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Study of Polypropylene, Filled With Nitrogen-, Phosphorus- And Metal--Containing Oligomers: Structure and Properties

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Absract

The synthesis of fire-resistant oligomers, used as additives for protection of polymer materials from fire and various aggressive environments - is currently an urgent problem for chemistry of high-molecular compounds. In this regard, we have studied in detail heat-resistant and mechanically strong thermoplastic composite materials, obtained by filling polypropylene (PP) with two new nitrogen-, phosphorus- and metal--containing oligomers.

One of these additives was synthesized, based on carbamide dihydrogen phosphate, ethylene glycol, melamine and magnesium oxide (DP.EG.M.MO), and the second is based on ammonium phosphate, carbamide, sodium metasilicate and zinc oxide (AP.C.SM.ZO). Using these additives, we created two new composite materials with polypropylene. After this, were studied in detail the physical and mechanical properties of the resulting polypropylene compositions, filled with protective oligomers: their bending strength, tensile strength and relative elongation. To obtain a composite material, based on polypropylene, the content of nitrogen-, phosphorus-, and metal-containing oligomer additives was 5% by weight.

The resulting composites were studied by IR spectroscopy. Based on the analysis of IR spectra, it was assumed, that polar oxygen-containing fragments, formed on the surface of the fillers, can participate in adsorption interaction with the polyethylene matrix, which can lead to an improvement of polypropylene-based nanocomposites. The developed composite materials were studied for flammability: their burning rate was determined, and their thermophysical properties were studied, using thermogravimetric and differential thermal analysis (TG and DTA).

It has been established, that oligomeric additives, containing metal, nitrogen and phosphorus are capable of both increasing the physical and mechanical properties of polymers and reducing their burning rate, which means they can be used as fire-fighting fillers for polymers. The synergism of the nitrogen-phosphorus system can be explained by the formation of P-N bonds during thermal decomposition, which promote phosphorylation of the polymer and enhance the effect of flame retardants as a dehydration catalyst.

Keywords: polypropylene; metal-containing oligomers; atomic force microscopy; bending deformation; tensile strength; heat resistance; mixtures and composites; functional polymers; burning rate.

Introduction

When producing polymers for composite materials, various fillers are often added to them to improve the physical, mechanical and thermal properties. This improvement in properties can be achieved by introducing primarily nitrogen-, phosphorus- and metal-containing oligomers as fillers [1].

Fire-safe polymers are polymers that are resistant to degradation at high temperatures. There is need for fire-resistant polymers in the construction of small, enclosed spaces such as skyscrapers, boats, and airplane cabins [2]. In these tight spaces, ability to escape in the event of a fire is compromised, increasing fire risk. In fact, some studies report that about 20% of victims of airplane crashes are killed not by the crash itself but by ensuing fires [3]. Fire-safe polymers also find application as adhesives in aerospace materials [4] insulation for electronics and in military materials such as canvas tenting [5].

Some fire-safe polymers naturally exhibit an intrinsic resistance to decomposition, while others are synthesized by incorporating fire-resistant additives and fillers. Current research in developing fire-safe polymers is focused on modifying various properties of the polymers such as ease of ignition, rate of heat release, and the evolution of smoke and toxic gases [2].

Standard methods for testing polymer flammability vary among countries. in the United States common fire tests include the UL 94 small-flame test, the ASTM E 84 Steiner Tunnel, and the ASTM E 622 National Institute of Standards and Technology (NIST) smoke chamber [2]. Research on developing fire-safe polymers with more desirable properties is concentrated at the University of Massachusetts Amherst and at the Federal Aviation Administration where a long-term research program on developing fire-safe polymers was begun in 1995. The Center for UMass/Industry Research on Polymers (CUMIRP) was established in 1980 in Amherst, MA as a concentrated cluster of scientists from both academia and industry for the purpose of polymer science and engineering research [2].

In works [6,7] we have previously obtained and proposed new nitrogen-, phosphorus- and metal-containing compounds as fillers for polymers, on the basis of which more than ten new composite products were synthesized. Oligomeric fire-fighting fillers based on carbamide, ammophos and metal oxide can increase the heat resistance of polymer materials [8].

We have already obtained nitrogen-, phosphorus-, and metal-containing oligomers of grade "17-A" [9], which can be used as a filler for polypropylenes, and we have determined the thermophysical efficiency of a composite material using it.

These oligomers, or more precisely, their physicochemical properties, are the object of study in the presented work.

Nitrogen-, phosphorus- and metal-containing oligomers and composites were also studied in detail in the work [10].

In work [11] were studied the physicochemical and thermophysical properties, and was also carried out an analysis6 using an Atomic Force Microscope, for a number of oligomers, which, when added to polymer materials, enhance heat-resistant properties.

Previously, we found that the optimal weight concentration for improving the mechanical and fireproof properties of polymers is 5% by weight [8,9]. An assessment of the rheological properties for composites, filled with additives, showed that with an increase in the content of these oligomers, the fluidity of the compositions decreases, however, the resulting compositions can be processed by injection molding.

Materials and Methods

The Synthesis of Oligomeric Additives was Carried Out by the Following Way:

Commercially available chemically pure substances were used in the synthesis.

The reaction was carried out at pH 8-9 between carbamide dihydrophosphate (NH_2-CO-$NH_3^+ \cdot H_2PO_4^-$, CDP), ethylene glycol (HO-CH_2-CH_2-OH, EG), melamine (M), magnesium oxide (MgO) in relation to 2:1:0,5:0,5 at 165-170 °C. We conventionally called the additive to polymers, obtained in this way, DP.EG.M.MO. The structure of this additive, which is an oligomeric compound by chemical nature, can be represented as follows:
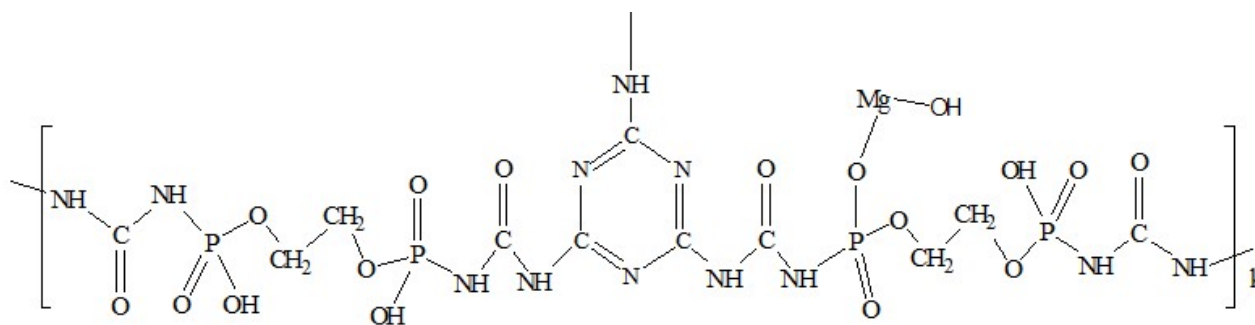
The second additive, conventionally called AP.C.SM.ZO, is obtained from ammonium phosphate $((NH_4)_3PO_4)$, carbamide $(NH_2\text{-}CO\text{-}NH_2)$, sodium metasilicate (Na_2SiO_3) and zinc oxide (ZnO) in relation to 2:1:0,5:0,5 at 165-170 °C:
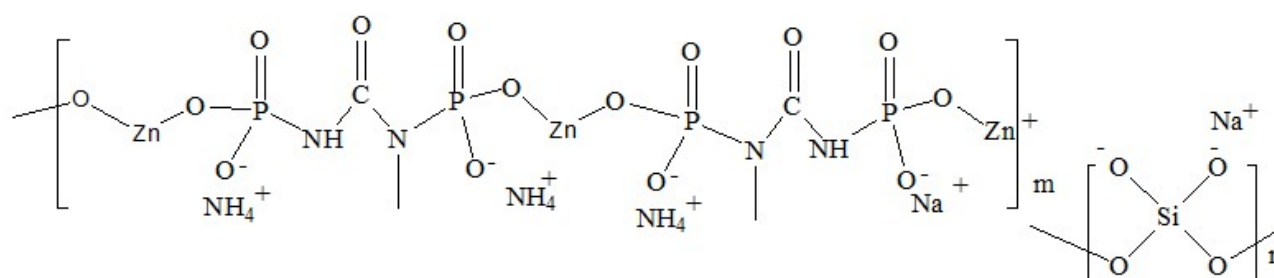
The calculated amount of carbamaide dihydrophosphate (3,16 g; 0,02 mole) (or ammonium phosphate (2,64 g; 0,02 mole) for AP.C.SM.ZO) and ethylene glycol (0,62 g; 0,01 mole) (or pre-molten carbamaide (0,60 g; 0,01 mole) for AP.C.SM.ZO) was loaded into a flask, equipped with a mechanical stirrer, thermometer and refrigerator. Mixture was stirred at 100-120 °C for 1 hour with simultaneous distillation of water (in the case of AP.C.SM.ZO ammonia was released). Next, the calculated amount of melamine (0,63 g; 0,005 mole) (or sodium metasilicate (0,61 g; 0,05 mole) for AP.C.SM.ZO) and magnesium oxide (0,20 g; 0,005 mole) (or zinc oxide (0,40 g; 0,005 mole) for AP.C.SM.ZO) was added and the mixture was stirred for 1 hour at 165-170 °C.

After cooling the mixtures in quantitative yield we obtain the resulting oligomeric compounds - dark yellow viscous masses.

Next, using the extrusion method, after adding the resulting oligomers to the polymer melt, composite materials with polyethylene were obtained. For this purpose, as well as as a comparison sample, was used granulated polypropylene (PP) JM350-Uzcorgaz.

Thermoplastic composite materials, based on polypropylene, were studied in detail using the following methods:

Determination of the Strength during Bending Deformation

The flexural (bending) strength of composites BS was determined in accordance with Russian State Standard (GOST) 4648-71 «Plastics. Static bending test method using double-support bending method». The tests were carried out on samples in the form of bars 55.5 (length) x 6 (width) x 4 (thickness) mm with an intersupport distance of 40 mm. The bending strength (BS, MPa) was calculated using the equation:

$BS = 1{,}5(F_b \cdot a)/(b \cdot c^2)$,

where BS – bending strength (breaking stress); a – intersupport distance; b, c – width and thickness of the sample, respectively; F_b is the magnitude of the applied load [12]. Flexural strength values were determined as the average of ten experiments performed for each sample.

Determination of the Strength Characteristics at Break

Tensile strength and elongation at break (TS), was determined in accordance with Russian State Standard (GOST) 14236-81 «Plastics. Tensile test method» (corresponding to standard testing method ASTM D882-97) using a testing machine RM-50. These parameters were determined at a deformation rate of 50 mm/min for a blade-shaped sample with geometric parameters 50 (length) x 6 (width) x 4 (thickness) mm. Tensile strength (TS, MPa) was calculated according to equation:

$TS = F_t/(a \cdot b),$

where F_t – is maximum load needed to pull the sample apart; a, b – thickness and width of the sample at the narrowest part, mm. So, a·b is cross-sectional area (m^2) of the samples.

Elongation at break (EB) determined by noting the initial length of the sample (L_0 = 50 mm) and increasing the length of the sample at the moment of its destruction (ΔL) according to equation:

$EB = (\Delta L/50) \cdot 100\%$

Determination of Fire Resistance and Flame Propagation Speed (Burning Speed)

Flammability tests for thermoplastics were carried out in accordance with Russian State Standard (GOST) 30402-96 «Construction materials, flammability test methods», corresponding to international standard ISO 5657-86, using a special equipment UL-94 for testing flammability. For the study were prepared 10 samples, having the shape of a rectangle with a side length of 165 mm, a width of 6 mm and a thickness of 4 mm. Asbestos sheets 10 mm thick were used as a non-combustible base. Before testing, the samples were kept until constant weight was reached at a temperature of 23±2 °C and a relative humidity of 50±5%.

The combustible gas flow (propane-butane mixture) was 20 ml/min, the air flow was 160 ml/min. The length of the burner flame was maintained at 15 mm. The constant heat flux density, acting on the initial fragment of the sample, gradually increased from 10 kW/m^2 to the value until ignition began within 15 minutes of continuous exposure to the heat flux on the sample. This value is called the Critical Constant Heat Flux Density (CCHDD). After the start of ignition, the time during which the entire sample burned was recorded. The burning rate v was determined by the formula:

$v = L_0/\tau,$

where L_0 – sample length, a τ – its burning time.

Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) is a technique, used to study the behavior of polymers when heated. DSC can be used to study the melting or glass transition of a crystalline polymer. The DSC setup consists of a measuring chamber and a computer. The two cups are heated in the measuring chamber. The first cup contains the material under study, and the second is empty and is used as a standard. A computer is used to control the temperature and regulates the rate at which the temperature of the cups changes. Typical heating rate is about 10º C/min. In this work, a DSC instrument Netzsch Simultaneous Analyzer STA 409 PG (Germany), with K-type thermocouple (Low RG Silver) and aluminum crucibles. All measurements were carried out in an inert nitrogen atmosphere with a nitrogen flow rate of 50 ml/min. The temperature range of measurements was 25-370 °C, the heating rate was 5 K/min. The amount of sample per measurement is 5-10 mg. The measuring system was calibrated with a standard set of substances KNO_3, In, Bi, Sn, Zn.

The degree of crystallinity of the samples is determined using the equation:

$\alpha = (\Delta H/\Delta Hcm) \cdot 100\%,$

where ΔHst - a standard value that represents the heat of fusion of a 100% crystalline polymer (293 J/g for PP). ΔH – heat of melting of the test sample.

X-ray Phase Analysis (XRF)

Identification of samples was carried out on the basis of diffraction patterns, which were recorded on an XRD-6100 apparatus (Shimadzu, Japan), controlled by a computer. Was applied CuK_α-irradiation and a constant detector rotation speed of 4 degrees/min in increments of 0.02 degrees, and the scanning angle varied from 4 to 80°.

Accurate results of X-ray phase analysis can be obtained by experimentally studying the size and shape of particles using an X-ray phase analysis method such as the Debye-Scherer method. The size of coherent distribution zones (CDZ) (size of nanocrystals) can be determined by the formula:

$Dp = K\lambda/(Bcos\theta),$

where Dp - average crystal size (nm), K - Scherrer constant. K varies from 0.68 to 2.08. For spherical crystals with cubic symmetry K = 0,94. λ - x-ray wavelength. Cu (Kα) = 1,54 Å. B - integral length of reflections in a diffractometer FWHM (full width at half maximum). cosθ - cosine of the x-ray diffraction angle.

Study of the Structure of Composite Materials Using IR Spectroscopy

IR spectra (device SHIMADZU IRAFFINITI-1, Japan) were removed from films of composite samples with a thickness of about 10 microns.

Atomic force microscopy

Analysis of polypropylene samples with refractory additives was carried out by atomic force microscopy (AFM) using silicon cantilevers with a tip rotation radius of 10 nm.

The size of the scanned area ranged from 1 to 50 μm. Microscopy was carried out in air using the semi-contact method, recording changes in the amplitude of oscillations of the counting needle, which made it possible to study the surface topography.

Statistical Analysis

The results statistically were performed by analysis of 5% significance level, using Minitab 17.0 software and MS excel. The experiments were repeated three times, while the standard deviations corresponded to the condition $p<0.05$.

Results and Discussion

The results of mechanical and refractory properties analyzes are shown in Table 1.

Table 1: Physico-mechanical properties of polypropylene, filled with nitrogen-, phosphorus- and metal-containing oligomers

Parameters	PP	PP + 5%DP.EG.M.MO	PP + 5%AP.C.SM.ZO
Elongation at break (EB), %	250	285	300
Bending strength (BS, MPa)	1,000	0,888	0,826
Tensile strength (TS, MPa)	0,799	0,794	0,763
Critical Constant Heat Flux Density (CCHDD), kWt/m^2	20,6	28,2	27,8
Burning rate (v, mm/min	45	40	40

Analyzes of the results show that the introduction of nitrogen-, phosphorus-, and metal-containing oligomers into polypropylene improves its thermophysical properties without significant deterioration in strength. It was previously established, that the use of nitrogen-, phosphorus-, and metal-containing oligomers as fillers contributes to a significant increase in the heat resistance of the original polypropylene [13-15].

Let us pay special attention to the last line of Table 1, showing a decrease in the burning rate of composites compared to polypropylene without additives.

To clarify the question of why the properties of the polymer are improved, the IR spectra of the AP.C.SM.ZO samples were determined (Figure 1):
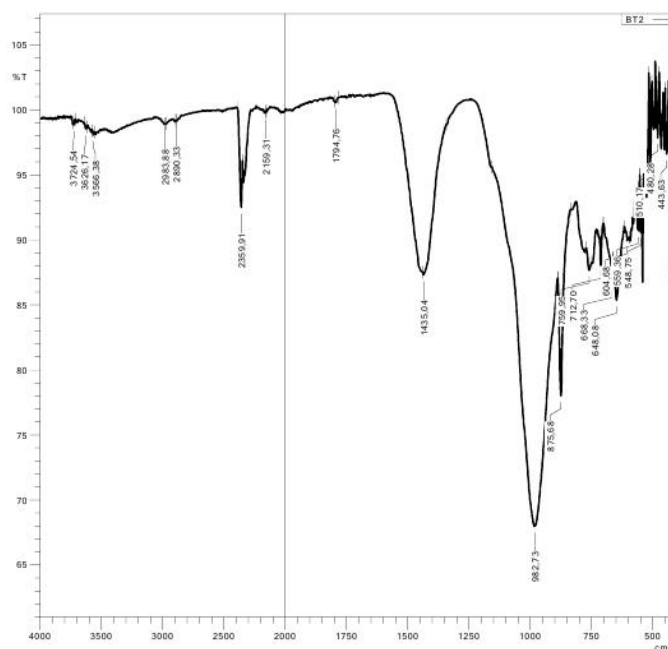

Figure 1: IR spectrum of compositions PP + 5% AP.C.SM.ZO

On the IR spectrum in the areas of 2850-1470 cm^{-1} there are absorption bands confirming the presence of -CH$_2$- groups; absorption bands corresponding to =CH–groups at 2987 cm^{-1}; and absorption bands in the region of 2892 cm^{-1} corresponding to =CH$_2$ group. Absorption bands in the regions of 2000 и 2800 cm^{-1}, confirm the presence of –NH groups. The presence of Si-O- groups is confirmed by an intense band in the region of 880-870 cm^{-1}.

Thus, it can be assumed that polar oxygen-containing fragments formed on the surface of fillers can participate in adsorption interaction with the polyethylene matrix, which leads to an improvement in some properties of polypropylene-based nanocomposites.

An important issue is to study the effect of the introduction of nitrogen-, phosphorus- and metal-containing oligomers on the morphology of the polypropylene surface [16,17]. The study and analysis of the surface of modified polypropylene shows the distribution of nitrogen-, phosphorus- and metal--containing oligomers between polymer macromolecules and their interaction. The results were obtained for a polymer composite material obtained from a reaction mixture of polypropylene with 5% DP.EG.M.MO additive.

Figure 2 shows the surface of polypropylene, modified with nitrogen-, phosphorus-and metal-containing oligomer (PP + 5% DP.EG.M.MO). It can be seen that the surface roughness of pure polypropylene is 100 nm, the surface roughness of PP+5% DP.EG.M.MO is 210 nm:
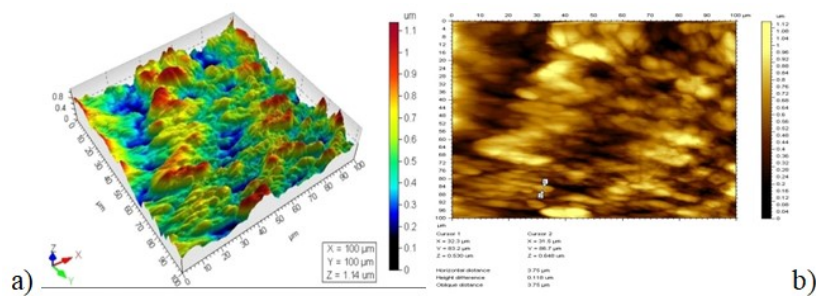
Figure 2: PP + 5% DP.EG.M.MO: a) - three-dimensional image, b) two-dimensional image

Figure 3 shows the surface of polypropylene modified with PP + 5% AP.C.SM.ZO. It can be seen that the surface roughness of pure polypropylene is 100 nm, PP + 5% AP.C.SM.ZO is 212 nm:
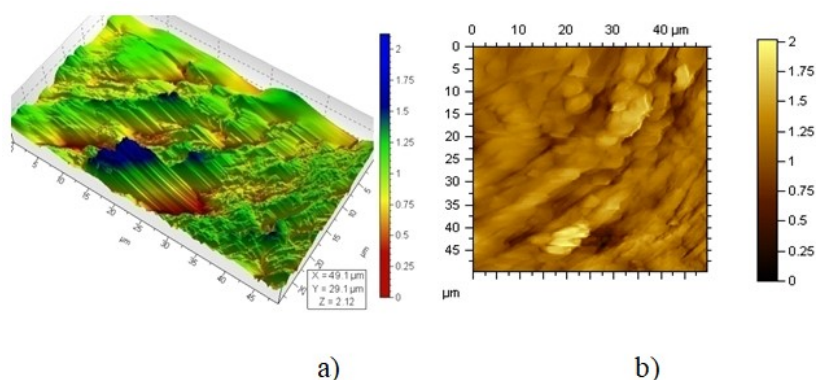
Figure 3: PP+5% AP.C.SM.ZO: a) - three-dimensional image, b) - two-dimensional image

Next, based on thermo-analytical studies, we studied the thermo-oxidative properties of the resulting composite materials. It is known, that polymers, filled with nanocomposites with nitrogen-, phosphorus- and metal-containing oligomers, decompose at higher temperatures, than pure polymers, and this decomposition is characterized by the formation of ash residue [18, 19]. This phenomenon indicates the thermal stability of the resulting composite materials.

Mechanical and operational properties of composite materials depend on chemical changes in the composition of polymers during their processing. But these processes take place at high temperatures. Therefore, in this work we studied thermal and thermophysical properties: change in melting point, heat resistance.

The melting point and crystallization rate of polymer composite materials were determined by differential scanning calorimetry (DSC) [20-22].

To determine the operating temperature range of polymers using differential scanning calorimetry (DSC), thermograms of samples, filled with refractory additive, were obtained.

The results obtained are presented in Tables 2 and 3:

Table 2: Thermodynamic properties of composite materials, based on PP + 5% DP.EG.M.MO

Composition	Start ofmelting, T °C	Melting Peak, T °C	Enthalpy,ΔH, J/g	Degree of crystallinity α, %
PP	160	224	188	55
PP+1 % DP.EG.M.MO	186	241	197	61
PP+3 % ДКЭММ	197	243	203	59
PP+5 % ДКЭММ	209	245	210	58

Table 3: Thermodynamic properties of composite materials based on PP + 5% AP.C.SM.ZO

Composition	Start ofmelting, T $^{\circ}$C	Melting Peak, T °C	Enthalpy,ΔH, J/g	Degree of crystallinity α, %
PP	160	224	188	55
PP+1% AP.C.SM.ZO	189	241	197	61
PP+3% AP.C.SM.ZO	195	243	203	59
PP+5% AP.C.SM.ZO	211	245	210	58

We also carried out a comparative analysis of the diffraction spectra for composite materials by type of filler (Table 4.5):

Table 4: Results of calculating the size of composite nanoparticles based on PP+5% DP.EG.M.MO according to the Debye-Scherrer formula

№	2Θ - Scan angle	Dp (nm) average crystallite size	Dp (nm) average
1	8.2	17.42	15.61
2	8.7	14.61	
3	21.3	15.12	
4	24.0	15.70	
5	26.2	15.23	

Table 5: Results of calculating the size of nanoparticles of a composite based on PP + 5% AP.C.SM.ZO using the Debye-Scherrer formula

№	2Θ - Scan angle	Dp (nm) average crystallite size	Dp (nm) average
1	6.03	24.03	23.53
2	8.56	17.94	
3	12.21	23.99	
4	24.87	29.65	
5	26.46	22.04	

The results showed that the higher the filler concentration, the higher the rate of its diffusion into the polymer structure.

Based on the results of X-ray phase analysis, it was established that the particle size in the resulting composite materials is in the range of 15-24 nm.

Conclusion

1. To improve the thermophysical properties of polypropylene, we have obtained two new nitrogen-, phosphorus- and metal-containing additives. One of them is synthesized based on urea dihydrogen phosphate, ethylene glycol, melamine and magnesium oxide (DP.EG.M.MO), and the second is based on ammonium phosphate, urea, sodium metasilicate and zinc oxide (AP.C.SM.ZO).

2. The preservation of physical and mechanical properties (modulus of elasticity, deformation, tensile strength) and improvement of thermophysical properties (burning rate) of polypropylene filled with new refractory additives have been established.

3. It is assumed that the improvement in physical and mechanical properties can be explained by the intrinsic strength and rigidity of nitrogen-, phosphorus- and metal-containing oligomers, which is transferred to the entire polymer structure.

4. The phase structure of polypropylene, modified with new

refractory oligomers, can be explained by strong adhesion between the matrix and oligomers, which leads to the formation of new adsorption layers at the interface between phases and the junction of amorphous components.

5. Modification of the polymer with the oligomers we obtained leads to a decrease in the degree of flammability of its surface and enhances the fire-resistant properties.

6. Based on the results of X-ray phase analysis, it was established that the particle size in the resulting composite materials is in the range of 15-24 nm.

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