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STRUCTURE AND PROPERTIES OF POLYMERS HAD BEEN FILLED WITH BIVALENT METAL PHOSPHATES

Abstract: *Polyolefins (PO) are the largest-tonnage, available and demanded from the whole range of polymers produced by the industry. Therefore, the search for technical solutions aimed at simplifying the processing of polyolefins and creating new types of composite materials based on them is an urgent task.*

The physical and mechanical properties of filled polyethylene and polyamide compositions, determination of the melt flow rate by viscisometry methods, determination of bending strength by two-support bending methods, determination of Charpy impact strength were studied.

The rheological characteristics of composite materials based on polyethylene with divalent metal phosphates have been determined.

To obtain a composite material based on polyethylene, the content of bivalent metal phosphates was changed from 1 mass to 5 mass parts. Evaluation of the rheological properties of filled composites showed that with an increase in the content of metal phosphates, the fluidity of the compositions decreases, but the resulting compositions can be processed by injection molding. The data obtained show that the optimal compositions are those containing: 3 mass.h. metal phosphates.

Key words: *polymers, composite materials, metal phosphates.*

Language: *English*

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Introduction

Today, the rapid growth of the world population and production volumes leads to a growing demand for polymer composites from year to year. In particular, the demand for polymer composite materials in industry and manufacturing is growing every day. In this regard, the

automotive industry makes high demands on the design of polymer materials. Currently, the basis of scientific research is the production of composite materials that meet several requirements, such as the modification of polymers, improvement of their physical and mechanical properties, the addition of additives without changing their composition [1; 2].

In this work, it was used as fillers for double condensed phosphates of divalent ammonium metals (DKFMeA) in polymeric materials of various compositions (polyamide-6, polyethene), which showed the promise of using some compounds of this class as fire retardants [3].

Method and materials.

The object of research is thermoplastic composite materials based on polyethene and polyamide. Estimating the Melt Flow Index (MFR), which is the selected temperature and viscosity of an average molecular weight melt, is usually a quantitative guideline for pipe manufacturers. The fluidity of a polyethene melt and composites based on it was determined in a capillary displacement viscometer under the action of a constant force of the IIRT-M type according to GOST 11645-73 (ASTM D 1238) [2] The bending strength of composites was determined according to GOST 4648-71 (ASTM D 638) [3] by the method of double-sided bending. Samples were tested as 55.5 x 6 x 4 mm beams with 40 mm side-to-side spacing. The flexural strength was calculated using the following formula. σi = 1.5 (Pa) / (bc^2).

In this study, the impact resistance was determined in accordance with GOST 4647-80 (ASTM D 638) according to Charpy [4]. The Charpy impact strength was calculated using the following equation: an = An / (ab), where an is the Sharpe impact force; An - threefold dissipated impact energy of destruction of the sample without cutting; a, b - width and thickness of the middle part of the sample; Sharpe exposure values were taken as the average of dozens of experiments performed for each sample. To test frost resistance, the samples were kept at a temperature of minus 30 ° C for 100 days, and then their impact resistance was measured using the Sharpe method. ... The tensile strength and elongation of the sample were determined in accordance with GOST 11262-80 (ASTM D 638) [5]. These parameters were determined on a cutting machine with a deformation rate of 50 mm/min on blade-shaped specimens 50 x 6 x 4 mm in size. The tensile strength (σp) of the samples was determined according to the following equation: σp = P / (a • b), where P is the load; a, b - thickness and width of the narrowest part of the sample, mm. Elongation at break (εp) was determined by the equation (εp) = (ΔL / L0) 100% taking into account the initial length of the sample (L0) and the increase in its length at break (ΔL).

Discussion of the results.

In this study, an improvement in the physical and mechanical properties of polyethene P-Y-342 and polyamide PA-6 in MeO - NH4PO3 systems was observed. The aim of this work is to improve the physical and mechanical properties of polymers as a result of their addition of phosphates of divalent metals [6]. This also implies the development of methods for producing composite materials based on nanosized modifiers that enhance the physicomechanical properties of polymers and affect polymer macromolecules.

Experimental part.

The process of chemical modification of polymers includes the improvement of their physical, mechanical and chemical properties by introducing new functional groups into the polymer macromolecule by copolymerization or crosslinking. [7] In this study, nanocomposites were obtained by modifying polyethene and polyamide with divalent metal phosphates. Table 1 shows that the properties of nanocomposites based on polyethene P-Y 342 and polyamide-PA-6 filled with phosphates of divalent metals changed in comparison with the original polymers. The results of the analysis show that the inclusion of nanoparticles in the polymer improves the physicomechanical properties of the polymers. [8]

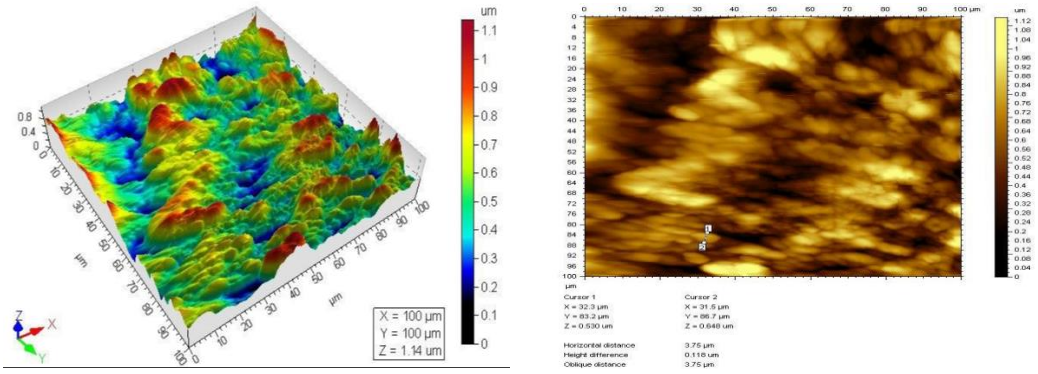
Table 1. Comparative analysis of the physical and mechanical properties of composite materials based on polyethene P-Y 342 and polyamide-PA-6 filled phosphates of divalent metals in MeO - NH_4PO_3 systems

The component of the composition	Impact strength, kJ / m2	σ bending, mPa	σ rupture, mPa	Elongation,%	Shrinkage%
	ГОСТ 4647-80	ГОСТ 4648-80	ГОСТ 14236-81	ГОСТ 14236-81	ГОСТ 18599-21
P-Y 342	50	24	21	750	3
P-Y 342/3% CuO - NH_4PO_3	56	35	33	174	2,7
P-Y 342/3% CoO - NH_4PO_3	60	36	36	170	2,8
P-Y 342/3% NiO - NH_4PO_3	68	38	48	155	2,2
ПА-6/	120	100	80	150	2,6

ПА-6/ 3% CuO - NH_4PO_3	125	118	92	114	2,8
ПА-6/ 3% CoO - NH_4PO_3	128	130	103	106	2,3
ПА-6/ 3% NiO - NH_4PO_3	130	146	114	96	2,8

When adding 3% NiO - NH4PO3 to polyethene, it can be seen that the impact resistance increased from 50 to 68 kJ / m2 compared to the original polyethene, the flexural strength increased from 24 to 38 MPa, and the tensile strength increased from 21 to 48 MPa. [nine]. In a composite material based on polyamide, it can be observed that the impact resistance increased from 120 to 130 kJ / m2 compared to the original polyethene, the flexural strength increased from 100 to 146 MPa, and the tensile strength increased from 80 to 114 MPa. [ten]. Thus, the addition of 3% NiO - NH4PO3 to polyolefins led to high physicomechanical properties of polymer composite materials. In all cases, it can be noted that the high hardness and strength of the results obtained were due to phosphates of divalent metals [11].

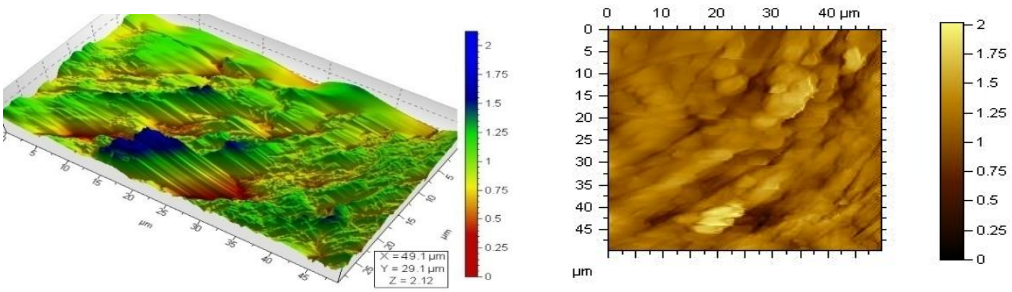
AFM (atomic force microscopy) is widely used to study the specificity of the microstructure and topography of various materials. This method is very sensitive to pixels and can shape the surface of a nanoscale sample onto a 3D surface. This method visually shows the change in surface, shape and size of particles, as well as the mechanical properties of the surface of the material. [12]. Also, in this work, we studied the effect of the modification of particles of phosphates of divalent metals on the morphology of the polymer surface. Analysis and study of the surface of modified polyolefins show that particles of divalent metal phosphates are scattered among the polymer macromolecules and interact. The results of AFM analysis of a composite material based on polyamide-6 and polyethene with double condensed phosphates of divalent nickel and ammonium were obtained. The analysis was carried out on AFM using silicon cantilevers with a turning radius of 10 nm [13].



а) б)

Figure 1. AFM images of a composite material based on polyethene with bivalent nickel phosphates a) three-dimensional image, b) two-dimensional image.

The scanning area ranged from 1 to 50 µm. Microscopy was carried out in the air by the semicontact method; on the lines of registration of changes in the amplitude of oscillations of the reaction axis, oscillations of the intermediate movement and the surface relief are reflected, which indicates the adhesion of individual surfaces to each other. In fig. 1 shows the surface of polyethene modified with divalent nickel phosphates. The results show that the roughness of pure polyethene is 100 nm, and the surface roughness of polyethene modified with aluminium oxide is 210 nm [14].



а) б)

Figure 2. AFM images of a composite material based on polyamide-6 and bivalent nickel phosphates: a) - three-dimensional image, b) - two-dimensional image.

In fig. 2 shows an AFM image of the obtained composite material, according to which the roughness is 100 nm in polyamide-6 and 122 nm in a composite material based on PA-6, obtained from divalent nickel phosphates.

In this work, on the basis of thermoanalytical studies, the thermo-oxidative properties of the obtained composite materials are studied. It is known that polymer composite materials filled with nanocomposites with divalent nickel phosphate particles decompose at higher temperatures than pure polymers, and this decomposition is characterized by the formation of an ash residue. This indicates the thermal stability of the obtained composite materials. The complex physical and mechanical properties of composite materials are determined on the basis of chemical changes in the composition of polymers during their processing. These processes take place at high temperatures. In this work, the thermal and thermophysical properties were studied: the change in the melting point, heat resistance.

The melting point and crystallization rate of polymer composite materials were determined by differential scanning calorimetry (DSC). The results are shown in Table 2.

Table 2. Thermodynamic properties of composite materials based on PA-6 polyamide with divalent metal phosphates

The component of the composition	Melting starts, T° C	Melting peaks, T°C	Enthalpy, ΔH, Дж/г	Crystallinityα, %
ПА-6/	220	224	188	55
ПА-6/ 3% CuO - NH_4PO_3	236	241	197	61
ПА-6/ 3% CoO - NH_4PO_3	237	243	203	59
ПА-6/ 3% NiO - NH_4PO_3	239	245	210	58

To determine the range of operating temperatures of polymers by differential scanning calorimetry (DSC), thermograms of samples filled with fillers were obtained.

Table 3. Thermodynamic properties of composite materials based on P-Y 342 polyethene with divalent metal phosphates

The component of the composition	Melting starts, T° C	Melting peaks, T°C	Enthalpy, ΔH, Дж/г	Crystalliniyαs, %
P-Y 342	125	134	182	62
P-Y 342/3% CuO - NH_4PO_3	136	147	199	68

P-Y 342/3% CoO - NH_4PO_3	137	149	193	66
P-Y 342/ 3%NiO - NH_4PO_3	139	153	190	65

Comparative analysis of the diffraction spectra of composite materials by types of fillers and polymers showed that the larger the interlayer distance of the modified fillers and the higher the filler concentration, the higher the diffusion rate of the filler included in the composition. Thus, the degree of dispersion of divalent metal phosphates in the polymer composition also depends on the duration of mixing of the components and the melt viscosity.

Accurate results can be obtained by experimentally studying the size and shape of particles using X-ray phase analysis (Debye-Scherer method). The size of the coherent distribution zones (CZD) (nanocrystal size) is determined by the Debye-Scherrer formula: $Dp = K \lambda / (B\cos \theta)$

Dp - Average crystal size (nm) K - Scherrer's constant. K varies from 0.68 to 2.08. For spherical crystals with cubic symmetry, $K = 0.94$ λ is the X-ray wavelength. Cu Ka = 1.54178 Å. B - integral length of reflections in the FWHM diffractometer (full width at half of the maximum). $\cos\theta$ is the cosine angle of X-ray diffraction.

Table 4. The results of calculating the size of nanoparticles of a composite based on PA-6 polyamide and divalent nickel phosphates according to the Debye-Scherrer formula

№	2theta- Scan angle	FWHM - integral reflex width	Dp (nm) average crystallite size	Dp (nm) average
1	8.2	0.478	17.42	15.61
2	8.7	0.57	14.61	
3	21.3030	0.5584	15.12	
4	24.0203	0.54	15.70	
5	26.2200	0.56	15.23	

Table 5. The results of calculating the size of nanoparticles of a composite based on polyethene P-Y 342 and phosphates of divalent nickel according to the Debye-Scherrer formula

№	2theta- Scan angle	FWHM - integral reflex width	Dp (nm) average crystallite size	Dp (nm) average
1	6.025	0.3461	24.03	23.53
2	8.5571	0.4642	17.94	
3	12.212	0.3481	23.99	
4	24.8731	0.2868	29.65	
5	26.459	0.3871	22.04	

According to the results of X-ray phase analysis, it was found that the particle size in the obtained composite materials is on the nanoscale.

Conclusion.

Thus, the maximum amount of divalent metal phosphates added as a filler to improve the physical and mechanical properties of polymeric composite materials based on polyamide and polyethene was 3%. All the results obtained showed that the addition of 3% divalent metal phosphates to polymers increases the strength and heat resistance of polymers.

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