
# INFLUENCE OF MINERAL FILLERS ON THE PHYSICAL AND MECHANICAL PROPERTIES OF COMPOSITE MATERIALS BASED ON POLYETHYLENE AND POLYAMIDE-66

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**ABSTRACT:** The article studied the physical and mechanical properties of filled polyethylene and polyamide compositions, the determination of the melt flow index by vizkizometry methods, the determination of bending strength by the methods of two-bearing bending, the determination of Charpy impact strength.

The rheological characteristics of composite materials based on polyethylene with mineral fillers are determined.

To obtain a composite material based on polyethylene, the content of fillers was changed from 30 mass to 50 mass parts. The data obtained show that the optimal compositions are those containing: 30 wt.h. mineral fillers.

**Key words:** polyethylene, polyamide-66, mineral fillers, atomic force microscopy, physicochemical and mechanical properties

**Introduction.** Today, the rapid growth of the world population and production volumes leads to a growing demand for polymer composite materials from year to year. In particular, the demand for polymer composite materials in industry and production is growing every day. In this regard, the automotive industry places high demands on the design of polymer materials. Currently, the basis of scientific research is the production of composite materials that meet a number of requirements, such as modification of polymers, improvement of their physical and mechanical properties, addition of additives without changing their composition [1;2].

In the work, basalt and vermiculite were used as mineral fillers in polymeric materials of various compositions (polyamide-66, polyethylene), which showed the promise of using some compounds of this class as flame retardants [3].

**Method and materials**. The object of research is thermoplastic composite materials based on polyethylene and polyamide. An estimate of the melt flow index (MFR), which is the selected temperature and viscosity of a medium molecular weight melt, is typicallyis a quantitative benchmark for pipe manufacturers. The fluidity of a polyethylene melt and composites based on it was determined in a capillary displacement viscometer under the action of a constant force type IIRT-M 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. The specimens were tested as 55.5 x 6 x 4 mm beams with a side spacing of 40 mm. The flexural strength was calculated using the following formula. σi \u003d 1.5 (Ra) / (bc2)

In this study, 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 - three times the 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 on each sample. To test frost resistance, the samples were kept at minus 30 °C for 100 days and then their impact resistance was measured using the Sharpe method. . The tensile strength and relative elongation of the sample were determined in accordance with GOST 11262-80 (ASTM D 638) [5]. These parameters determined on a cutting machine with a deformation rate of 50 mm / min on samples of a spatula shape measuring 50 x 6 x 4 mm. The tensile strength (σr) of the samples was determined by the following equation: σr = P/(a b), where P is the load; a, b - thickness and width of the narrowest part of the sample, mm. The 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).

**The discussion of the results.** In this study, an improvement in the physical and mechanical properties of polyethylene P-Y-342 and polyamide PA-66 filled with mineral fillers was observed. The aim of the work is to improve the physical and mechanical properties of polymers as a result of their addition of mineral fillers [6]. This also implies the development of methods for obtaining composite materials based on nanoscale modifiers that enhance the physical and mechanical properties of polymers and affect polymermacromolecules**.**

**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 polyethylene and polyamide with mineral fillers. Table 1 shows that the properties of nanocomposites based on polyethylene P-Y 342 and polyamide-PA-6 filled with mineral fillers have changed compared to the original polymers. The results of the analysis show that the inclusion of nanoparticles in the polymer improves the physical and mechanical properties of the polymers. [eight]By adding PEMA/TEAS/VC to polyethylene, it can be seen that the impact resistance increased from 50 to 68 kJ/m2 compared to the original polyethylene, the bending resistance increased from 24 to 38 MPa, and the tensile strength increased from 21 to 48 MPa. [9]. In the polyamide-based composite material, it can be observed that the impact resistance has increased from 120 to 130 kJ/m2 compared to the original polyethylene, the bending resistance has increased from 100 to 146 MPa, and the tensile strength has increased from 80 to 114 MPa. [ten].

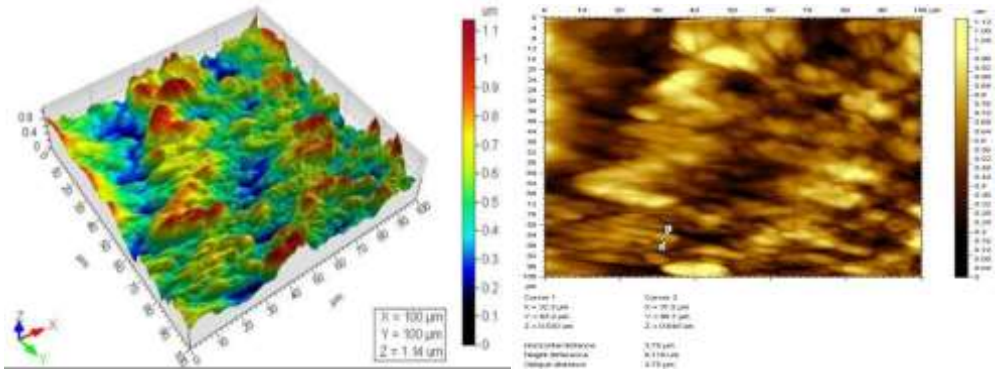
**Table 1. Comparative analysis of physical and mechanical properties of composite materials based on polyethylene P-Y 342 and polyamide PA-66 filled with mineral fillers (30%).**

| The composition of the composition | Impactstrength, kJ/m2 | σ bending, MPa | fracture σ, MPa | Elongation, % | Shrinkage, |
|---|---|---|---|---|---|
| | GOST 4647-80 | GOST 4648-80 | GOST 14236-81 | GOST 14236-81 | GOST 18599-21 |

| | | | | | |
|---|---|---|---|---|---|
| P-Y 342 | 50 | 24 | 21 | 750 | 3 |
| P-Y 342/VK | 56 | 35 | 33 | 174 | 2,7 |
| P-Y 342/TEAS/VK | 60 | 36 | 36 | 170 | 2,8 |
| P-Y 342/PEMA/TEAS/VK | 68 | 38 | 48 | 155 | 2,2 |
| PA-66 | 120 | 100 | 80 | 150 | 2,6 |
| PA-66 /BT | 125 | 118 | 92 | 114 | 2,8 |
| PA-66/TEAS/BT | 128 | 130 | 103 | 106 | 2,3 |
| PA-66/PEMA/TEAS/BT | 130 | 146 | 114 | 96 | 2,8 |

Thus, the addition of PEMA/TEAS to polyolefins resulted in high physical and mechanical 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 mineral fillers [11].

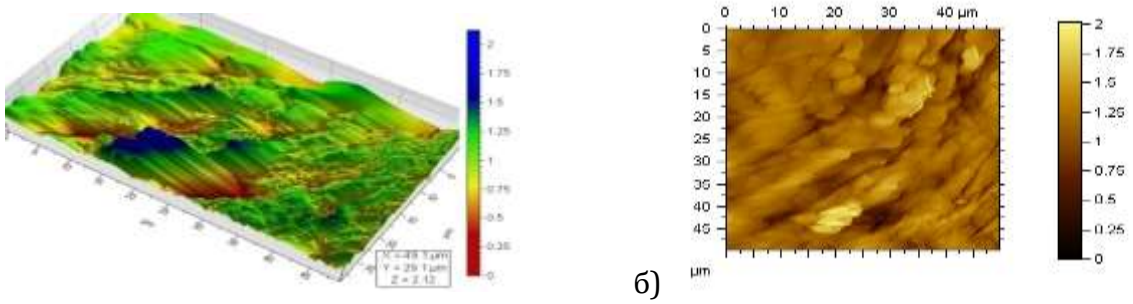
AFM (atomic force microscopy) is widely used to study the specifics of the microstructure and topography of various materials. This method is very sensitive to pixels and can form the surface of a sample taken in the nanoscale range on a three-dimensional surface. This method clearly shows the change in the surface, shape and size of the particles, as well as the mechanical properties of the surface of the material. [12]. Also, in this work, we studied the effect of modifying the particles of mineral fillers on the morphology of the polymer surface. Analysis and study of the surface of modified polyolefins show that the particles of mineral fillers are scattered among polymer macromolecules and interact. The results of AFM analysis of a composite material based on polyamide-66 and polyethylene with mineral fillers are obtained. The analysis was carried out on an AFM using silicon cantilevers with a turning radius of 10 nm [13].



а) б)

**Figure 1. AFM images of a composite material based on polyethylene with vermiculite a) - three-dimensional image, b) - two-dimensional image.**

The scanning area ranged from 1 to 50 μm. Microscopy was carried out in air by the semi-contact method; on the lines of registration of the change in the amplitude of oscillations of the counteraction axis, oscillations of the intermediate movement and the surface relief are reflected, which indicates the adhesion of individual surfaces to each other. On fig. 1 shows the surface of polyethylene modified with vermiculite. The results show that the roughness of pure polyethylene is 100 nm, and the surface roughness of alumina-modified polyethylene is 210 nm [14].




а) б)

**Figure 2. AFM images of a composite material based on polyamide-66 and basalt: a) - three-dimensional image, b) - two-dimensional image.**

On fig. 2 is an AFM image of the obtained composite material, according to which the roughness is 100 nm in polyamide-66 and 122 nm in a composite material based on PA-66 obtained from basalt.

In this work, on the basis of thermoanalytical studies, the thermal-oxidative properties of the obtained composite materials are studied. It is known that polymer composite materials filled with nanocomposites with particles of mineral fillers 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, thermal and thermophysical properties were studied: a change in the melting point, heat resistance.

The melting temperature and crystallization rate of polymer composite materials were determined by differential scanning calorimetry (DSC). The results obtained are presented in table 2.

**Table 2. Thermodynamic properties of composite materials based on polyamide PA-66 with basalt.**

| The composition of the composition | Start melting, then C | Meltingpeak, T°C | Enthalpy, ΔN, J/g | Degreeofcrystallinity α, % |
|---|---|---|---|---|
| PA-66 | 220 | 224 | 188 | 55 |
| PA-66 /BT | 236 | 241 | 197 | 61 |
| PA-66/TEAS/BT | 237 | 243 | 203 | 59 |
| PA-66/PEMA/TEAS/BT | 239 | 245 | 210 | 58 |

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

**Table 3. Thermodynamic properties of composite materials based on polyethylene P-Y 342 with vermiculite**

| The composition of the composition | Start melting, then C | Meltingpeak, T°C | Enthalpy, ΔN, J/g | Degreeofcrystallinity α, % |
|---|---|---|---|---|
| P-Y 342 | 125 | 134 | 182 | 62 |

| | | | | |
|---|---|---|---|---|
| P-Y 342/VK | 136 | 147 | 199 | 68 |
| P-Y 342/TEAS/VK | 137 | 149 | 193 | 66 |
| P-Y 342/PEMA/TEAS/VK | 139 | 153 | 190 | 65 |

A comparative analysis of the diffraction spectra of composite materials by types of fillers and polymers showed that the greater 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 mineral fillers in the polymer composition also depends on the duration of mixing of the components and the viscosity of the melt.

Accurate results can be obtained in an experimental study of the size and shape of particles using X-ray phase analysis methods (Debye-Scherer method). The size of coherent distribution zones (CZR) (the size of nanocrystals) 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 wavelength of X-rays. Cu Ka = 1.54178 Å.

B is the integral length of reflections in the FWHM diffractometer (full width at half maximum). cosθ is the cosine angle of X-ray diffraction.

**Table 4. Results of calculating the size of nanoparticles of a composite based on polyamide PA-66 and basalt using the Debye-Scherrer formula**

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

**Table 5. Results of calculating the size of nanoparticles of a composite based on polyethylene P-Y 342 and vermiculite using the Debye-Scherrer formula**

| № | | 2theta - ScanAngle | FWHM - integral width of reflections | Dp (nm) average crystallite size | Dp (nm) average |
|---|---|---|---|---|---|
| 1 | | 6.025 | 0.3461 | 24.03 | |
| 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 | 23.53 |

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

**Conclusions Thus,** the maximum amount of mineral fillers added as a filler to improve the physical and mechanical properties of polymer composite materials based on polyamide and polyethylene was 30%. All of the results obtained showed that the addition of 30% mineral fillers to the polymers increased the strength and thermal stability of the polymers.

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