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## ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

### **OBTAINING AND RESEARCH OF FIRE-RESISTANT COMPOSITE MATERIALS BASED ON A MIXTURE OF LOW AND HIGH DENSITY POLYETHYLENES, BENTONITE AND ALUMINUM HYDROXIDE**

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## INTRODUCTION

**Relevance and approbation of the topic.** With the improvement of technique and technology of industrial production rises more acute problem of developing high-quality plastic structural products that operate in more severe extreme conditions. Despite the fact that industrially produced polyolefins have a fairly good set of physicommechanical and technological characteristics, they cannot always meet the increased requirements of modern technology in terms of their quality characteristics. Considering that one of the most important requirements for polymer materials that can be used in construction, transport, electronics, aviation and space technology, automotive and shipbuilding is high fire safety, reducing the flammability of polyolefins, obtaining fire-resistant materials based on them is one of the urgent problems requiring constant attention and urgent solutions.

There is a search for environmentally friendly flame retardants with low levels of smoke and toxic combustion products that would reduce the flammability of polyolefins. In this regard, aluminum hydroxide and bentonite are among the most widely used flame retardant-fillers in recent years. A number of researchers and authors have provided some information on the preparation of fire retardant polymer composites based on various polymers belonging to the class of polyolefins and these flame retardant-fillers, as well as the effect of their size and quantity, their use in combination with other fire retardants on the properties of composites and the mechanism of action. Although the scientific literature contains the results of studies on the preparation and properties of composites based on these fillers and polymer blends, there are very few studies on the preparation and detailed study, comprehensive analysis of the structure and properties of fire-resistant polymer composites based on a polyethylene blend.

Availability of low-density polyethylene production in Azerbaijan at the Ethylene-Propylene Plant, high density polyethylene at the High Density Polyethylene Plant, bentonite clay deposits Dash Salakhli in Gazakh region and bauxite deposits in Nakhchivan makes

research work in the direction indicated in the dissertation even more relevant.

**Object and subject of research.** The object of research is composite materials based on a mixture of low and high density polyethylene and environmentally friendly flame retardants. The subject of this research is the study of the kinetic regularities of the crystallization process, rheological, physicochemical, thermomechanical properties and fire resistance of the obtained composite materials.

**The purpose and objectives of the research.** The aim of the study was to determine the influence of the type and amount of environmentally friendly flame retardants (aluminum hydroxide, bentonite) on fire resistance, physicochemical and rheological properties, kinetic regularity of the crystallization process of composite materials obtained on the basis of a mixture of low and high density polyethylene. To achieve this goal, the following issues have been resolved:

- obtaining compositions based on mixtures of low and high density polyethylene, studying the effect of the ratio of components on the physicochemical, thermomechanical, rheological properties and kinetic regularities of the crystallization of the polymer mixture;
- development of polymer composites based on mixtures of low and high density polyethylene and environmentally friendly flame retardant-fillers;
- research of physicochemical, rheological properties, kinetic regularities of the crystallization process, fire resistance of polymer composites based on a mixture of low and high density polyethylene, depending on the type and amount of filler;
- research of the technological mode of processing low-combustion polymer composites by extrusion.

**Research methods.** Research on the topic of the dissertation was carried out using modern methods of physical, physicochemical analysis: infrared spectroscopy (Agilent Cary 630 FTIR), rheology (CEAST MF50 capillary rheometer), differential thermal analysis and thermogravimetric analysis (derivatograph of the Paulik-Paulik-

Erdey system), dilatometry (dilatometer based on IIRT-1 device), thermomechanics (Kanavets device), Vicat temperature resistance, oxygen index (MODUL FTA instrument). To obtain nanosized fillers, an A-11 basic analytical mill was used, a Mastersizer 3000 laser analyzer was used to determine the particle size, and a tensile testing machine was used to determine the mechanical properties of polymer composites.

**Basic provisions for defense:**

1. Results of the study of the influence of the ratio of components on the physicomechanical, thermomechanical, rheological properties, kinetic regularities of the crystallization process of compositions based on a polyethylene mixture;
2. Results of the study of the effect of the type and amount of filler on fire resistance, physicomechanical, rheological properties and kinetic regularities of the crystallization process of composites based on a mixture of polyethylene.

**Scientific novelty of the research.** The scientific novelty of the research lies in the fact that for the first time: fire-resistant composites based on a mixture of low and high density polyethylene in a ratio of 50/50, bentonite and aluminum hydroxide have been obtained and their properties have been comprehensively studied; the inclusion of S-(carballyoxy)methyl-N,N-diethyldithiocarbamate plasticizer and DuPont<sup>TM</sup> Fusabond® P353 compatibilizer into the composite material based on a polyethylene mixture and aluminum hydroxide not only improved the physicomechanical properties, but also increased the fire resistance; the technological mode of processing the obtained polymer composite materials of low combustion by the method of extrusion has been investigated.

**Theoretical and practical significance of the research.** Fire-resistant composite materials based on a mixture of low and high density polyethylene, bentonite and aluminum hydroxide have been developed. The used flame retardant-fillers and their decomposition products during combustion are distinguished by a high level of environmental safety. Considering the presence in Azerbaijan of production of types of polyethylene used in the obtaining of

composites, as well as the fact that flame retardant-fillers are readily available (there are deposits in Azerbaijan) and are cheap, research in this area is of great practical importance. It is expected that these new slow burning polymeric composites based on blends of low and high density polyethylene will significantly expand the use of polyethylene in industry, and especially in construction.

The mode of processing technology in the extruder of composite materials obtained on the basis of a mixture of low and high density polyethylene and mineral fillers was studied and approved by the test report carried out by METAK LLC. The laboratory technological regulations "Obtaining slow burning composite materials based on a mixture of low and high density polyethylenes and aluminum hydroxide" were developed and approved.

**The author's personal involvement.** The main directions of dissertation work, conducting experiments, analyzing and summarizing the obtained results, writing scientific papers and dissertations were carried out directly by the applicant.

**Approbation and application of research.** 37 scientific papers have been published on the topic of the dissertation, of which 12 articles, 14 conference materials, 10 abstracts and 1 Azerbaijan patent. The main results of the dissertation were presented and discussed at the following international and republican scientific conferences: International Scientific Conference on "Actual Problems of Modern Natural and Economic Sciences" (Ganja, 2018), International scientific and practical conference "Innovative perspectives of development of oil refining and petrochemistry" dedicated to the 110th anniversary of Academician V.S. Alieva (Baku, 2018), International Scientific Conference on "Actual problems and solutions of construction materials production areas" dedicated to the memory of Professor Sardarov B.S. (Baku, 2018), I Republican Scientific Conference of Students dedicated to the 96th anniversary of national leader Heydar Aliyev (Baku, 2019), Republican scientific-practical conference on "Ways of application of scientific innovations in the educational process" dedicated to the 96th birthday of national leader Heydar Aliyev (Lankaran, 2019),

International Scientific Conference on "Actual Problems of Modern Chemistry" dedicated to the 90<sup>th</sup> anniversary of the Institute of Petrochemical Processes named after Academician Y.H. Mammadaliyev of ANAS (Baku, 2019), International scientific conference on "Prospects for innovative development of chemical technology and engineering" (Sumgayit, 2019), V International scientific conference "Material science. Nonequilibrium phase transformations" (Varna, 2019), XVI International Scientific Conference of Young Scientists "Youth in Science - 2019" (Minsk, 2019), I International scientific-practical conference "Azerbaijan and Turkey University: education, science, technology" (Baku, 2019), International Scientific and Technical Conference of Young Scientists "Innovative Materials and Technologies-2020" (Minsk, 2020), Second International Scientific Conference of Young Scientists on "Multidisciplinary approaches in solving modern problems of fundamental and applied sciences (Natural sciences)" (Baku, 2020), I International Scientific Conference of Students and Young Researchers on "Sustainable Development in Chemistry and Chemical Engineering" dedicated to the 97<sup>th</sup> anniversary of national leader Heydar Aliyev (Baku, 2020), XVII International Scientific Conference of Young Scientists "Youth in Science 2.0'20" (Minsk, 2020), All-Russian Scientific and Practical Conference "Advanced Technologies and Materials" (Sevastopol, 2020), II scientific-technical conference "Materials with specified properties in the transition to a new technological structure: chemical technologies" (Moscow, 2020), V All-Russian Conference "Chemistry and Chemical Technology: Achievements and Prospects" (Kemerova, 2020), International Scientific and Practical Conference "Young Researchers named after D.I. Mendeleev" (Tyumen, 2020), International Scientific and Technical Conference of Young Scientists "Innovative Materials and Technologies" IMT 2021 (Minsk, 2021), "Kirpichnikov Readings - XV International Conference of Young Scientists, Students and Postgraduates" Synthesis and Study of Properties, Modification and Processing of High-Molecular Compounds" (Kazan, 2021), II International

Scientific Conference of Students and Young Researchers on "Sustainable Development in Chemistry and Chemical Engineering" dedicated to the 98<sup>th</sup> anniversary of national leader Heydar Aliyev (Baku, 2021).

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

**The volume and structure of the work.** The thesis consists of an introduction, 6 chapters, results, bibliography and applications. The dissertation materials are reflected on 202 computer pages, including 48 figures, 17 tables, 185 references. The total volume of the thesis is 239930 symbols: introduction 13219, I chapter 66134, II chapter 28288, III chapter 40035, IV chapter 55328, V chapter 24109, VI chapter 9366, results 3451 symbols.

**In the introductory part,** the relevance of the dissertation work is substantiated, the purpose of the work, the main research issues, scientific novelty, theoretical and practical significance of the work are given.

**The first chapter** provides a detailed analysis of the current state of this problem based on the available literature on the production of fire-resistant composite materials based on polyethylene and polypropylene, aluminum hydroxide, bentonite and montmorillonite, and the study of the properties of the composites obtained.

**The second chapter** contains the physical characteristics of the primary raw materials and substances used in the research, the method of obtaining polymer composite materials, methods of researching various properties and devices.

**In the third chapter,** the influence of the ratio of the components of a mixture of low and high density polyethylene on the physicomechanical, thermomechanical properties, the regularity of the crystallization process of composites based on them, a comparative analysis of the rheological properties of low and high density polyethylene, their mixture in a 50/50 ratio, discussion of the results of the influence modifying agents on the physicomechanical properties of compositions based on a polyethylene mixture is given.



**The fourth chapter** presents a discussion of the results of the study of physicomachanical, thermomechanical, rheological properties, kinetic regularities of the crystallization process of polymer composites based on a mixture of low and high density polyethylene, aluminum hydroxide, bentonite and montmorillonite.

**The fifth chapter** discusses the results of studies of the effect of modifiers on the properties of polymer composite materials based on mixtures of low and high density polyethylene and aluminum hydroxide.

**The sixth chapter** describes the features of the technological mode of processing composites based on mixtures of low and high density polyethylene, mineral fillers by extrusion.

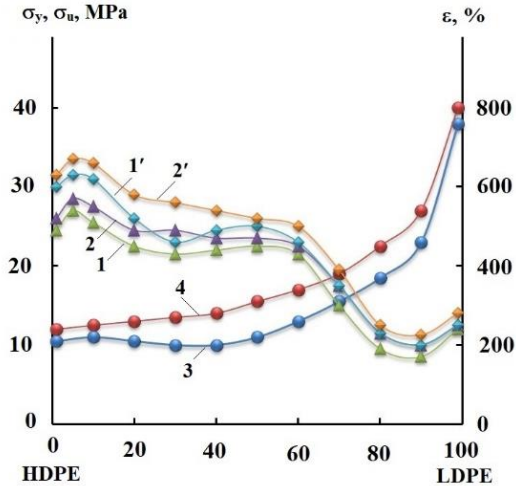
At the end of the dissertation, the results obtained on the basis of research, a list of used literature, a test report, the first page of the laboratory technological regulations, confirmed by seals, a list of abbreviations and designations are presented.

## **MAIN CONTENT OF THE WORK**

### **PROPERTIES OF COMPOSITIONS BASED ON A MIXTURE OF LOW AND HIGH DENSITY POLYETHYLENE**

The effect of the ratios of the components of compositions based on low density polyethylene (LDPE) and high density polyethylene (HDPE) on the change in the properties of the mixture was studied.

**Physicomachanical properties and kinetic regularities of the crystallization process of composite materials obtained on the basis of mixtures of high and low density polyethylene.** When studying the effect of the HDPE/LDPE ratio on the physicomachanical properties of the HDPE + LDPE mixture, it was found that as the concentration of LDPE in the mixture increases, a regular decrease in strength properties is observed (figure 1). The presence of a slight increase in strength in samples with a HDPE/LDPE ratio of 40-60/60-40 can be associated with phase inversion, i.e., when the dispersed phase becomes a dispersed medium or vice versa. It is not excluded that, with this ratio, a special mechanism of crystallization of the polymer mixture is possible, in which the growth of crystalline formations promotes the formation of



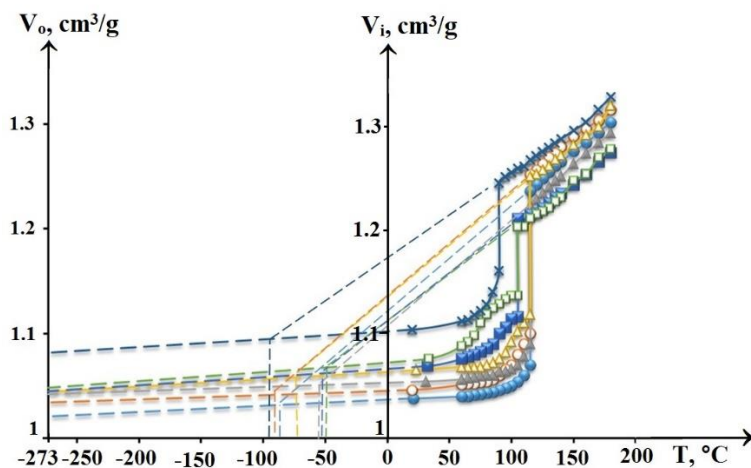
**Figure 1.** Effect of HDPE / LDPE ratio on ultimate tensile stress ( $\sigma_u$ ) (1,2), tensile yield strength ( $\sigma_y$ ) (1', 2') and elongation at break (3,4) of composite materials based on the initial mixture of HDPE + LDPE (1 , 1', 3) and its modified TiO<sub>2</sub> compositions (2, 2', 4).

a supramolecular structure with a minimum content of defects in it. Apparently, it will be appropriate to assert about the "concentration" compatibility of the components being mixed at certain ratios. This is confirmed by the results of a study of the elongation at break of composite materials.

The results of dilatometric studies of the crystallization process confirm concept of phase inversion at 50 wt % LDPE content in the composition (table 1). When analyzing dilatometric dependences, one can see that the curves of mixtures are located between the curves of the initial components (figure 2). It can be assumed that, during the cooling of the samples, the crystallization of HDPE macromolecules and the growth of spherulite formations are accompanied by the displacement of relatively branched LDPE macromolecules into amorphous regions. Apparently, at low LDPE concentrations (up to 20 wt %), the crystallization of HDPE macromolecules is accompanied by the formation and growth of intrinsic spherulite formations, which facilitate the displacement of

**Table 1.** Influence of the LDPE / HDPE components ratio on their density and the onset temperature of the crystallization process

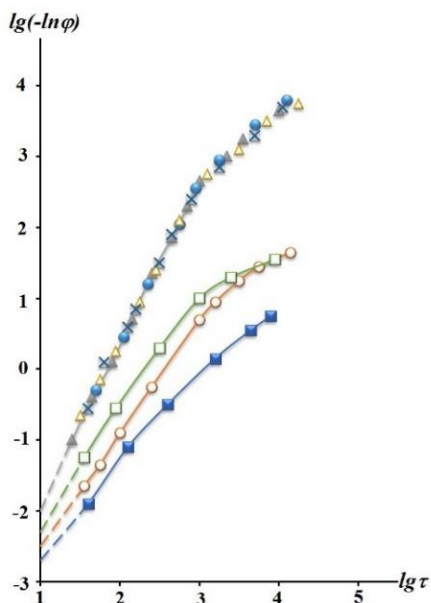
№	Formulation of the composition, wt. %	The onset temperature of crystallization, °C	Density, kg / m <sup>3</sup>
1	HDPE	114	963
2	10LDPE +90HDPE	114	960
3	20LDPE +80HDPE	114	948
4	30LDPE +70HDPE	114	948
5	40LDPE +60HDPE	114	943
6	50LDPE +50HDPE	114	941
7	60LDPE +40HDPE	110	938
8	70LDPE +30HDPE	110	935
9	80LDPE +20HDPE	105	935
10	90LDPE +10HDPE	102	929
11	LDPE	90	912



**Figure 2.** Kinetic regularities of crystallization of the initial LDPE, HDPE and their mixtures at various ratios: ●– 100 wt. % HDPE, ○– 10 wt. % LDPE + 90 wt. % HDPE, ▲– 20 wt. % macc. LDPE + 80 wt. % HDPE, △– 50 wt. % LDPE + 50 wt. % HDPE, ■– 80 wt. % LDPE + 20 wt. % HDPE, □– 90 wt. % LDPE + 10 wt. % HDPE, ×– 100 wt. % LDPE

LDPE macromolecules into amorphous regions. The further process of crystallization of LDPE macromolecules in the constrained conditions of the amorphous regions of HDPE becomes excessively difficult. That is why the process of crystallization of compositions up to 50 wt % of LDPE content occurs at the same temperature close to the crystallization temperature of HDPE, i.e. 114°C.

Figure 3, in Avrami coordinates, shows the effect of the HDPE / LDPE ratio on the mechanism of their crystallization. The initial HDPE and its compositions containing up to 50 wt % LDPE at the crystallization temperature are characterized by a constant “n” in the range of 2.12 - 2.31, while for the samples of the initial LDPE and its compositions up to 20 wt % the content of HDPE, the value of this



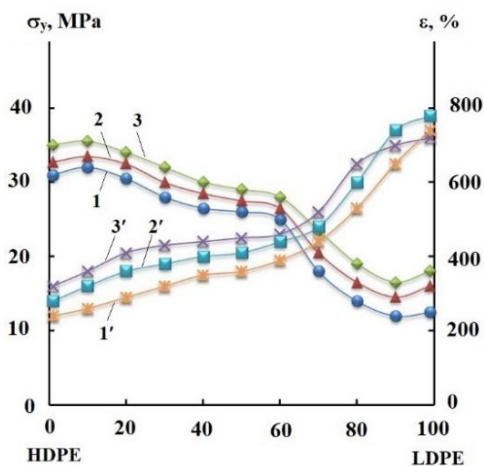
**Figure 3.** Effect of HDPE / LDPE ratio on the mechanism of their crystallization: ●– 100 wt. % HDPE, ○– 10 wt. % LDPE + 90 wt. % HDPE, ▲– 20 wt. % LDPE + 80 wt. % HDPE, △– 50 wt. % LDPE+ 50 wt. % HDPE, ■– 80 wt. % LDPE + 20 wt. % HDPE, □– 90 wt. % LDPE + 10 wt. % HDPE, ×– 100 wt. % LDPE

indicator fluctuates in the range of 1.38-1.57. With an increase in the concentration of LDPE in the composition of the HDPE + LDPE mixture, the lamellar two-dimensional growth of crystals is replaced by a linear type of growth of crystalline formations.

**Physicomechanical properties of modified compositions based on mixtures of low and high density polyethylene.** The effect of TiO<sub>2</sub> structurant (1.0 wt %) on the physicomechanical properties of the HDPE / LDPE polymer mixture was studied. The loading of TiO<sub>2</sub> contributes to a noticeable improvement in the strength properties and elongation at break of polymer mixtures (figure 1). Such a noticeable increase in these indicators unambiguously testifies to the fact that the loading of a TiO<sub>2</sub>, promotes the formation of heterogeneous nucleation centers. The simultaneous growth of crystalline formations during cooling on homogeneous and heterogeneous nucleation centers contributes to the formation of small-spherulite formations, which, as a rule, contribute to an increase in the strength characteristics of composite materials.

Figure 4 shows the curves of the dependence of the tensile yield stress and elongation at break of polymer mixtures on the sulfur concentration. The initial object of the study was a mixture of HDPE + LDPE + 1.0 wt % TiO<sub>2</sub>. The sulfur concentration was varied in the range of 1-5 wt %. At all ratios of the mixture components, the maximum strength is practically not observed. The latter circumstance is associated with the fact that when sulfur vulcanization is carried out in the melt, the process of chemical crosslinking proceeds uniformly throughout the entire volume of the samples. Such thermophysical and technological properties of samples as heat resistance, melting point and melt flow index (MFI) were investigated (table 2). Sulfur vulcanization proceeds at the site of rupture of a relatively small number of double bonds, and therefore contributes to the formation of a sparsely reticulated spatial structure. This is confirmed by the results of the study of the MFI of composite materials. As the sulfur concentration increases, a regular decrease in the MFI of the samples is observed. At the same time, the MFR values of vulcanized polymer mixtures remain at a level

sufficient for their processing by injection molding and extrusion methods and for obtaining structural products with a given structure and properties.



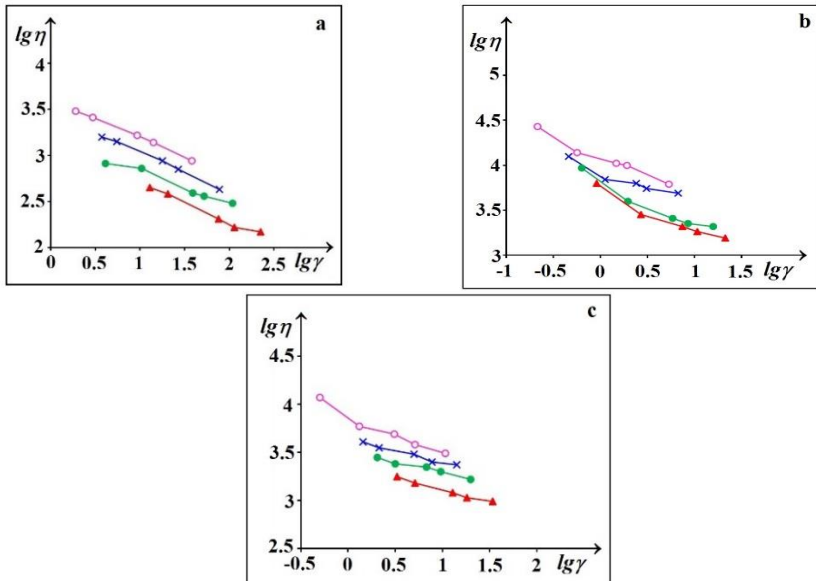
**Figure 4.** Influence of HDPE / LDPE ratio and sulfur concentration on tensile yield strength (1, 2, 3) and elongation at break (1', 2', 3') of vulcanized compositions: 1- 1.0; 2- 3.0; 3- 5.0 wt. %

**Table 2.** Influence of sulfur concentration on thermophysical and technological parameters of composite materials based on HDPE + LDPE mixtures.

№	Formulation of the composition, wt. %	Vicat heat resistance, °C	Melting point, °C	MFI, g/10 min.
1	HDPE+20LDPE+1.0TiO <sub>2</sub>	132	139	5.6
2	HDPE +20LDPE +1.0TiO <sub>2</sub> +1.0 S	132	139	4.1
3	HDPE +20LDPE +1.0TiO <sub>2</sub> +3.0S	134	139	2.5
4	HDPE +20LDPE +1.0TiO <sub>2</sub> +5.0S	135	140	1.3
5	HDPE +50LDPE +1.0TiO <sub>2</sub>	119	132	4.9
6	HDPE +50LDPE +1.0TiO <sub>2</sub> +1.0 S	120	132	3.8
7	HDPE +50LDPE +1.0TiO <sub>2</sub> +3.0S	122	132	2.3
8	HDPE +50LDPE +1.0TiO <sub>2</sub> +5.0S	123	133	0.9
9	HDPE +80LDPE +1.0TiO <sub>2</sub>	105	118	4.2
10	HDPE +80LDPE +1.0TiO <sub>2</sub> +1.0 S	106	118	2.9
11	HDPE +80LDPE +1.0TiO <sub>2</sub> +3.0S	107	118	1.6
12	HDPE +80LDPE +1.0TiO <sub>2</sub> +5.0S	109	118	0.5

**Thermomechanical properties of composite materials based on mixtures of low and high density polyethylene.** Studies at the Kanavets device showed that the thermomechanical curves of polymer mixtures are located between the initial curves of HDPE and LDPE. As the concentration of LDPE in the HDPE + LDPE mixture increases, a regular decrease in the softening temperature and the temperature of transition to the viscous-flow state is observed.

**Rheological properties of polymer mixtures based on low and high density polyethylene.** Rheological studies of initial polyethylenes and their HDPE / LDPE (50/50) mixture were carried out on a CEAST MF50 capillary rheometer (INSTRON, Italy) in a temperature range of 190-250°C and in a load range of 3.8-21.6 kg. An analysis of the curves of the dependence of the effective viscosity on the shear rate showed that it occupies an intermediate position in comparison with the viscosity of the original polyethylenes (figure 5). The latter circumstance testifies to the technological compatibility



**Figure 5.** The dependence of the melt viscosity of a) LDPE, b) HDPE and c) polymer blend of LDPE / HDPE on shear rate at various temperatures: ○-190°C; ×-210°C; ●-230°; ▲-250°C

of the initial components of the mixture (HDPE with LDPE) and their uniform mutual dispersion.

## **SELECTIVE EFFECT OF MINERAL FILLER ON PROPERTIES OF COMPOSITES OBTAINED ON THE BASIS OF A MIXTURE OF LOW AND HIGH DENSITY POLYETHYLENES**

The effect of the concentration and type of fire retardant on the structure and properties of composites based on a mixture of HDPE / LDPE in a 50/50 ratio was studied.

**Physicomechanical properties, kinetic regularities of the crystallization process and fire resistance of nanocomposites based on a mixture of low and high density polyethylene and bentonite.** Nanocomposites based on HDPE / LDPE (HDPE-ultimate tensile stress ( $\sigma_u$ ) - 28.4 MPa, elongation at break ( $\epsilon$ ) - 350 %, MFI (190°C, 5 kg) (J) - 1.7 g/10min, LDPE-  $\sigma_u$ =8.7 MPa,  $\epsilon$ =580%, J=7.9 g/10min (190°C, 5 kg) ) and bentonite nanoparticles 80-120 nm in size, taken in an amount of 1, 3, 5, 10, 20, 30 wt. %, have been obtained and researched. Table 3 shows the effect of the concentration of bentonite on the physicomechanical properties of the HDPE / LDPE + bentonite system. The increase in tensile strength in relatively large amounts of bentonite indicates that bentonite plays a reinforcing role as a filler. A decrease in the elongation at break of composites with an increase in the amount of bentonite is associated with the low compatibility of polyethylene with clay fillers.

Filler particles accumulating in amorphous areas increase the density of the entire composition. In this case, in a certain way, there is a decrease in the mobility of the "through" chains, which directly affects the increase in heat resistance and softening temperature of polymeric materials.

According to differential thermal analysis (DTA) data, the melting point of the initial HDPE / LDPE and its composites containing 1, 3, 5, 10, 20, 30 wt. % bentonite, changed in the following order: 120,



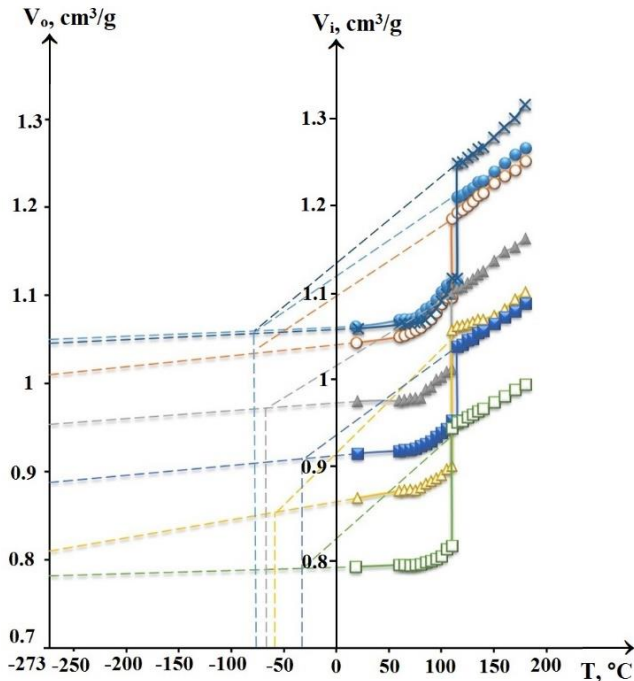
**Table 3.** Effect of bentonite concentration on the properties of nanocomposites based on a mixture of high and low density polyethylene (*B-bentonite*)

№	Formulation of the composition, wt. %	Tensile yield strength, MPa	Ultimate tensile stress MPa	Elongation at break, %	Vicat heat resistance, °C
1	HDPE/LDPE	20.7	14.5	480	112
2	HDPE/LDPE+1 B	21.6	15.5	450	112
3	HDPE/LDPE+3 B	19.8	13.0	370	114
4	HDPE/LDPE+5 B	19.3	12.8	65	114
5	HDPE/LDPE+10 B	19.4	12.7	40	115
6	HDPE/LDPE+20 B	19.2	16.3	30	117
7	HDPE/LDPE+30 B	18.2	15.7	30	118

120, 120, 121, 123, 125 and 125°C. As can be seen from the above data, at a bentonite concentration above 10 wt. %, a slight increase in the melting point of composite materials is observed, i.e. more thermal energy is required for thermal-fluctuation destruction of the supramolecular structure of composites.

As the amount of bentonite increases, the specific volume of composites, that is, the distance between macromolecular packages, decreases, and the density increases accordingly (figure 6). For composites with bentonite content in the range of 3-30 wt. %, the phase transition of the first type occurred at a temperature of 110-113°C. Approximate values of the glass transition temperature of the studied nanocomposite materials were found by the method of intersecting the upper and lower branches of the dilatometric curves. The glass transition temperature of the initial HDPE / LDPE and its mixtures with 1, 3, 5, 10, 20, 30 wt. % filler corresponds to -73, -73, -73, -65, -58, -34, -34 °C.

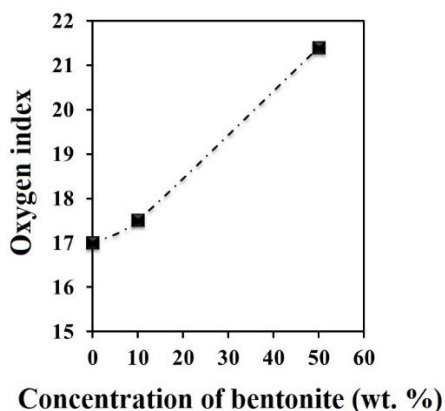
The free specific volume for the samples was calculated. The loading of bentonite into the composition of the polymer mixture helps to reduce the free specific volume. This fact makes it possible to assert that the filler particles are predominantly incorporated into the free volume of polymers.



**Figure 6.** Influence of bentonite concentration (wt. %) on the regularity of changes in specific volume from temperature for nanocomposites based on HDPE / LDPE + bentonite: ×– initial HDPE/LDPE mixture, ●– 1% bentonite, ○– 3% bentonite, ▲– 5% bentonite, Δ– 10% bentonite, ■– 20% bentonite, □– 30% bentonite

Thermomechanical studies have shown that composites based on HDPE / LDPE + bentonite are characterized by a solid and viscous state. As the amount of filler increases, the softening point of the composites and the transition temperature to the viscous state increase. Apparently, in composites with a relatively high concentration of bentonite, filler particles, accumulating in the interspherulite region, contribute to the limitation of the segmental mobility of the "through-chain", which ultimately shifts the softening temperature to the region of high temperatures.

In order to study the effect of the concentration of bentonite on the flammability of the LDPE / HDPE mixture, the method for determining the oxygen index (OI) was used. The OI increased as the amount of bentonite in the LDPE / HDPE + bentonite composite increased (figure 7). The maximum OI value (21.4) was obtained with a 50 wt. % bentonite content in the composite. The decrease in the flammability of the composite can be explained by the appearance of a clay-enriched protective char on the surface of the burning sample. In this case, the char acts as a protective barrier for the polymer matrix, preventing it from further combustion.

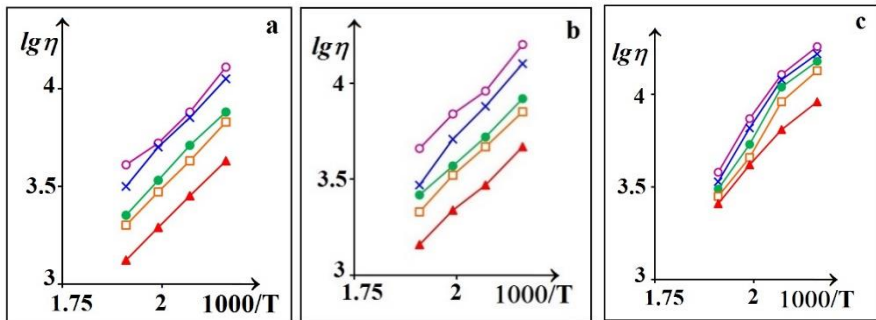


**Figure 7.** Dependence of the oxygen index on the concentration of bentonite (wt%) in the composites of LDPE / HDPE + bentonite

The synergistic effect of bentonite and aluminum hydroxide (AH) on the flammability of a HDPE / LDPE mixture was studied and compared with similar systems HDPE / LDPE + bentonite and HDPE / LDPE + AH. The total filler concentration was maintained at 10 wt. %. The results obtained on the basis of OI showed that the addition of 5 wt. % bentonite and 5 wt. % AH to the HDPE / LDPE mixture practically did not change its flammability and was equal to 17.1. In a comparative analysis of the AH of composites HDPE / LDPE + 10 wt. % bentonite, HDPE / LDPE + 10 wt. % AH and HDPE / LDPE + 5 wt. % bentonite + 5 wt. % AH, it was found that

the highest value (17.9) was obtained for composites containing AH. This result can be explained by the different mechanisms of action of the fillers.

**Rheological properties of nanocomposites based on a mixture of low and high density polyethylene and bentonite.** The effect of bentonite concentration, temperature and shear stress on the regularity of changes in effective viscosity and shear rate has been established. The dependence of the viscosity on the inverse of the absolute temperature in Arrhenius coordinates (figure 8) was determined, according to which the calculated “apparent” activation energy of the viscous flow of composites containing 1, 5, and 30 wt%. bentonite changes, respectively, in the range 14.4-27.2 kJ / mol, 17.8-29.4 kJ / mol and 17.2-39.2 kJ / mol.



**Figure 8.** Viscosity versus temperature in Arrhenius coordinates for polymer composites HDPE / LDPE +1 wt. % bentonite (a), HDPE / LDPE +5 wt. % bentonite (b), HDPE / LDPE +30 wt. % bentonite (c) at various loads (○–3.8 kg, ×–5.0 kg, ●–10 kg, □– 12.5 kg, ▲–21.6 kg).

**Physicomechanical and thermomechanical properties of nanocomposites based on montmorillonite, low density polyethylene and high density polyethylene.** Nanocomposites based on HDPE and LDPE were obtained, containing 1, 5, 10, 20, 30 wt. % montmorillonite (MMT). For HDPE-based nanocomposites, the maximum ultimate tensile stress is achieved at 10 wt % MMT

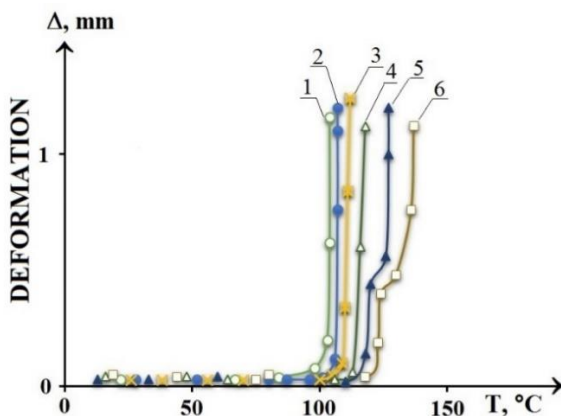
content (table 4). For nanocomposites based on LDPE, with an increase in the MMT concentration from 0 to 30 wt %, the ultimate tensile stress constantly increases. Apparently, here it affects the effect of the initial degree of crystallinity of the polymer matrix.

**Table 4.** Physicomechanical properties of nanocomposites based on montmorillonite, HDPE and LDPE.

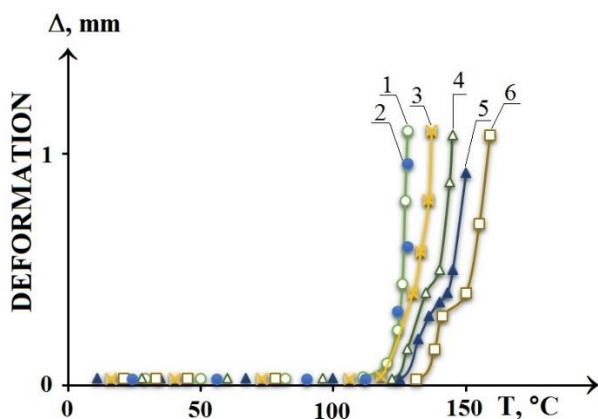
№	PE + MMT, wt %	Ultimate tensile stress, MPa	Elongation at break, %	Flexural modulus MPa	Heat resistance, °C	MFR, g/10min
1	HDPE	31.3	435	753	119	5.6
2	HDPE+1	32.2	480	835	119	6.0
3	HDPE+5	35.8	285	972	121	7.4
4	HDPE+10	36.0	115	1044	123	9.8
5	HDPE+20	28.5	35	1110	125	9.1
6	HDPE+30	24.6	20	1250	125	7.2
7	LDPE	13.1	720	196	85	1.3
8	LDPE+1	13.9	720	205	85	2.0
9	LDPE+5	14.7	440	222	87	4.2
10	LDPE+10	15.3	295	255	88	5.8
11	LDPE+20	16.5	105	317	91	5.2
12	LDPE+30	16.9	65	428	95	3.7

The amorphous region is represented by defects in crystal structures with a disordered arrangement of "through chains", which take part in the formation of several spherulite formations at the same time. And, the greater the degree of crystallinity of the initial polymer matrix, the less foreign mineral particles are required to fill the amorphous region. Accumulating in the amorphous region, nanoparticles impair the conformational mobility of the running chains until they are completely blocked. This, in fact, explains that the loading of a mineral filler primarily contributes to the deterioration of the elongation at break of nanocomposites.

Based on a comparative analysis of the curves presented in figures 9 and 10, it was determined that the softening temperature and the temperature of the beginning of the transition to the viscous-flow state of nanocomposites increase with an increase in the concentration of MMT in the composition of LDPE and HDPE.



**Figure 9.** Influence of MMT concentration (wt. %) on the character of changes in thermomechanical curves of compositions based on LDPE + MMT: ○ - initial LDPE; ● - 1% MMT; × - 5% MMT; Δ - 10% MMT; ▲ - 20% MMT; □ - 30% MMT



**Figure 10.** Influence of MMT concentration (wt. %) on the character of changes in the thermomechanical curves of compositions based on HDPE + MMT: ○ - initial HDPE; ● - 1% MMT; × - 5% MMT; Δ - 10% MMT; ▲ - 20% MMT; □ - 30% MMT

Nanoparticles contribute to the formation of a hardening phase that acts as mesh nodes that prevent the free movement of segments

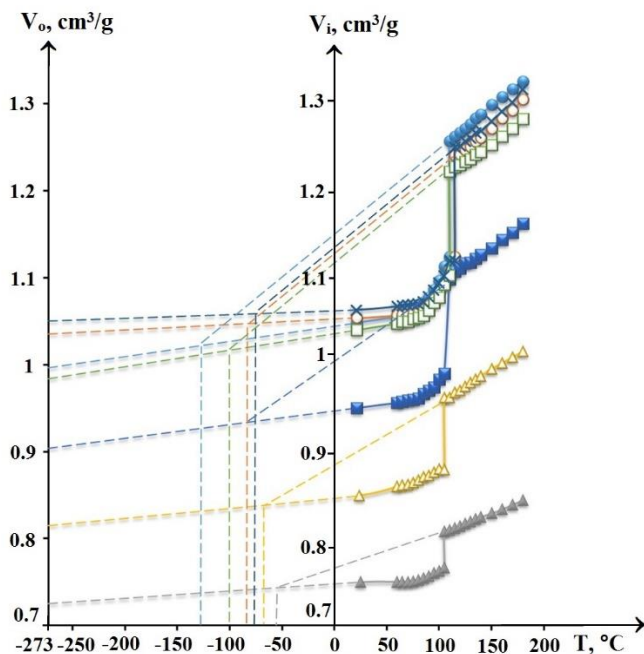
of macromolecules, which is accompanied by a decrease in the deformability of the composite and a corresponding increase in their softening temperature, and hence the heat resistance of the test samples.

**Physicomechanical properties and kinetic regularities of the crystallization process of composites based on a mixture of low and high density polyethylene and aluminum hydroxide.**

Composites based on a mixture of HDPE / LDPE (HDPE-  $\sigma_u=29.96$  MPa,  $\epsilon=50$  %,  $J=16.9$  g/10min (190°C, 5 kg), LDPE-  $\sigma_u=15.45$  MPa,  $\epsilon=820\%$ ,  $J=8.9$  g/10min (190°C, 5 kg)) and 1, 3, 5, 10, 30, 50 wt. % AH have been obtained and researched. Analysis of dilatometric curves shows that with an increase in the concentration of AH, the specific volume of composite materials decreases, that is, compaction of the material is observed (figure 11). If we compare the maximum and minimum specific volume of the samples under study at the phase transition temperature (figure 11), it can be seen that with an increase in the concentration of AH in composites, the difference between these values first increases, and for samples containing 5-50 wt. % AH, a decrease in this value is observed. The data obtained show that at low concentrations of AH, it contributes to the complete course of the crystallization process at concentrations of 5-50 wt. %, on the contrary, the crystallization process in filled composites is somewhat hampered.

The onset temperature of the composites crystallization in the amount of 3-10 wt. % AH is 110°C, and for composites containing 30 and 50 wt. % AH is 105°C.

The glass transition temperature of the initial HDPE / LDPE and composites based on it containing 1, 3, 5, 10, 30, 50 wt. % AH, respectively, is -73, -84, -128, -100, -84, -67, -57°C. All these data allow us to assert that at low concentrations of AH, the latter in the melt of the material exhibit the properties of nucleating agents, thereby contributing to the production of samples with a fine-spherulite structure, which favorably affects the improvement of



**Figure 11.** Influence of the concentration of AH (wt. %) on the regularity of the change in the specific volume from temperature for composites based on HDPE / LDPE + AH:  $\times$ – initial HDPE/LDPE,  $\circ$ – 1 % AH,  $\bullet$ – 3 % AH,  $\square$ – 5 % AH,  $\blacksquare$ – 10 % AH,  $\triangle$ –30 % AH,  $\blacktriangle$ –50 % AH

properties. At a higher concentration, the filler particles accumulate in the amorphous regions, hindering the conformational mobility of the "through" chains, increasing the brittleness of the material and, accordingly, deteriorating the strength and glass transition temperature of composite materials.

According to DTA data, the melting point of the initial HDPE / LDPE mixture and composites based on it containing 1, 3, 5, 10, 30, 50 wt. % AH changed in the following sequence: 120, 120, 120, 120, 125, 125, 125°C. In this case, a decrease in the conformational



mobility in relatively highly filled composites affects a certain increase in their melting point.

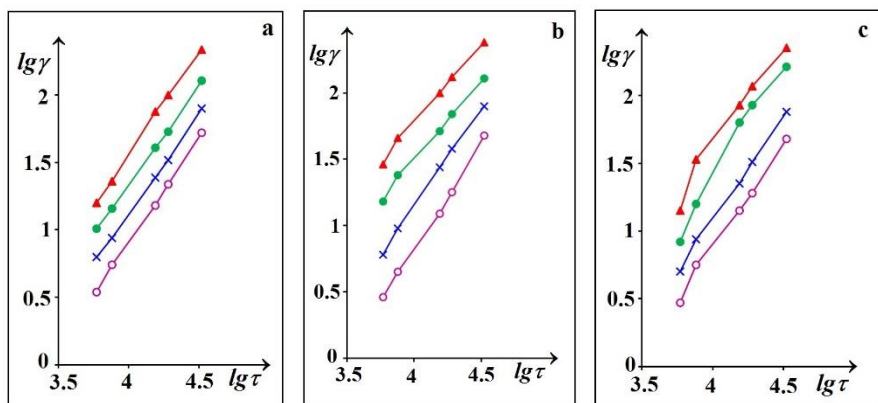
Comparative analysis of the curves of dependences shows that the value of free specific volume for composites filled with AH is higher than for the initial HDPE / LDPE mixture. This can be explained by the fact that AH particles can create steric obstacles for the complete crystallization of macromolecules in the phase transition region, thereby facilitating the loosening of the material and a decrease in their density. Due to the incompatibility of the polymer mixture of polyethylene with AH, their contact in the interfacial layer becomes weak. AH, lowering the packing density of macromolecules, promotes loosening of its structure. This can also be attributed to a certain decrease in the ultimate tensile stress of composites at a relatively high content of AH, the results of which are summarized in table 5.

**Table 5.** Influence of AH concentration on the physicomechanical properties of composites based on a mixture of high and low density polyethylene

№	Formulation of the composition, wt. %	Ultimate tensile stress, MPa	Elongation at break, %	MFR, g / 10min.
1	HDPE / LDPE	22.2	56	11.784
2	HDPE / LDPE +1 AH	20.0	37	11.300
3	HDPE / LDPE +3 AH	20.1	36	10.387
4	HDPE / LDPE +5 AH	18.5	36	10.222
5	HDPE / LDPE +10 AH	18.7	28	11.733

**Rheological properties of composites based on a mixture of low and high density polyethylene and aluminum hydroxide.** The influence of the concentration of AH, temperature and shear stress on the regularity of changes in the effective viscosity and shear rate of composites based on 1, 3 and 5 wt. % AH and a HDPE / LDPE mixture was studied. As can be seen from figure 12, at low temperatures, the flow curves are linear, and at relatively high

temperatures, the linear dependence of the shear rate on the shear stress is violated. At 1 wt. % loading of the polymer blend of HDPE / LDPE with AH, it can be said that the melt flow is close to the Newtonian regularity. However, with an increase in the concentration of the filler by 3-5 wt. %, the Newtonian regularity of the flow is violated. An analysis of the flow curves showed that with an increase in temperature and shear stress, a uniform increase in the shear rate of composites is observed, and this is primarily due to the stability and regulating ability of heterogeneous nucleation centers in the melt. In this case, the process of thermal fluctuation decomposition and restoration of agglomerates is controlled by heterogeneous centers.



**Figure 12.** Shear rate versus shear stress a) HDPE / LDPE + 1 wt. % AH; b) HDPE / LDPE + 3 wt. % AH; c) HDPE / LDPE + 5 wt. % AH at different temperatures: ○-190°C; ×-210°C; ●-230°; ▲-250°C

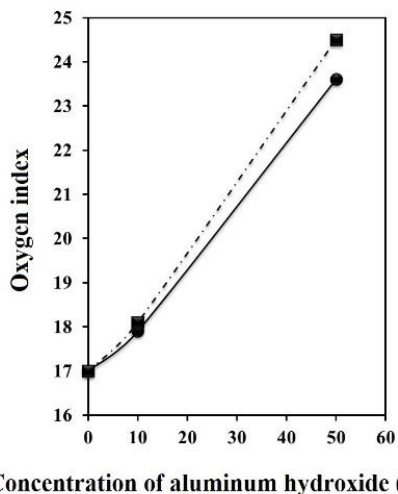
### INFLUENCE OF A MODIFIER ON THE PROPERTIES OF COMPOSITES BASED ON A MIXTURE OF LOW AND HIGH DENSITY POLYETHYLENE AND ALUMINUM HYDROXIDE

The influence of various modifiers on the properties of composite materials based on HDPE / LDPE mixture and AH has been studied.

**Influence of a plasticizer on the properties of composites based on a mixture of low and high density polyethylene and aluminum hydroxide.** Plasticizer S- (carballyoxy) methyl-N, N-diethyldithiocarbamate was used as a modifier. The amount of the modifier was 1, 3, 5 wt. %. The loading of a modifying additive contributes to some improvement in physicomechanical parameters, which clearly indicates the plasticizing role of the additive with a simultaneous increase in the compatibility of the reacting components. In all likelihood, when a polar modifier is loaded into the melt, the latter envelops the AH particles, as a result of which a thin layer of the modifier is formed on its surface. Polymer macrochains interact with the modifier monolayer, orienting themselves on the surface of AH particles. As a result of this orientation, the polymer-particle adhesive bond increases, which ultimately affects the increase in ultimate tensile stress and elongation at break.

However, with a further increase in the concentration of the modifier (over 3 wt. % ), a slight decrease in the ultimate tensile stress is observed. This circumstance once again confirms our assumptions about the multifunctionality of the studied modifying additive: plasticizing effect and improving the compatibility of the investigated components. It will be correct if the used modifier is considered as a plasticizer and compatibilizer of composite materials based on HDPE / LDPE and AH.

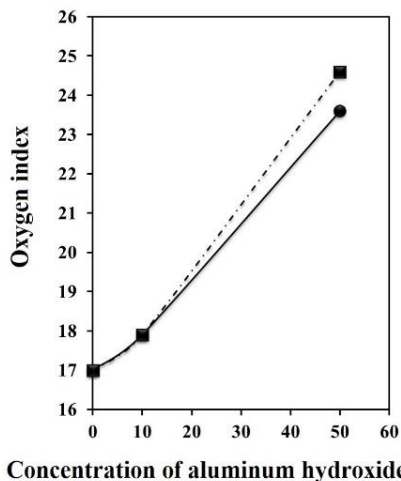
For a comparative assessment of the flammability of LDPE / HDPE + AH and LDPE / HDPE + AH + plasticizer, the method for determining the OI was used (figure 13). It should be noted that the OI values of composites with a plasticizer are higher than the OI values of HDPE / LDPE + AH composites.



**Figure 13.** Dependence of the oxygen index on the concentration of AH (wt. %) in the composites: ●- HDPE / LDPE + AH, ■- HDPE / LDPE + AH + 3% modifier.

**Influence of the compatibilizer on the properties of composites based on a mixture of low and high density polyethylene and aluminum hydroxide.** The effect of the concentration (1, 3, 5 wt. %) of the DuPont™ Fusabond® P353 additive compatibilizer (random copolymer of ethylene with propylene modified with maleic anhydride) on the physicomechanical properties, MFI, OI, heat resistance, kinetic regularities of the composite crystallization process of the HDPE/LDPE + 50 wt. % AH composite has been studied. In the study of physicomechanical properties, it was found that 1-3 wt. % is more optimal amount in terms of the effectiveness of the compatibilizer.

To assess the influence of the compatibilizer on the flammability of composites, a comparative analysis of the OI values of composites with and without a compatibilizer was made; it was found that the OI of the modified composites increased (figure 14).



**Figure 14.** Dependence of the oxygen index on the concentration of AH (wt. %) in composites: ●- HDPE / LDPE + AH, ■- HDPE / LDPE + AH + 3% compatibilizer

### **TECHNOLOGICAL FEATURES OF EXTRUSION OF COMPOSITE MATERIALS BASED ON A MIXTURE OF LOW AND HIGH DENSITY POLYETHYLENES AND MINERAL FILLERS**

The results of experimental studies on the selection of the optimal extrusion mode for polymer blends of HDPE + LDPE with different mixture ratios showed that, as the proportion of HDPE in the HDPE + LDPE blend increases, the optimal temperature regime of extrusion shifts to the region of relatively high temperatures. This is due to the fact that the melting point of HDPE is higher than LDPE. The existence of certain regularities in the change of characteristic properties and technological parameters indicates mutual uniform dispersion of the mixture components.

The next stage of the research was to study the separate effect of various mineral fillers (MMT, bentonite and AH) on the selection of the optimal extrusion mode for composite materials. The loading of various types of mineral fillers into the composition of the polymer mixture has a selective effect on the technological features of their

processing. Fillers with a layered structure (bentonite and MMT) have a positive effect on increasing the MFI of composites and on the technological features of their processing. The loading of AH affects the decrease in the MFR of the samples and thereby complicates their processing by extrusion, especially at high concentrations in the polymer mixture. After identifying the role of a single mineral filler on the technological features of the extrusion process, the influence of 2 pairs of fillers: MMT + AH and bentonite + AH on the optimization of the processing of composites based on HDPE / LDPE (50/50) will be considered. The simultaneous use of a layered filler with AH makes it possible to maintain the MFI of the composites at a satisfactory level.

## RESULTS

1. Compositions based on a mixture of LDPE and HDPE were obtained, and the effect of the ratio of polyethylenes on the physicomechanical, thermomechanical, rheological properties and the kinetic regularity of the crystallization of the mixture was studied [6], [21], [30]. It was found that at 40-60 / 60-40 HDPE + LDPE ratio the samples have the best physicomechanical properties. When the concentration of LDPE in the polyethylene mixture is up to 50 wt. %, the onset temperature of crystallization does not undergo any changes and remains within 114°C; with the content of LDPE over 50 wt. %, the first-order phase transition temperature and the density of the polymer composition begins to decrease naturally. Both cases are interpreted by phase inversion.
2. Composites based on a mixture of HDPE / LDPE (50/50) polyethylene, bentonite and AH were obtained, dilatometric and rheological studies were carried out, physicomechanical and thermomechanical properties, fire resistance, heat resistance, thermostability were studied. Research of the physicomechanical properties of HDPE / LDPE + bentonite nanocomposites showed that with an increase in the amount of filler, the elongation at break decreases, and the composite containing 20 wt. % bentonite, exhibits maximum strength properties [24]. As the amount of AH

in composites based on HDPE / LDPE + AH increased, their physicomechanical properties deteriorated [23].

3. The effect of the amount of filler on the regularities of changes in the temperature dependence of the specific volume and free specific volume was studied by the dilatometry method, and the approximate glass transition temperatures were determined [23], [24]. For HDPE / LDPE + bentonite nanocomposites, the first-order phase transition varied in the range 110-113°C. In composites HDPE / LDPE + HA containing 3-10 wt. % HA, the onset temperature of crystallization of the composites was 110°C, and for composites with a content of 30 and 50 wt. % - 105°C.
4. The rheological properties of composites based on a mixture of polyethylene are investigated - the dependence in logarithmic coordinates of the shear rate on the shear stress, the dependence of the viscosity of melts on the shear rate and inverse temperature [18], [26]. It was found that the “apparent” activation energy of viscous flow of nanocomposites based on bentonite (1, 5, 30 wt. %) varies in the range 14-39 kJ / mol, and for composites based on AH (1, 3, 5 wt. %) it varies in the range 11-31 kJ / mol. A universal temperature-invariant characteristic of the viscosity properties of composites has been constructed, which makes it possible to predict the change in the viscosity of the composites melt in a wide range of rates and shear stresses.
5. The fire resistance of the obtained composites was evaluated by the method of determining the oxygen index, and it was found that, with the same mass percentage of filler, AH-containing composites have a higher fire resistance than bentonite-containing ones. In the studied composites, no synergistic effect of bentonite and AH on the ignition of the polyethylene mixture was observed.
6. The addition of S- (carbalyoxy) methyl-N, N-diethyldithiocarbamate plasticizer and DuPont™ Fusabond® P353 compatibilizer to HDPE / LDPE + AH composite not only improved the physicomechanical properties, but also increased the fire resistance of the composites [25]. Among the studied samples,

the composite with HDPE / LDPE + 50 wt. % AN + 3 wt. % compatibilizer composition had the highest OI (24.6). A laboratory technological procedure for the obtaining of low-combustible composite materials has been developed and approved.

7. The mode of processing technology in the extruder of composites based on a polyethylene mixture was studied, it was found that the loading of various types of mineral fillers selectively affects the technological characteristics of their processing [17]. The research results are confirmed by the test report carried out by METAK LLC.

**The main provisions of the dissertation are published in the following scientific works:**

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