*Досліджено вплив властивостей інгредієнтів полімерних композицій, а саме полімерної матриці та типу і концентрації наповнювачів-апнтипіренів, на вогнестійкість та фізико-механічні властивості композиційних матеріалів кополімерів етилену з вінілацетатом. В якості наповнювачів-антипіренів використано тригідрати оксиду алюмінію, дигідрати оксиду магнію, гідромагнезити*

*Ключові слова: кополімер етилену з вінілацетатом, антипірени, фізико-механічні властивості, кисневий індекс*

*Исследовано влияние свойств ингредиентов полимерных композиций, а именно полимерной матрицы, типа и концентрации наполнителей-антипиренов, на огнестойкость и физико-механические характеристики композиционных материалов на основе сополимеров этилена с винилацетатом. В качестве наполнителей-антипиренов использовали тригидраты оксида алюминия, дигидраты оксида магния, гидромагнезиты*

*Ключевые слова: сополимер этилена с винилацетатом, антипирены, физико-механические характеристики, кислородный индекс*

UDC 679.7:678:544
DOI: 10.15587/1729-4061.2017.112003

# EFFECT OF FLAME RETARDANT FILLERS ON THE FIRE RESISTANCE AND PHYSICAL-MECHANICAL PROPERTIES OF POLYMERIC COMPOSITIONS

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## 1. Introduction

Polymeric composite materials are widely used in the manufacture of cable products [1]. Despite the progress in the field of polymeric materials science, there is a need to improve materials and technologies of polymerics processing. It is necessary to create special-purpose materials that meet specific operational requirements – non-proliferation of burning and use the halogen-free material.

The design of such compositions is particularly relevant in order to solve a problem on cable fire safety, all more frequent in recent cases of fires in places with large number of people, the cause of which was the ignition of cables' insulation. As a consequence, requirements for fire safety of cables increased. Previously, it was enough to provide for the non-proliferation of flame, while today it is also necessary to reduce the release of smoke and decrease the toxicity of combustion products.

Thus, manufacturers of polymeric composite materials constantly face the task of choosing a filler that would serve as a flame retardant. The European market has witnessed a trend for the accelerated growth in using halogen-free cable compositions. The principle of creation of formulations for halogen-free cable compositions is based on enhancing the oxygen index to the value of 35–40. This is achieved by introducing into the base polymer the fire retardants – hydroxides of metals. Industrially used are the hydroxides of aluminum $Al(OH)_3$ and magnesium $Mg(OH)_2$, of synthetic and natural origin, hydromagnesites.

Therefore, studying the dependence of change in the physical-mechanical properties and oxygen index on the chemical composition and the amount of flame retardant fillers, as well as dispersion, is a relevant task.

## 2. Literature review and problem statement

An analysis of the scientific literature demonstrates that one of the ways to reduce the flammability of polymeric materials based on polyolefins is the introduction of flame retardant fillers to the polymeric composition [2]. In particular, aluminum oxide trihydrates, magnesium oxide dihydrates [3].

In order to solve a complex task on protecting cable tracks from fires, industrialized countries have over the past decade made extensive use of the new-generation cables. The HF, FR, LS series with insulation and sheath made of halogen free polymeric compositions. Due to this, they exhibit reduced smoke and gas release. In these compositions, the process of endothermic reaction, observed under the influence of fire, results in dehydration with the release of water whose vapors dilute combustible gases and thus insulate the surface of cable from the effect of oxygen. In addition, metal oxides, which are formed during this reaction, create an additional isolating layer, as a result of which the place of ignition that occurred on the cable is not able to spread further and gradually fades [4].

Polyolefins are employed as a polymeric matrix of such compositions, and non-organic fillers – aluminum oxide trihydrates, magnesium oxide dihydrates, hydromagnesites – as flame retardants.

This issue is addressed in a large number of studies, which are considered in detail below. The fire-resistant properties of composite materials were investigated, whose base polymer is LDPE/EVA. Hydroxides of metals (magnesium hydroxide and aluminum oxide trihydrate) were used as the flame retardant fillers. Fire-resistant properties were estimated by horizontal burning and by the Oxygen Index [5].

Fire resistance of copolymer of ethylene with vinyl acetate was examined, which was filled with metal hydroxides (aluminum oxide trihydrate and magnesium oxide dihydrate) and silicon dioxide. It was noted that silica provides an advantage if the amount and other properties of fillers contribute to the formation of a protective mineral layer [6].

The possibility was explored of using magnesium hydroxide, zinc borate, as well as a combined action, as an inhibitor for the burning of a polypropylene fiber. The effectiveness of applying zinc borate was shown. Using magnesium hydroxide is ineffective. The thermal, mechanical and morphological properties were investigated [7].

There were studies into the influence of magnesium hydroxide and nanoclay on the deceleration of combustion of ethylene-vinyl acetate copolymer (EVA). The studies were carried out applying the methods for determining an oxygen index, calorimetric methods of analysis. Research results have shown effectiveness of replacing magnesium hydroxide and aluminum hydroxide in the compositions of EVA with 1–2 % of nanoclay [8].

It was established that ensuring better safety of electrical equipment and devices requires the use of composite materials that do not support combustion. Achieving this goal is possible due to the high degree of filling the polymeric matrix. Filling is performed by metal hydrates. A high degree of filling leads to a loss of flexibility and to low mechanical properties. In addition, recycling is more complicated [9].

Table 1

EVA characteristics

| Indicator | EVA 1 | EVA 2 |
|---|---|---|
| Density, kg/m$^3$ | 939 | 951 |
| Melt flow index, 2.16 kg, g/10 min | 2.5 | 5 |
| Content of vinyl acetate, % | 18 | 28 |

Table 2

Characteristics of fillers-fire retardants

| Indicator | $Al(OH)_3$ | | $Mg(OH)_2$ | | $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$; $Mg_3Ca(CO_3)_4$ |
|---|---|---|---|---|---|
| | Sample No. 1 | Sample No. 2 | Sample No. 3 | Sample No. 4 | Sample No. 5 |
| Mass fraction, %: | | | | | |
| – $Mg(OH)_2$ | – | – | >93 | >93.2 | 98.96 |
| – $Al(OH)_3$ | >99.2 | >99.5 | – | – | – |
| – $SiO_2$ | <0.05 | <0.1 | <0.05 | 2.2±0.2 | 0.67 |
| – $Fe_2O_3$ | <0.035 | <0.03 | <0.3 | 0.12±0.02 | 0.04 |
| – $Na_2O$ | <0.6 | <0.4 | <0.05 | – | <0.05 |
| – CaO | – | – | – | 2.2±0.2 | – |
| Median particle size, μm: | | | | | |
| – medium ($D_{50}$) | 1.5 | 3 | 3 | 3.7 | 1.4 |
| – max. ($D_{98}$) | 3.6 | 18 | 20 | 12.5 | 8.35 |
| – min. ($D_{10}$) | 0.5 | 1 | 1 | 1.1 | 1.02 |

## 3. The aim and objectives of the study

The aim of present research is to study the influence of the type and concentration of flame retardant fillers on fire resistance and physical-mechanical properties of composite materials of copolymers of ethylene with vinyl acetate that do not support combustion.

To achieve the set aim, the following tasks have to be solved:

– to study a dependence of the Oxygen Index of polymeric compositions on their formulations composition and properties of the ingredients;

– to establish the patterns of change in the physical-mechanical properties of polymeric compositions on the formulations of ingredients.

## 4. Materials and equipment to study fire resistance and physical-mechanical properties

We studied of ethylene-vinyl acetate copolymers (EVA) whose characteristics are given in Table 1, as well as flame retardant fillers, which were alumina trihydrate, magnesium oxide dihydrate, a mixture of magnesite and hydromagnesite. Characteristics of the flame retardant fillers are given in Table 2.

The data given in Tables 1, 2 were taken from the certificates of suppliers and are the results of acceptance control of raw materials.

Experimental samples of polymeric compositions of EVA with a different percentage of the flame retardant fillers of 40, 45, 50, 55, 60 % by weight in the formulation of each composition were made by a roller method at a temperature of (443±5) K over (7–10) minutes. Rollers have friction 1.5.

EVA and the flame retardant fillers were weighed on the scale accurate to 0.001 g and then loaded on the rollers. Temperature of the working roller is (443±5) K. Temperature of the cold roller is (438±5) K. The samples were rolled for 3 minutes on a gap of 0.4–0.5 mm. Then the gap was changed to 2 mm. In the process of rolling, we undercut them periodically, not less than 2 times per minute. No undercuts were made over the last minute of rolling.

The samples were conditioned at a temperature of (293±2) K for not less than 24 hours.

In order to test polymeric compositions for fire resistance and to conduct physical-mechanical studies, they were pressed at a temperature of (443±5) K. Thickness of the samples is 4 mm and 2 mm, respectively.

The study of fire resistance was performed by the method of Oxygen Index (OI). Oxygen Index is the minimum volumetric amount of oxygen in the oxygen-nitrogen mixture at which value a candle-like combustion of materials is possible under conditions of special tests and which is expressed as a percentage [10]. Determining the Oxygen Index was carried out using special measuring devices, in which the combustion of tested materials is performed in an oxygen-nitrogen mixture whose composition is controlled by atmospheric pressure and at a normal temperature. That is, the value of Oxygen Index is the minimum concentration of oxygen in the flow of oxygen-nitrogen mixture at which an independent burning of vertically positioned sample occurs, which is lit from top in accordance with EN ISO 4589-2:1999 Plastics – Determining of burning behavior by oxygen index. Oxygen Index (OI) in percentage was determined by formula:

$$KI = \frac{V_1}{V_1 + V_2} \cdot 100,$$

where $V_1$ is the volumetric flow rate of oxygen, $cm^3/s$; $V_2$ is the volumetric flow rate of oxygen $cm^3/s$.

Error of tools for measuring the concentration of oxygen in the flow of mixture does not exceed ±1 %.

Studies into physical-mechanical properties of polymeric compositions (tensile strength, relative elongation at break, the modulus of elasticity at break) were conducted in accordance with EN 60811-501:2012 Electric and optical fibre cables. Test methods for non-metallic materials. Part 501. Mechanical tests. Test for determining the mechanical properties of insulating and sheathing compounds.

Motion speed of clamps of the tensile testing machine while determining tensile strength and relative elongation at break was 50 mm/min.

Processing of results and construction of charts were performed using the software Microsoft Office Excel 2007.

Adequacy of regression equation was checked by examining statistical significance of determination coefficient $R^2$ for the $F$-criterion derived from formula [11]:

$$F_p = \frac{R^2}{1 - R^2} \times \frac{n - m - 1}{m},$$

where $n$ is the number of observations; $m$ is the number of factors in the regression equation.

## 5. Results of research into fire resistance and physical-mechanical properties of polymeric compositions

Flame-retardant properties of polymeric compositions, filled with aluminum oxide trihydrates, magnesium oxide dihydrates, hydromagnesites, were estimated by the method of oxygen index. We changed the formulation of fillers in the compositions from 40 % by weight to 60 % by weight. We determined how much oxygen index depends on the amount of a flame retardant fillers. Research results are shown in Fig. 1, 2.

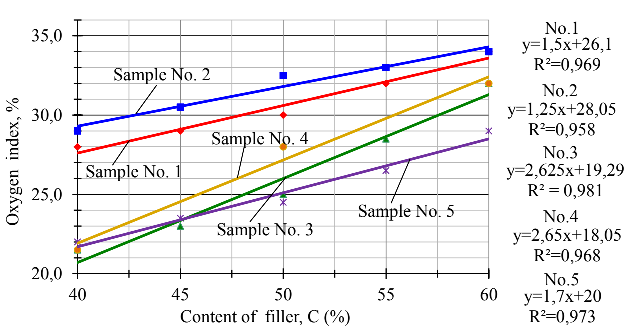


Fig. 1. Dependence of OI on the content of a flame retardant filler (Samples Nos. 1–5) in the polymeric compositions based on EVA 1

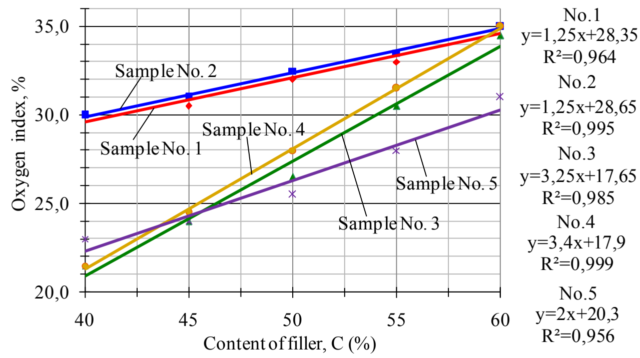


Fig. 2. Dependence of OI on the content of a flame retardant filler (Samples Nos. 1–5) in the polymeric compositions based on EVA 2

The estimated level of significance σ=(0.009–0.02)<0.05 confirms the significance of $R^2$. The significance testing of $R^2$ is based on checking the indicator $F_r$ in the critical region ($F_{cr}$, ∞). Since $F_r$=(22.8–999) matches the critical interval (18.5; +∞), then the hypothesis about $H_0$: $R^2=0$ is rejected, that is, determination coefficient $R^2$ is significant. Thus, the regression equations constructed by the Fisher's $F$- criterion are adequate.

We explored physical-mechanical properties of the polymeric compositions. Destructive stress at break was determined from formula [12]:

$$\sigma_p = \frac{P_p}{S}, \quad S = a \cdot d,$$

where $\sigma_p$ is the value of destructive stress, MPa; $P_p$ is the load at breaking, N; $S$ is the cross-sectional area, $mm^2$; $a$ is the width of the working part of the blade, mm; $d$ is the thickness of the working part of the blade, mm.

Relative elongation at break was determined from formula [12]:

$$\varepsilon_0 = \Delta l_{op} / l_0 \cdot 100,$$

where $\varepsilon_0$ is the relative elongation, %; $\Delta l_{op}$ is the change in the estimated length of sample at a moment of breaking, mm; $l_0$ is the estimated length, mm.

Results of the experiments are shown in Fig. 3–6.

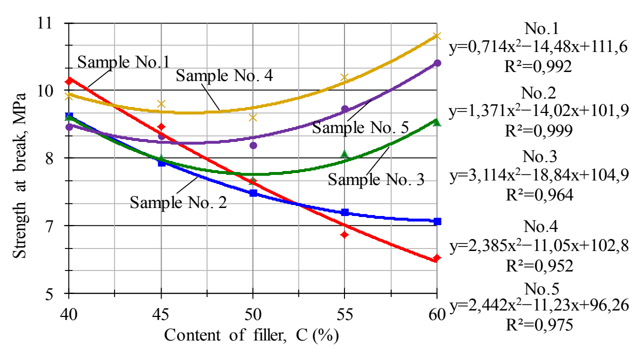


Fig. 3. Dependence of strength at break on the content of a flame retardant filler (Samples Nos. 1–5) in the polymeric compositions based on EVA 1

Destructive stress and relative elongation at break were determined after ageing the samples of polymeric compositions ($T$=373 K, $\tau$=168 hours). Research results are shown in Fig. 7–10.

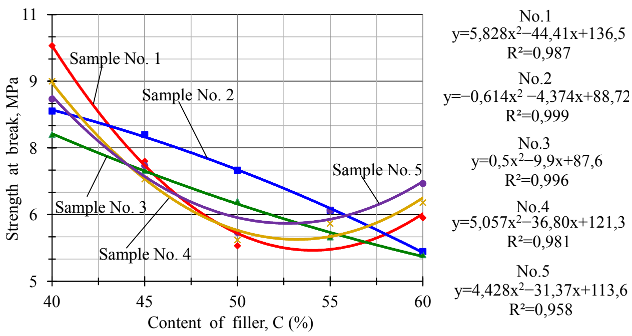


Fig. 4. Dependence of strength at break on the content of a flame retardant filler (Samples Nos. 1–5) in the polymeric compositions based on EVA 2

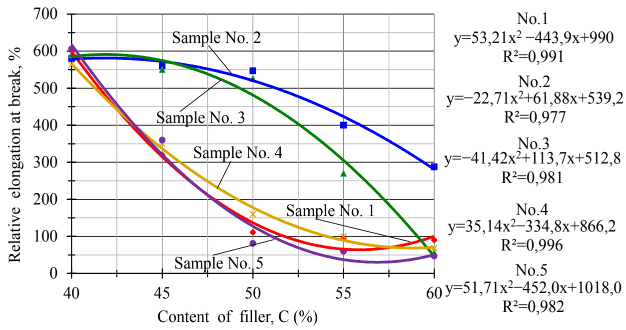


Fig. 5. Dependence of relative elongation at break on the content of a flame retardant filler (Samples Nos. 1–5) in the polymeric compositions based on EVA 1

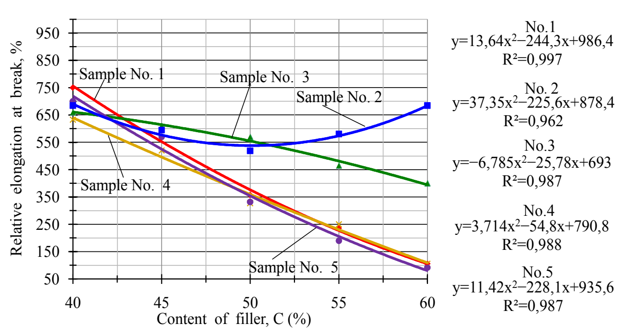


Fig. 6. Dependence of relative elongation at break on the content of a flame retardant filler (Samples Nos. 1–5) in the polymeric compositions based on EVA 2

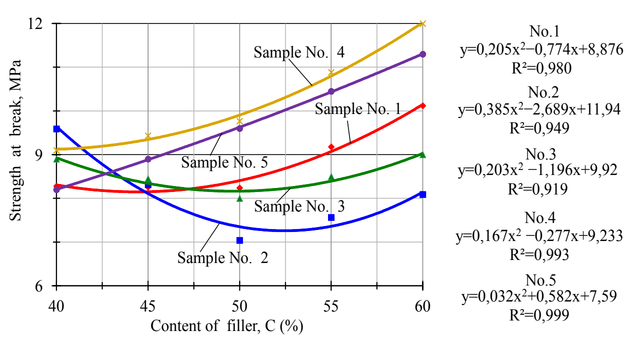


Fig. 7. Dependence of strength at break on the content of a flame retardant filler (Samples Nos. 1–5) in the polymeric compositions based on EVA 1 after aging

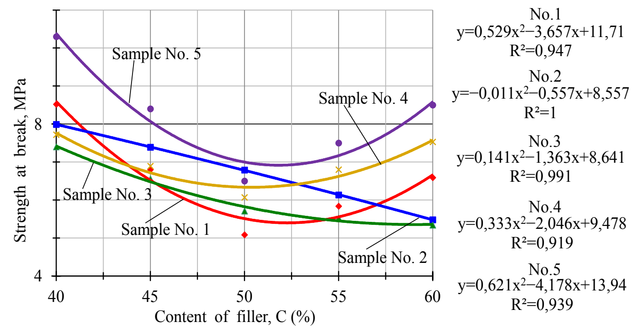


Fig. 8. Dependence of strength at break on the content of a flame retardant filler (Samples Nos. 1–5) in the polymeric compositions based on EVA 2 after aging

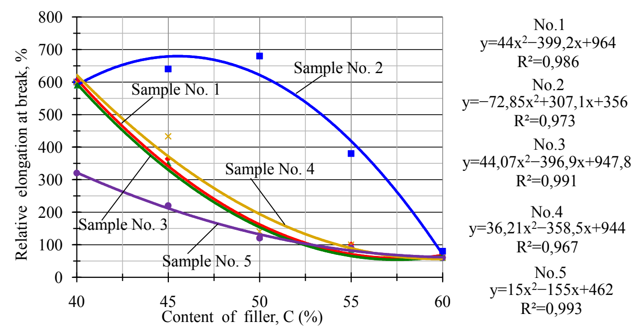


Fig. 9. Dependence of relative elongation at break on the content of a flame retardant filler (Samples Nos. 1–5) in the polymeric compositions based on EVA 1 after aging

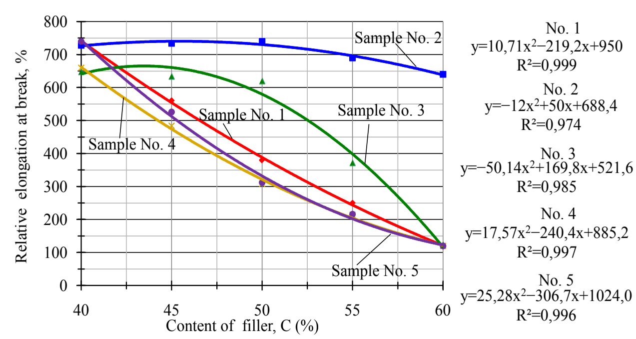


Fig. 10. Dependence of relative elongation at break on the content of a flame retardant filler (Samples Nos. 1–5) in the polymeric compositions based on EVA 2 after aging

The modulus of elasticity at stretching ($E_p$) was determined from formula, in line with GOST 9550-81 "Plastic masses. Methods for determining the modulus of elasticity at stretching, compression and bending":

$$E_p = \frac{(F_2 - F_1)\cdot l_0}{A_0 \cdot (\Delta l_2 - \Delta l_1)},$$

where $F_2$ is the load that corresponds to the relative elongation of 0.3 %; $F_1$ is the load that corresponds to the relative elongation of 0.1 %; $l_0$ is the estimated length of the sample; $A_0$ is the initial cross-sectional area of the sample, mm$^2$; $\Delta l_2$ is the elongation that corresponds to load $F_2$, mm; $\Delta l_1$ is the elongation that corresponds to load $F_1$, mm.

Research results are shown in Fig. 11, 12.

Results of the studies, which are shown in Fig. 11, 12, indicate a substantial increase in the value of modulus of elasticity with an increase in the content of flame retardant fillers for all compositions.

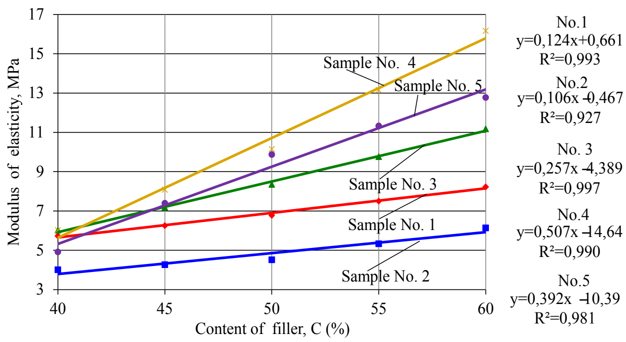


Fig. 11. Dependence of the modulus of elasticity on the content of a flame retardant filler (Samples Nos. 1–5) in the polymeric compositions based on EVA 1

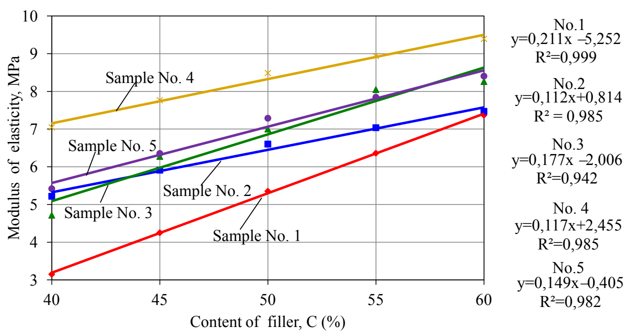


Fig. 12. Dependence of the modulus of elasticity on the content of a flame retardant filler (Samples Nos. 1–5) in the polymeric compositions based on EVA 2

## 6. Discussion of results of studying fire resistance and physical-mechanical properties of polymeric compositions

Fire resistance of the polymeric compositions EVA 1 and EVA 2, filled with flame retardants (sample No. 1–5), were determined using a method of Oxygen Index. The results of research, shown in Figs 1, 2, demonstrate that Oxygen Index increases with an increase in the content of flame retardant fillers for all polymeric compositions. It should be noted that Oxygen Index of the polymeric compositions EVA 2 grows more intensively than that of the polymeric compositions EVA 1. The polymeric compositions EVA 2 contain 28 % of vinyl acetate and their melt flow index is 2.5 g/10 min. The polymeric compositions EVA 1 contain 18 % of vinyl acetate and their melt flow index is 1.8 g/10 min. In other words, the content of vinyl acetate and melt flow index affect the values of Oxygen Index. That is, degradation products of the polymeric matrix also affect the inhibiting process of combustion of polymeric compositions.

If we compare Oxygen Indexes of polymeric compositions with flame retardant fillers, then more effective are aluminum oxide trihydrates (sample No. 1, No. 2). At the same time, Oxygen Index of the polymeric composition, filled with flame retardant of Sample 1, is larger than that of flame retardant of Sample 2. Intermediate value of Oxygen Index characterizes polymeric compositions, filled with magnesium oxide dihydrate (Sample No. 3, No. 4), out of which Sample No. 3 is more efficient. That is, the flame retardant fillers of aluminum oxide trihydrate and magnesium oxide dihydrate, which have a smaller average diameter of particles, are more effective inhibitors of combustion of polymeric compositions. The least effective flame retardant is hydromagnesite (Sample No. 5).

According to [13], the content of oxygen in air is about 21 % by volume, which is why the Oxygen Index (OI) of materials that burn in the atmosphere of air is lower than 21 %. Polymeric materials, which have OI<20 %, burn fast in the atmosphere of air. The materials with OI from 20 % to 27 % burn slow in the atmosphere of air. Those polymeric materials whose OI exceeds 27 % under condition of fire termination are considered to be difficult to burn and are self-extinguishing. Thus, according to the research results, such polymeric compositions based on EVA 1 with the following content of fire retardants are difficult to burn:

– from 40 % by weight to 60 % by weight of a fire retardant – Sample No. 1;

– from 45 % by weight to 60 % by weight of a fire retardant – Sample No. 2;

– from 48 % by weight to 60 % by weight of a fire retardant – Sample No. 3;

– from 52 % by weight to 60 % by weight of a fire retardant – Sample No. 4;

– from 57 % by weight to 60 % by weight of a fire retardant – Sample No. 5.

When creating difficult-to-burn polymeric compositions based on EVA 2, the content of fire retardants must be as follows:

– from 40 % by weight to 60 % by weight of a fire retardant – Sample No. 1 and Sample No. 2;

– from 47 % by weight to 60 % by weight of a fire retardant – Sample No. 3;

– from 48 % by weight to 60 % by weight of a fire retardant – Sample No. 4;

– from 50 % by weight to 60 % by weight of a fire retardant – Sample No. 5.

By analyzing the results prior to aging (Fig. 3–8), we established a pattern of change in the physical-mechanical properties. The introduction of flame retardant fillers significantly affects the value of destructive strain and relative elongation at breaking, the modulus of elasticity at stretching. There is a different change in the destructive stress depending on the content of a filler in the polymeric composition. When using the fire retardants Sample No. 1 and Sample No. 2, an increase in the content of a filler results in a decrease in the destructive stress from 9.7 MPa to 5.3 MPa, and from 8.7 MPa to 6.1 MPa, respectively.

In the polymeric compositions with flame retardants Sample No. 3, Sample No. 4, Sample No. 5, the minimum value of destructive stress is achieved when the content of a filler makes up from 50 % by weight to 5 5 % by weight. Then there is an increase of this indicator to the value of 6.0 MPa in the case of using EVA 1 as a polymeric matrix, and to 7.5 MPa in the case of exploiting EVA 2 as a polymeric matrix.

The modulus of elasticity of all polymeric compositions grows if the content of a flame retardant increases. It acquires the greatest value in the compositions based on EVA 1, as well as while employing the fire retardants (Samples No. 3, No. 4, No. 5).

The relative elongation at break of all polymeric compositions reduces with an increase in the content of fillers and has a larger value for polymeric compositions based on EVA 2.

The physical-mechanical properties of polymeric compositions change upon aging. The destructive stress at breaking has a certain minimum at the value of filling of about 50 % by weight (flame retardants Samples No. 1, No. 2, No. 3 and EVA 1; flame retardants Samples No. 1, No. 3, No. 5 and EVA 2). Destructive stress rises with a further increase in the content of a filler. Destructive stress of polymeric compositions based on EVA 1 and fire retardants (Samples No. 4, No. 5) increases during aging. For the polymeric compositions based on EVA 2 and flame retardant fillers (Samples No. 2 and No. 3), destructive stress reduces with an increase in the content of fillers.

Relative elongation of polymeric compositions employing all fire retardants decreases during aging with an increase in the content of fire retardants. A sharp decline in these characteristics is observed under conditions of using all compositions based on EVA 1. The polymeric compositions based on EVA 2 exhibit the same tendency, except for the material, which is filled with a fire retardant (Sample No. 2).

The given studies have shown the effect of such fillers as aluminum oxide trihydrates, magnesium oxide dihydrates, hydromagnesites on the fire resistance and physical-mechanical properties of polymeric compositions based on EVA, which 1 do not support combustion. Carrying out such a complex of research allows us to demonstrate the possibilities to control operational characteristics of polymeric compositions based on EVA, which do not support combustion.

Thus, the results of present study demonstrate the dependence of operational properties of polymeric materials on the chemical composition and dispersion of the flame retardant fillers. Previous research showed effect of separate flame retardant fillers. This renders comparative assessment of their action impossible.

Continuation of the present studies might address the regulation of operational properties using the nano-structured modification of polymeric compositions.

## 7. Conclusions

1. Oxygen Index of polymeric compositions that do not support combustion depends on the properties of ethylene-vinyl acetate copolymer. EVA 2 has a larger value of the content of vinyl acetate and the melt flow index than that of EVA 1. Moreover, it inhibits the combustion process more effectively, under conditions of filling with various flame retardants.

Flame retardant fillers increase the Oxygen Index of polymeric compositions to the required value (27–37 %) at the concentration of fillers from 40 % by weight to 60 % by weight depending on their composition and dispersion. The most effective is aluminum oxide trihydrate (Sample No. 1) with a smaller average diameter of the particles. The lowest efficiency is demonstrated by hydromagnesites (Sample No. 5).

2. Employing the flame retardant fillers with different composition and dispersion exerts a certain effect on the physical-mechanical properties. It was established that an increase in the content of flame retardant fillers in polymeric compositions results in reduced destructive stress and relative elongation at break. The modulus of elasticity of all polymeric compositions increases.

During aging, the physical-mechanical characteristics change. Tensile strength of polymeric compositions based on EVA 1 and all fire retardants grows. It passes the minimum value for the filling of about 50 % by weight of the EVA compositions. Relative elongation at stretching decreases for all polymeric compositions.

Thus, the studies conducted have shown the possibility to control operational characteristics of polymeric compositions of EVA, which do not support combustion, and fire retardants-fillers of varying qualitative composition.

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