistance of the polymers.

Introduction

Currently, there is a growing interest in the use of industrial polymers (PE, PP, PA) and polymer composite materials based on various nanoparticles by the general public. Among the fillers, minerals such as metal oxides have a special place. The low cost of these minerals in our country, the breadth of their physicochemical, mechanical and other properties, further increases the possibility of their use in the polymer industry. Special mention should be made of the research work carried out in recent years by S.S. Negmatov, N.R. Ashurov, A.T. Djalilov and young scientists P.J. Tojiev, N.Kh. Bozorova, D. Kenjaev and others. They have developed a technology for obtaining anti-corrosion coatings and similar materials by developing suitable additives for modification of polymers, studying a wide range of properties of the resulting product. [1]. The cheapness of metal oxides increases the breadth of physicist-chemical, optician, mechanic and other properties, their possibility of use in the polymer industry. As a result of scientific research work, the development of appropriate additives for modification of polymers, the development of technologies for obtaining coatings anti-corrosion, flammable heat-resistant materials by studying the properties of a wide range of products, increase the efficiency of construction and production composites [2]. In addition, the exchange of inorganic cations with organic cations in the layers of mineral particles has

been studied. However, research in the use of reaction-active modifiers with individual and multi-function effects is an important focus in improving the quality of additives, anticorrosion coatings, and flammable materials. Scientific research is carried out to study the dependence of the obtained composite materials on the nature and composition of colloid-chemical, physicochemical and operational properties of modifiers, to obtain and create technology of polymer composites for convenient and inexpensive special properties based on local raw materials. The modification of metal phosphates with polyethylene (PE), and polypropylene (PP) is indispensable for public safety and environmental protection. Modification of PE and PP with flame retardant (ammonium polyphosphate) is recommended. In this work, its microencapsulation was proposed using metal oxides as a filler, which is PE and PP composites with filler (MeO), ammonium polyphosphate (AP). The fire resistance of composite samples with different compositions was tested according to the UL-94 method, it was found that the PE/3-5% composite had the best performance. In addition, thermogravimetric analysis showed that this composite showed a higher decomposition temperature than pure PE and PP composite, which revealed the synergistic effect of PN in improving the flame resistance of PE composites. Thermal reactions of aluminum and titanium powders with fluorinated polymers obtained in stoichiometric ratios were studied. The effect of the strength of the aluminum-halogen bond on the amount of heat released by the interaction of the powdered metal with the halogen oxidant is shown. Influence of production and modification on the properties of natural rubber and use of nanocomposite material: natural rubber/nano- ZnO. Modified nano-ZnO is well distributed in natural rubber. An increase in the mechanical properties of the rubber was observed with a decrease in the ZnO dose. Reducing the amount of ZnO in rubber is in line with the general focus on protecting the environment. The use of nano-ZnO in tires and shock absorbers has been proven to be very effective. Therefore, in this study, the refinement of ammonium polyphosphate compounds to the above-mentioned fillers resulted in fire-resistant nanocomposites.[3-5].

Materials and Methods

The study used thermogravimetry, differential scanning calorimetry and differential thermal analysis (TGA, DSC, DTA), IR-infrared spectroscopy, ASM, X-ray analysis and others [6-9]. Scientific substantiation of the following solutions for the production of automotive and household plastic parts on the basis of new composite materials: selection of various reactive modifiers for micro and nano-sized mineral modifiers added to polymers; modification of polymers using dispersed particles allows to significantly change the properties of the polymer by adding various additives, to regulate the technological and operational properties. In particular, for the purposeful improvement of the physicochemical properties of PE, PP modification methods consisting of the creation of new composite materials are now widely used. The choice of modifier, the composition of which depends on the nature of the polymer, is one of the most convenient and inexpensive ways to obtain a polymeric material with different properties. [10–12].

Polymers were treated with organometallic compounds as fillers under laboratory conditions using the literature. Using metal-organic compounds as a filler, add 2 gm to a float tube at the bottom, Ammonium polyphosphate was obtained and 2 gm. The reaction mixture has been heated in an electric furnace to increase a temperature up to 90-220 °C by adding cobalt oxide (CoO). The solubility of the substance is observed at a temperature of 120 °C. The mixture has been stirred vigorously while heated to a temperature up to 120-180 °C. As a result, after 3-4 hours a cookie-like viscous mass is formed. The resulting powdery viscous mass was transferred to a porcelain bowl and dried in an oven about at 80-90 °C for 24 hours. The dried sample is pulverized in a mortar and the low molecular weight compounds were first washed with a 3% concentrated NaOH of solution and then several times with distilled water. The resulting product consisted of fine, dark red grains with a reaction yield of 85% [13–15].

Results and its discussion

The process of chemical modification of polymers involves the improvement of their physical, mechanical and chemical properties by the introduction of new functional groups into the polymer macromolecule through copolymerization. In this study, nanocomposites were obtained by processing PE and PP using metal oxides. As can be seen from Table 1, the properties of Ammonium polyphosphate metal nanoparticles and polymer-based nanocomposites have changed relative to the original polymers. The results of the analysis show that the inclusion of metal particles in the polymer composition improves the physical and mechanical properties of the polymers. Moreover, as well as large-scale measures are being taken to develop a scientific basis for the production of multi-component coatings and supply the domestic market with import-substituting products. The changes in the physicomaterial properties of the polymer are given in Tables 1 and 1.1 below [16-18].

Table 1

The composition obtained on the basis of polyethylene physical and mechanical properties

Parameters	Standards	HDPE-J2210	PE+ 3% ZnO	PE+ 3% Al ₂ O ₃	PE + 3% Co	PE + 3% NiO	PE + 3% FeO
Density, g/sm ³	ASTM D1505	0,96	0,98	0,99	0,99	0,99	0,99
Flexibility module, MPa	ASTM D 790	1100	1300	1200	1260	1310	1350
Elongation,%	ASTM D 638	300	310	300	310	280	290
Consistency limit, MPa	ASTM D 638	22	23	23	24	24	23
Izod effect viscosity, according to s/n,+23°C, kJ/m ²	ASTM D 256	4	4,5	5	4	5	4
HDT 0.45 MPa, °C	ASTM D 648	72	76	82	84	84	82
Cutting after 24 hours	ASTM D 955	1,5	1,4	1,35	1,4	1,35	1,4
Combustion rate UL-94, mm	Sample thickness 3,2 mm	45	≤40	≤40	≤40	≤40	≤40

Table 2

Polypropylene based composition physical and mechanical properties

Parameters	Satandarts	PP-J350	PP + 3% ZnO	PP + 3% Al ₂ O ₃	PP + 3% Co	PP + 3% NiO	PP + 3% FeO
Density, g/sm ³	ASTM D 1505	0,9	0.99	0.99	0.99	0.99	0.99
Flexibility module, MPa	ASTM D 1238	1280 100	1355	1310	1380	1300	1310
Elongation,%	ASTMD 790		98	95	100	95	96
Consistency limit, MPa	ASTM D638	24	24	26	25	25	25
Izod effect viscosity, according to s/n,+23°C, kJ/m ²	ASTM D638	6.5	6.51	6.4	6.1	6.2	6.4
Cutting after 24 hours	ASTM D648	1,2	1.6	1,05	1,05	1,05	1,15
Combustion rate UL-94, mm	Sample thickness 3,2 mm	45	≤40	≤40	≤40	≤40	≤40

In the analysis of the above results, the introduction of metal oxides modified by Ammonium polyphosphate compounds increased the physical and mechanical properties of polymers, in particular, the elastic modulus from 1280 MPa to 1355 MPa, bending under load increased the temperature by 45-50 °C.

The addition of metal phosphates to the polymer increased the thermal stability of the composite and shifted the decomposition onset temperature to a higher temperature range. That the reinforcement of polyethylene metal phosphates leads to a slight increase in the temperature of the onset of crystallization and a slight increase in the temperature of the onset of liquefaction of the composites. Also, the modification of metal phosphates leads to a decrease in the degree of crystallinity of polyethylene.

As a result, the phase structure of polyethylene can be explained by strong adhesion between matrix particles and metal compounds, which leads to the formation of new adsorption layers at the phase boundary and at the junctions of amorphous components.

If the interaction energy at the interface between the matrix and the modifier (dispersion and van der Waals forces) increases the interaction energy of polyethylene carbon macromolecules and leads to the formation of a crystalline phase, then crystalline claws may disappear, and the degree of crystallization may decrease. [19-20].

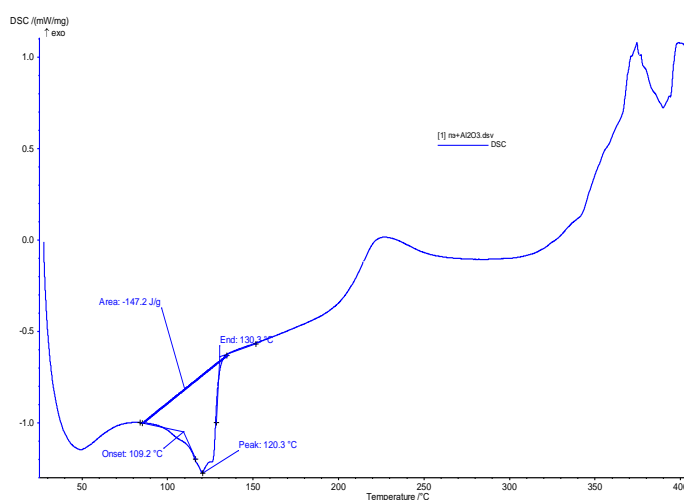


Figure 1. DSK diagram for metal phosphate modified polyethylene: PE/3% Al_2O_3

Figure 1 shows DSC thermograms of melting (sections from 109 to 147°C) and crystallization (sections from 120 to 430°C) of the initial PE, MeF polymers and their mixtures of 20 wt.metal % polar polymer.

For all composites, the ΔH liquid value is greater than the initial value of pure PE (152.4 J/g) and changes with increasing content of the modifier. This fact shows that the degree of crystallization of polyethylene usually does not depend on the degree of the modifier (Table 3). Based on the DSC diagram (Fig. 1), the process of heating polyethylene and nanocomposites is shown.

All samples have an endothermic melting point. Table 3 shows the pour points for nanocomposites PE/modifier with a content of 1, 3, 5%. For composites, the pour point slightly increases with increasing content of the modifier. The onset temperature of the liquefaction and the width of the liquefaction peak of the nanocomposites indicate the dispersion of the crystals. [21-22].

The analysis of the polymer composite material obtained as a result of the reaction mixture of polypropylene filled with metal phosphates was carried out at the Institute of Chemistry and Physics of Polymers AcSci RUz, and the bending radius of the needle is 10 nm. Lithium silicon cantilever, the size of the scanning area was 1-50 microns. The presence of atomic particles of metal oxide contributes to a significant increase in heat resistance.

Table 3

Differential Scanning Calorimetry data for polyethylene-based composites modified with 3% metal phosphate

Composite composition	T liquid, Operating systems	TPippl, °S	ΔH, enthalpy, J/g	T crist. start., °S	Tpikkr, °S	Cristal Level %
PE	125	116	152	134	139	63
PE+3% Al ₂ O ₃	120	109	147	130	130	68
PE+3% NiO	130	122	152	128	129	65
PE+3% CoO	127	123	153	132	135	71
PE+3% Fe ₂ O ₃	129	103	152	120	132	66
PE+3% ZnO	130	113	156	132	141	69

The TGA and DTA analysis of the modified polypropylene can be seen in Figure 2 below.

Two endothermic and two exothermic processes occurred in the TGA and DTA analysis of polypropylene modified on the basis of nickel oxide ammonium polyphosphate compounds. The first decomposition phase of the substances obtained for inspection was 28.480 °C, with 10.768 % from 310.180 °C to 31.768 %. This is explained by the formation of nitric oxide during decomposition. The second phase of the process was a loss of 3,402 mg from 3, 180 °C or a loss of 100, 365 %, the main phase from metal decomposition to 600, 390 °C. Oxides and nitrogen compounds are visible. In the DTA analysis, heat absorption was observed at a temperature of 168.490 °C in the first stage of the initial process of decomposition of heat and metal oxides. In the second stage, decomposition of carbonates and nitrates was observed at a temperature of 435, 250 °C

Thermal heating, i.e. in the first stage of an exothermic process, produces nitrogen oxides at a temperature up to 144.9×10 °C. The second stage of the exothermic process is observed at a temperature of 280 and 480 °C, which leads to the formation of urea and nitrites. [23–24].

Reinforcement of polypropylene with metal oxides leads to a slight increase in the onset temperature of crystallization, and a slight increase in the onset temperature of liquefaction of composites is also observed. Also, modification with metal oxides leads to a decrease in the crystallization rate of polypropylene. As a result, the phase structure of polypropylene can be explained by the strong adhesion between the particles of the matrix and the metal compounds, which leads to the formation of new adsorption layers at the interface of the phases and junctions of amorphous components. The study of the composition and structure of the polymer composite obtained by analysis of the synthesized modifier using a scanning electron microscope can observe the dispersion of reactive and non-reactive substances, as well as porous objects [25-26].

Table 4

Analysis of TGA and DTA results of polypropylene modified on the basis of Ammonium polyphosphate compounds with cobalt and nickel oxide

Temperature, °C	Lost of the mass mg (4,5)	Lost of the mass, %
100	0,060	1,33
200	1,125	25
300	2,012	44,7
400	2,896	64,4
500	3,402	75,6
600	4,215	93,67

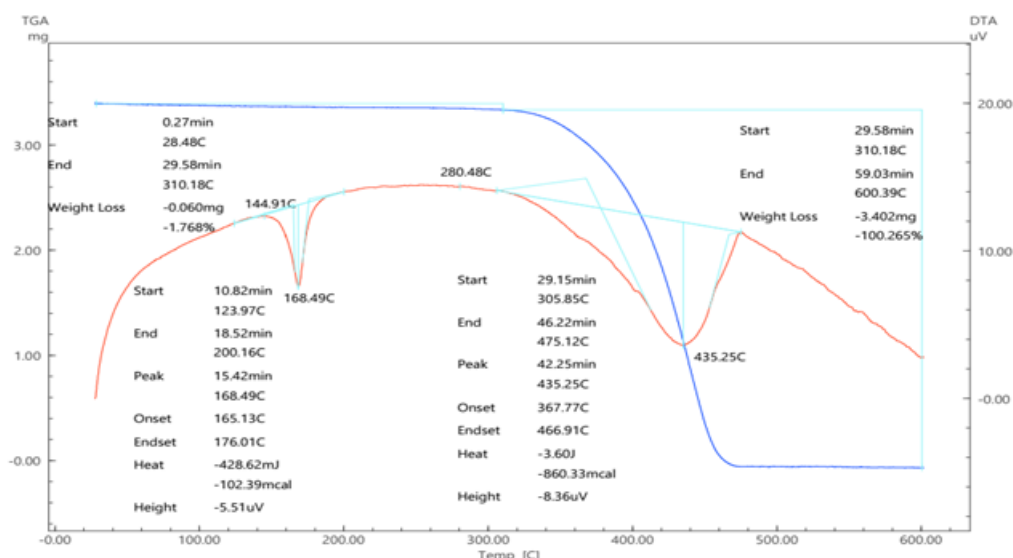


Figure 2. TGA and DTA analysis of polypropylene modified on the basis of Ammonium polyphosphate compounds with nickel oxide

It is also important to evaluate the required morphology, variance, and other parameters. However, the structure can be observed and studied at different technological stages of production.

The following figure 3 shows that there are no large agglomerates that can form sensory morphology metal particles between the polymer phases.

The appearance of the micro-images indicates that under these experimental conditions a harmonize morphology is formed between the metal particle modifier and the polymer phases. The absence of large agglomerates that can form the metal particles indicates that they are uniformly mixed with polymer matrices.

The above analysis shows that (Fig. 3) polymer composite material obtained on the basis of PE/Nickel oxide 3% has been magnified 3 times, and even when the sample size has been increased to 1000 times using an electron microscope, no particles were observed on the surface. This indicates that in obtaining the composites during the extrusion process, the metal particles are evenly distributed throughout the polymer.

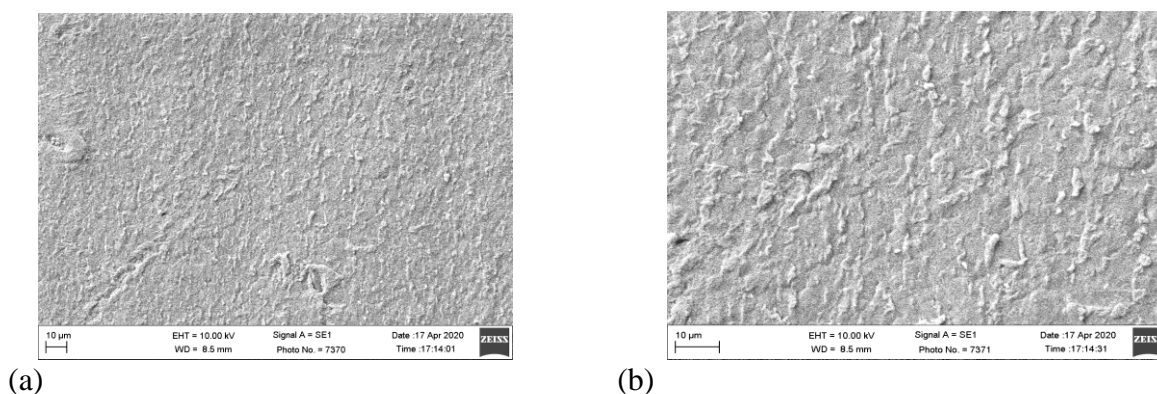


Figure 3. (a) PE/NiO 3%, (b) PE/NiO 3% Magnified 500 times. Magnified 1000 times

When the X-ray radiation phase of polymer composites is studied, this is accomplished by adding a large amount of weak absorbent (absorbent) or a small amount of strong absorbent to the diluted sample. These additions reduce the effect of the matrix. Analysis of the X-ray radiation spectrum gives qualitative results about the elemental composition of the sample. Comparing the spectrum of specimens with the spectra of specimens of known composition gives quantitative results (after some mathematical corrections for absorption, fluorescence, and atomic number) or diffraction gratings) propagate them by diffraction according to the wavelength, and finally, the

detector is placed at their focal points. From the radiographs obtained, it was studied that 3% of all the synthesized metal oxide particles had a size of 11.59 nm. Studies have been conducted to determine the degree of mixing of the synthesized metal oxides and their polymer composition. In the study of the particle size before the addition of metal oxides to the polymer and in the composition of the polymer material, it was found that the difference was 1.6nm. This fact is proved by the homogeneity of the synthesized metal oxides.

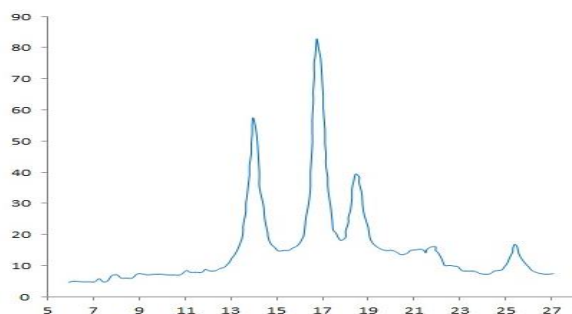


Figure 4. X-ray diffraction of PE sample

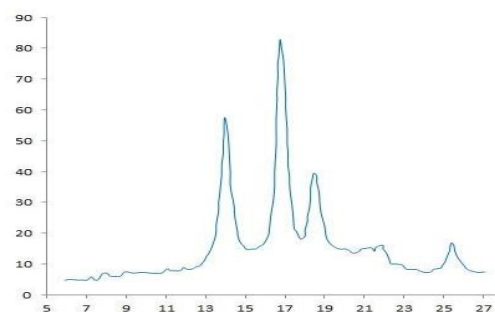


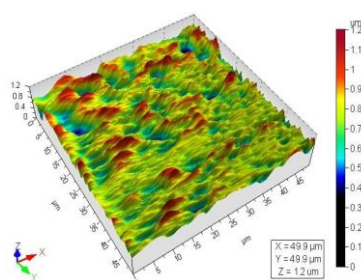
Figure 5. PE / NiO 3%. X-ray diffraction

It was found that the properties and technological performance of polymer composite materials depend not only on the type of modifier and the size of the added particles. Thus, the results of X-ray phase analysis confirm the presence of layered nanomodifiers in the crystallization process of polymers.

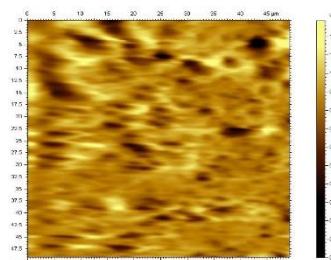
The development of modern nanotechnologies in the field of polymeric materials requires constant in-depth knowledge of the internal structure of micro- and nano-grade materials and their physical properties. While there are many ways to solve these problems, a new method—scanning Atomic Power Microscopy—can help achieve greater accuracy. Through the effective use of traditional methods, we can achieve high results. One of the most effective ways to solve these problems is atomic force microscopy (AFM) [27-28].

ILC is widely used to study the specificity of the topographic and microstructure of the surface of various materials. This method is high-pixel and sensitive and can shape the surface of the sample obtained in the nanoscale range on a three-dimensional surface. Numbers show changes in the size, shape, particle surface, and mechanical properties of materials on the surface through a processing program. In this section, the effect of modifying metal compound particles on the morphology of the polymer surface is studied. The study and analysis of the surface of modified polyethylene and polypropylene reveal the distribution of polymer macromolecular metal particles and their interaction properties.

The results were studied in relation to a polymer composite material obtained from a reaction mixture of polyethylene and polypropylene and 3% nickel oxide, and only a complete study of the sample itself was conducted, as we believe that the established laws would be valid for the remaining samples. [29-30].



(a)



(b)

Figure 6. Nickel oxide / PE based on Ammonium polyphosphate compounds:
(a) - three-dimensional image (b) - two-dimensional image.

The results show that pure polyethene surface is 100 nm, polyethene surface solution + 3% nickel oxide is 120 nm.

The results show that the surface roughness of pure polyethylene is 100nm, polyethylene +3% Zinc oxide surface roughness is 114nm. According to IR spectroscopy analysis, absorption bands characteristic of PE were observed. There is also an absorption band in the region of 1643, 1554 cm^{-1} of the average intensity, which belongs to the vibrations of the C=O group. A strong absorption observed in the 1080-145 cm^{-1} region is associated with flat deformation vibrations of methyl groups. Intense absorption lines of metals appear at 667 and 597 cm^{-1} .

The IR spectrum of the composite material obtained in the presence of zinc oxide shows that the presence of S and N (hydrogen) bonds is observed in the region of 2915 and 2848 cm^{-1} . CH_2 and CH_3 groups are observed in the region of absorption frequencies of 1400, 1471 cm^{-1} . Absorption at the frequency of 600-719 cm^{-1} confirms the characteristics of metal phosphates [31-36].

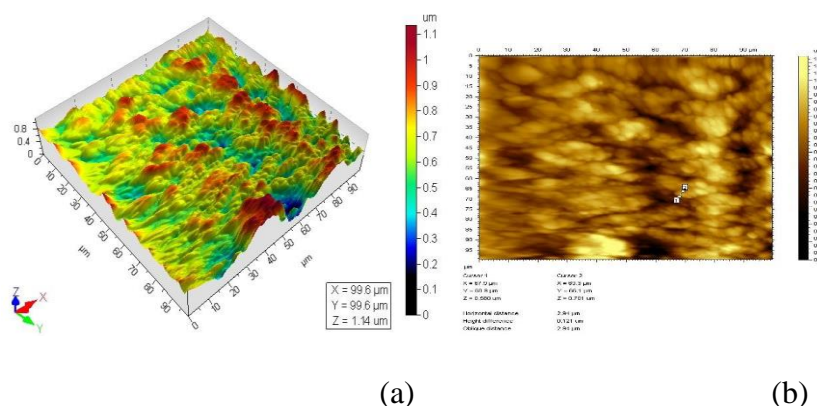


Figure 7. Zinc oxide / PE based on Ammonium polyphosphate compounds
(a) - three-dimensional image (b) - two-dimensional image.

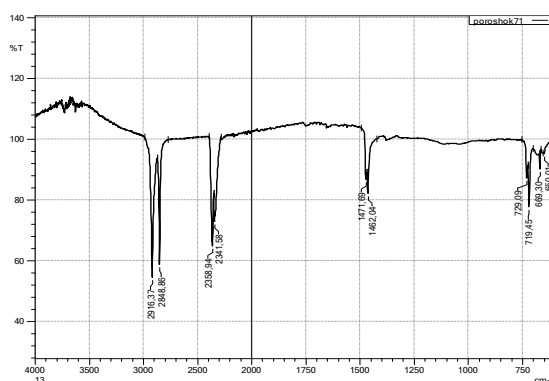


Figure 8. PE/ZnO 3%. Visibility in the IR spectrum

Phosphorus-containing fillers, such as ammophos and metal phosphates, are practical natural substances, which prevent ignition after ignition and reduce secondary ignition. This happens due to the carbonized layer of phosphorus, which is resistant to long-term heating. It is also observed in compounds with a traditional effect. In order to study the fire resistance properties of composite materials filled with metal oxides, the oxygen indicator parameters of the polymer were studied using test experiments.

For this, a sample of 50:25 mm in size was prepared in accordance with GOST 12.1.044-2018 and experiments were carried out on the device (model of the device for measuring the limited oxygen index: GD-ISO 4589-1), available in the laboratory of the Tashkent Institute of Chemical Technology. Institute for the purpose of determining the oxygen index.3 A sample of 50:25 mm in size was taken from the body and placed in the device. The oxygen index was measured up to 18%

before burning the sample. At each stage of the practical experiment, the oxygen index was kept at 1% in the process until the sample burned out. In the process, when the oxygen index reached 20.2%, a burning state of the sample was observed. A 50:25 mm coating sample was found to burn for 3 minutes 86 seconds at an oxygen index of 20.2%. In the course of a practical experiment, it was proved that the average value of the oxygen index of polymer composite materials filled with metal phosphates is 41.

Table 5

Influence of composite materials filled with mineral oxides on heat resistance and flammability

Compositing structure	Burning time s	Mass loss during combustion in air %	Oxygen index, % vol.
PE - 2210	240	61	17
PE + 1% MeF	114	30	20
PE+ 3% MeF	115	31	22
PE + 5% MeF	116	34	24
PP-J350	239	50	19
PP+ 1% MeF	123	25	21
PP+ 3% MeF	124	27	24
PP + 5% MeF	125	30	26
PA-66	240	58	25
PA+ 1% MeF	113	28	26
PA+ 3% MeF	115	29	27
PA + 5% MeF	116	30	28

Especially, with the introduction of PE fillers into the composition of polymers, the burning time decreased from 240 °C to 116 °C, the oxygen index increased from 17 to 24%, respectively, compared with the original polyethylene. [37-38]

And also, PP fillers burning time decreased from 239 °C to 125 °C, the oxygen index increased from 19 to 26%, respectively, compared to the original polypropylene. PA fillers, the burning time decreased from 240 C to 116 C, the oxygen index increased from 25 to 28%, respectively, compared with the original polypropylene (Table 5).

As a result of filling polymers with metal phosphates, their combustion time slows down by a factor of two compared to the initial polymer, and the mass loss during combustion in air is relatively reduced. A positive change in the flammability indices upon the introduction of metal phosphates into the composition of all polymers confirms the difficulty of burning the resulting composite materials. [39-40].

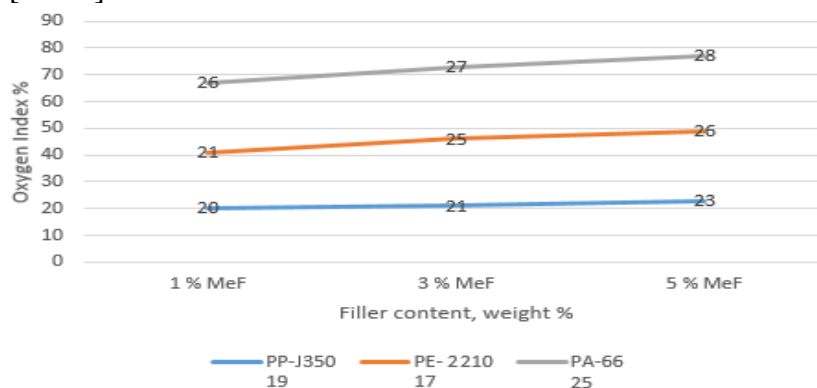


Figure 8. Flammability concentration (KI indicators) of polyethylene, polypropylene and polyamide-66 (1-4)

Conclusions

As a result of modification of polymers on the basis of various metal oxides and ammonium polyphosphate compounds, it was found that the physical and mechanical properties of polymer composite materials are improved, in particular, particles of zinc, nickel, cobalt, iron and aluminum oxides act as polymer macromolecules as modifiers of the crystal structure. The obtained results showed that as a result of filling PE, PP and PA with metal oxides with ammonium polyphosphate, despite the different nature of polymer and metal particles, it was found that all polymers have the same physical and mechanical properties improvement effect. As a result of joining Rolimer molecules with metal binders, the heat resistance of polymers increased by 2 times. Burn time from 239 C to 125 C, an increase in oxygen index from 19% to 26% was achieved. At the same time, as a result of filling polymers with metal phosphates, the modulus of elasticity increased from 1100 MPa to 1350 MPa, and the bending temperature under load increased by 45-50 °S. The addition of modifiers doubled the flexibility, impact and fire resistance of polymers. During this study, metal treatment of polymers based on ammonium polyphosphate compounds was found to be effective and it was recommended to add 1-3% to it.

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