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Increasing The Fire Resistance Of Polymer Building Materials Based On Technogenic Waste

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Abstract. **Today, comprehensive measures are being implemented around the world and certain results are being achieved in the field of further deepening economic reforms in the building materials industry and the rapid development of the network, increasing the production of new modern building materials, structures and products, and expanding their range. Expanding the range of manufactured building materials, increasing the share of production of modern, convenient, and high-quality products based on a localization program, reducing imports, as well as the further development of the industry remain pressing issues of today.**
The studies present methods for increasing the fire resistance of polymer building materials obtained based on fly ash from the furnaces of the Shirin Thermal Power Plant and JSC Uzmetkombinat, waste phosphogypsum generated at JSC Maxam-Ammafos, physical, mechanical, and thermodynamic properties, and also studied optimal proportions for obtaining composites of fire-resistant polymer building materials.
To conduct an experimental study, particles of technogenic industrial waste of the most optimal size, types of filler, and necessary components were selected. Studies of the elemental composition of industrial technogenic waste and X-ray diffraction analysis have shown that they consist mainly of silicon dioxide, which consists of more than 80% of the amorphous phase.
Based on the analysis of the X-ray phase structure, it was determined that the introduction of additives into the fly ash fillers of the Shirin Thermal Power Plant and Uzmetkombinat JSC, as well as phosphogypsum waste from Maxam-Ammofos JSC, leads to an increase in the thermal stability of polymer materials. The main reason for this is explained by the fact that the composition of fly ash and phosphogypsum waste introduced into the polymer composite contains metal oxides, silicon oxide, and phosphorus compounds, which are resistant to temperatures and have fire-retardant properties.
The fire resistance properties of the proposed polymer-containing building materials modified with fire-resistant compositions of the PPA, PPK and PPM brands, consisting of polymer binders, fire-retardant additives and fillers, were studied based on experimental tests, while the oxygen index was increased from 18.5 to 33.0%, while the weight loss during thermal oxidation was reduced from 97.5 to 73–75.5%, and the flammability level according to state standard 28157 was changed from PV-2 to PV-0.

Keywords: composite composition, fire resistance, fly ash, man-made waste, phosphogypsum, polyethylene, polymer building materials, thermal analysis.

I. INTRODUCTION

Today, comprehensive measures are being implemented around the world and certain results are being achieved in the field of further deepening economic reforms in the building materials industry and the rapid development of the network, increasing the production of new modern building materials, structures, and products, and expanding their range. Expanding the range of manufactured building materials, increasing the share of production of modern, convenient, and high-quality products based on a localization program, reducing imports, as well as the further development of the industry remain pressing issues of today.

Particular attention in the world is paid to the production of new structures of building materials on a global scale. In this regard, the creation of modern compositions of multifunctional additives based on

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technogenic waste for the production of building materials is one of the important tasks of research in this direction.

To substantiate appropriate scientific decisions on the development of new compositions of multifunctional effective additives and, with their participation, highly effective building materials, such issues as the creation of new methods for obtaining effective types of building products based on artificial additives, the use of new compositions for obtaining multifunctional building materials with the participation of recycled materials are becoming increasingly relevant. raw materials, optimizing the composition of raw materials to increase the strength of heat-resistant and fire-resistant building materials, creating energy-efficient building materials, modernizing technologies for the production of heat-resistant and fire-resistant building materials, and using local alternative sources of mineral additives to increase their volume.

Currently, one of the most commonly used materials in the construction of modern buildings under construction is polymeric materials, which, in addition to their effective properties for resistance to aggressive environments, must be fire resistant. The scientific and technical literature on the development of fire-resistant and fire-resistant building materials, on ensuring the fire safety of buildings and structures, in particular, effective scientific solutions for the development and use of fire-resistant materials, as well as ways to improve them, is analysed.

In the Republic of Uzbekistan, comprehensive measures are being taken to produce high-quality building materials, aimed at meeting their demand, modernizing the economy, and creating new production capacities.

The Development Strategy of New Uzbekistan for 2022-2026 defines the tasks of "development of production facilities, modernization and diversification of industry, practical application of inexpensive energy-efficient methods, development of the industry for the production of building materials containing polymers, preparation of import-substituting and export products." In this regard, scientific research aimed at developing new compositions of multifunctional additives based on technogenic waste, and with their participation new types of highly efficient building materials are extremely important [1].

This study will, to a certain extent, serve the implementation of the tasks set in the Resolutions of the President of the Republic of Uzbekistan dated February 20, 2019, № PP-4198 "On measures for radical improvement and comprehensive development of the building materials industry", dated May 25, 2019, № PP-4335 "On additional measures to accelerate the development of the building materials industry", dated February 13, 2021, № PP-4992 "On measures for further reform and financial recovery of chemical industry enterprises, development of production of chemical products with high added value", dated January 24, 2022, № PP-99 "On measures to create an effective system for the development of production and expansion of industrial cooperation in the republic", as well as in other regulatory legal acts related to activities in this area [2, 3, 4]

II. Materials and Methods

A. Goals and objectives of research into increasing the fire resistance of polymer building materials

The purpose of the research is to develop a new technology for producing effective additives based on industrial waste to create energy-efficient, heat-resistant, fire-resistant building materials.

The objectives of the study are: to study the influence of technogenic waste on the formation of heat-resistance and fire-resistant properties of polymer-based building materials; study technological, heat-resistant, and physical-mechanical properties of heat-resistant and fire-resistant building materials; optimization of the composition of heat-resistant and fire-resistant building materials obtained from industrial waste; determining the feasibility of creating and implementing modern technology for the production of heat-resistant and fire-resistant building materials obtained from industrial waste [5, 6, 7]

B. Production of fire-resistant polymer building materials based on fly ash and phosphogypsum

Phosphogypsum waste from Maxam-Ammofos JSC was used as technogenic waste.

Fly ash is a finely dispersed material, which consists of particles up to 0.14 mm in size, is formed as a result of the combustion of solid fuel at a thermal power plant and is captured by electric precipitators, after which it is taken in a dry state using an ash collector for production needs, or together with water and the slag is sent to the ash dump [8].

Phosphogypsum is a calcium sulfate hydrate formed as a by-product during the production of fertilizers from phosphate rock [9].

Based on the experiments carried out, three composite compositions of the brand were obtained, which were named PPA, PPK, and PPM.

Composite composition of the PPA brand. 40 g of fly ash and 40 g of vermiculite powder were placed in a heat-resistant glass and mixed using a special mixer at a temperature of 100 -150° C, and the mass ratio should be 1:1. Then 10 g of ammonium sulfate was added to the mixture, and the reaction process was continued for 1.0 hours, stirring at a temperature of 100° C. As a polymer binder, 10 g of polysulfide oligomer was added to the mixture, brought to a homogeneous state, and stirred for 0. 5 hours at a temperature of 80-90° C. The resulting finished product was brought to a state of readiness for modification with polyethylene. Let us give the ratio of substances in the composite composition of the PPA brand:

The ratio of substances in the first composite composition of the PPA brand:

fly ash – 40%;
vermiculite – 40%;
ammonium sulfate – 10%;
polysulfide oligomer – 10%.

Composite composition of the PPK brand. 30 g of fly ash and 30 g of phosphogypsum waste powder were placed in a heat-resistant glass and mixed using a special mixer at a temperature of 100–150°C, and the mass ratio should be 1:1. Then 20 g of silicon oxide and 15 g of amorphous adduct (orthophosphoric acid and amorphous 1:1) were added to the mixture, and the reaction process was continued for 1.0 hours, stirring at a temperature of 100º C. As a polymer binder, the mixture was brought to a homogeneous state, and 5 g of polysulfide oligomer was added and stirred for 0.5 hours at a temperature of 80–90º C. The resulting finished product was brought to a state of readiness for modification with polyethylene. Let us present the ratio of substances in the composite composition of the PPK brand:

The ratio of substances in the second composite composition of the PPK brand:

fly ash – 30%;
phosphogypsum – 30%;
silicon oxide – 20%;
amorphous adduct (orthophosphoric acid and amorphous 1:1) – 15%;
polysulfide oligomer – 5%.

Composite composition of the PPM brand. 30 g of fly ash and 25 g of vermiculite powder were placed in a heat-resistant glass and mixed using a special mixer at a temperature of 100–150°C. Then 20 g of amorphous adduct (orthophosphoric acid and amorphous 1:1) and 15 g of montmorillonite were added to the mixture, and the reaction process was continued for 1.0 hours, stirring at a temperature of 100º C. As a polymer binder, the mixture was brought to a homogeneous state, and 10 g of polysulfide oligomer was added and stirred for 0.5 hours at a temperature of 80–90º C. The resulting finished product was brought to a state of readiness for modification with polyethylene. Let us give the ratio of substances in the composite composition of the PPM brand:

The ratio of substances in the third composite composition of the PPM brand:

fly ash – 30%;
vermiculite – 25%;
ammophos adduct (orthophosphoric acid and ammophos 1:1) – 20%;
montmorillonite – 15%;
polysulfide oligomer – 10%.

III. Results and discussion

The thermal properties of oligomers of the sulfur-containing class were studied by thermal analysis. The mass of the polysulfide rubber sample does not change up to 210 º C (Fig. 1). The thermal analysis curve shows a single endothermic peak (at 190.1°C) in the temperature range 30–210°C, which corresponds to melting of the sample.

A sample at temperatures above 210°C begins to decompose in two stages: up to 270°C at a rate of 7% per minute and above 270°C at a rate of 2.6% per minute with a total weight loss of 75%. The decomposition reaction is endothermic with a total decomposition energy of 308.7 J/g. Based on the experiments carried out, the kinetic constants, energetic activity, and reaction order change as a result of the formation of various reactions due to the influence of temperature.

Thermogravimetric studies without both temperature ranges are not specifically studied, since experiments were conducted with multicomponent fire-resistant polymer composites and results of thermogravimetric studies were obtained in the range from 20º C to 800 º C.

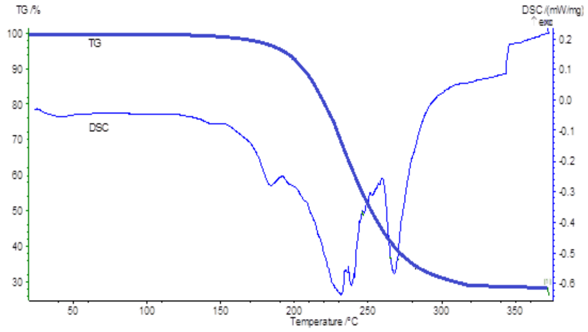

Fig. 1. Results of thermal analysis of polysulfide rubbers containing sulfur in their composition.

When calculating energy activity, the basic information depends on several methods for determining dynamic temperature indicators. The results obtained using these methods may vary. Based on the results of the research, scientific and theoretical research has proven that experimental research results obtained by the thermogravimetric method are highly effective.

In the process of modifying polyethylene building compositions at a temperature range of 200–220°C in an amount of 10, 20, and 30% wt. composite compositions of the PPA brand (fly ash, vermiculite, ammonium sulfate, polysulfide rubber), PPK brand (fly ash, phosphogypsum waste, silicon oxide, ammophos adduct, polysulfide rubber), PPM brand (fly ash, vermiculite, ammophos adduct, montmorillonite, polysulfide rubber) changes in the flow rates of fire-resistant polymer building materials are presented in Table. 1.

It should be noted that when a filler is introduced into a polymer composition at a temperature of 200–220°C, first the fluidity of polymers under the influence of temperature does not increase significantly, and then when the amount of filler (PPA: PE) was 10% wt ., it was found that when exposed to temperature the fluidity index was 1.0–1.5 g in 10 minutes, and at 20% wt. decreased to 0.7–0.6 g per 10 min. When the amount of fire-resistant filler (2–15 µm) reaches 20–30% wt. a significant decrease in the fluidity of polymers under the influence of temperature was observed [5].

As can be seen from the study results, the melt flow index changed little when the amount of filler was 30% wt. and particle size was 7 microns. It follows that the fluidity of compounds under the influence of temperature depends on the type of filler. It was revealed that the fluidity under the influence of temperature of fire-resistant polymer building materials in PPM: PE ratios of 10–30% wt. differs slightly compared to the original sample.

TABLE 1 FLOW INDEX OF FIRE-RESISTANT POLYMER BUILDING MATERIALS BASED ON MODIFIED POLYETHYLENE GRADE F-0220 WITH ADDITIVES GRADES PPA, PPK, AND PPM

Composition of compositions	Additive particle size, microns	Amount of polymer and fillers of PPA, PPK and PPM brands, % wt			
		100/0	90/10	80/20	70/30
		MTR g/10 min (200 °C; 2.16 kg)			
Polyethylene grade F–0220	–	1.5–2.5	–	–	–
PPA+PE	7	–	1.0–1.5	0.7–0.6	–
	10	–	0.7–0.6	0.4–0.3	–
	15	–	0.5–0.4	0.3–0.2	–
PPK+PE	7	–	1.0–2.0	0.8–0.7	0.5–0.3
	10	–	0.8–0.7	0.7–0.5	–
	15	–	0.7–0.6	0.5–0.4	–
PPM+PE	7	–	1.5–2.0	1.0–0.8	0.6–0.4
	10	–	0.8–1.0	0.9–0.6	0.4–0.2
	15	–	0.8–0.5	0.7–0.5	–

In the process of modifying polypropylene building compositions at a temperature range of 200–220°C in an amount of 10, 20 and 30% by weight with composite compositions of the PPA brand (fly ash, vermiculite, ammonium sulfate, polysulfide rubber), PPK brand (fly ash, phosphogypsum waste, silicon oxide, ammophos adduct, polysulfide rubber), grades of PPM (fly ash, vermiculite, ammophos adduct, montmorillonite, polysulfide rubber) changes in the fluidity indicators of fire-resistant polymer building materials are presented in the table. 2 [6].

From this table. 3. It can be seen that when a filler is introduced into a polymer composition at a temperature of 200–230°C, the fluidity of polymers under the influence of temperature does not increase significantly, and then when the amount of filler is 30% wt. With a particle size of 7 μm, it is possible to analyze the increase in the flow rate of polymers under the influence of temperature. As can be seen from the study results, the melt flow rate changed significantly when the amount of filler was 30% wt. and particle size was 7 microns.

TABLE 2 FLOW INDEX OF FIRE-RESISTANT POLYMER BUILDING MATERIALS BASED ON MODIFIED POLYPROPYLENE GRADE P-Y342 WITH ADDITIVES GRADES PPA, PPK, AND PPM

Composition of compositions	Additive particle size, microns	Quantity of polymer and grade fillers PPA, PPC, and PPM % wt.			
		100/0	90/10	80/20	70/30
		MTR g/10 min (200 °C; 2.16 kg)			
Polypropylene grade P-Y342	–	0.25–0.30	–	–	–
PPA+PP	7	–	0.25	0.2	0.1
	10	–	0.1	0.08	–
PPK+PP	7	–	0.20	0.15	0.1
	10	–	0.15	0.09	–
PPM+PP	7	–	0.2	0.15	0.09
	10	–	0.15	0.09	–

It follows that the fluidity of compounds under the influence of temperature depends on the type of filler. It was revealed that the fluidity index under the influence of temperature of fire-resistant polymer building materials in the ratio PPM: PP 30% wt. decreased slightly compared to the original polymer. This made it possible to scientifically substantiate the possibility of adopting an acceptable filler concentration of 30 wt.% for PP and an acceptable size of 7–10 microns.

Currently, polypropylene is one of the main products intended for the production of fire-resistant polymers. Taking into account the production of various products from it and its leading position among thermoplastics, we will also pay special attention to calculating percentages in the process of conducting a sequence of experiments.

Study of the thermophysical properties of building materials based on refractory polymers. The indicators of production and thermal decomposition of composites produced in our republic have been determined polyethylene with groups consisting of aluminum, calcium, magnesium, and silicon.

Polyethylene-based polymer composite materials are widely used in the processes of external and internal finishing of buildings and structures, in water-permeable pipelines, household electrical appliances, and the furniture industry, while the requirements for the fire resistance of these polymer materials are increasing from year to year. The indicators of thermal decomposition of a polymer composite material obtained by mixing at a temperature of 180-2000 s through a special extruder during laboratory processing of polyethylene brand F-0220S, produced in our republic, with 10-20% fire-resistant compositions of the PPA, PPK, and PPM brands, raw materials were studied which are available in our country (Fig. 2).

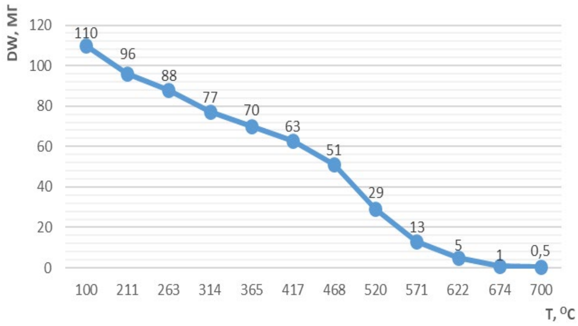

Fig. 2. Thermal analysis of polyethylene sample F-0220-S

On the derivatogram curve of a sample prepared by adding a mixture of polyethylene and fire-resistant compounds to obtain a polymer composite, five endothermic effects were detected at 114, 131, 183, 431, 657 o C, and nine exothermic effects at 241, 343, 369, 381, 512, 589. 621, 697, and 700 o C. The total reduction in mass in the temperature range of 100-700 o C of the thermogravimetric curve was 99.5% [7].

During the analysis, experimental tests were carried out to determine the average rate of mass loss of polymers and their composites, and the period of the experiment and the mass of the substance were determined. Thus, the properties of thermal-oxidative destruction of a polymer composite were studied, based on data from studies of the kinetics of processes in the temperature range from 440 K to 1023 K.

For oligomers with reactivity, the choice of modifier system is one of the main issues in the production of binders. Polymer composite materials with a modified polymer matrix must have high-performance properties. To achieve this, the polymer matrix must have high reactivity and high molecular mobility. On the other hand, the polymer matrix is subject to requirements for high heat resistance. This requires an increase in the crystallization temperature, and a high crystallization temperature, in turn, leads to a decrease in molecular mobility.

TABLE -3 DETERMINATION OF THE AVERAGE RATE OF WEIGHT LOSS USING THE THT ANALYSIS METHOD

Period, °C	Weight loss, mg	Average rate of weight loss, mg/min $v_m = \Delta m / \Delta\tau$
Polyethylene F-0220S		
211-314 365-520 571-700	33 48 28.5	1.8 1.98 1.19
Polyethlene (F-0220S) + PPA		
243-373 417-547 590-700	20 thirty 23	1.0 1.5 1.25
Polyethylene (F-0220S) +PPK		
268-364 412-508 556-700	19 40 24	1.26 2.66 1.23
Polyethylene (F-0220S) +PPM		
202-338 383-519 564-700	9.0 thirty 50	0.45 1.5 2.5

mass loss rate (v_m), Δm — lost mass, mg; $\Delta\tau$ - time.

The physical and mechanical properties of polymer building materials modified with fire-resistant fillers have been analyzed, and the influence of fire-resistant polymer building materials based on polyethylene and polypropylene, modified with fillers of the PPA, PPK, and PPM brands, on the properties of polymers has been studied. These filters are introduced into PE in an amount of 10–30% wt. and their properties are compared with analogs. The physical and mechanical properties of PE have been studied. A comparative analysis of the physical and mechanical properties of polymer building materials based on fire-resistant fillers is presented in Table. 4. When introducing a fire resistance modifier of the PPA brand into the polymer composite, it was found that the impact resistance partially increased from 48 to 50 kJ/m2 compared to the original polyethylene, and the tensile strength decreased from 25 to 23 MPa, and the bending strength decreased from 20 to 19 MPa, while it was analyzed that the indicators are higher compared to analogs [10].

TABLE 4 COMPARATIVE ANALYSIS OF THE PHYSICAL AND MECHANICAL PROPERTIES OF POLYMER BUILDING MATERIALS OBTAINED BASED ON FIRE-RESISTANT ADDITIVES AND POLYETHYLENE

№	Fire resistant composite	Impact resistance, kJ/m ^{2}	Tensile strength, MPa	Bending strength, MPa
1	PE	48	25	20
2	PPA:PE	50	23	19
3	PPK:PE	49	24	19
4	PPM:PE	48	22	18

The results of scanning electron microscopy (SEM) and elemental analysis of polyethylene with fire-resistant fillers of the PPA, PPK, and PPM brands were studied. SEM analysis shows that fire-resistant additives of the PPA, PPK, and PPM brands are evenly distributed over the surface of the polyethylene, in turn, this increases the heat resistance of the polyethylene.

As a result, it was found that adding fire-resistant additives to polyethylene makes it possible to create a more heat-resistant barrier to reduce the intensity of combustion and thereby actively increase the safety of polyethylene (Fig. 3, 4).

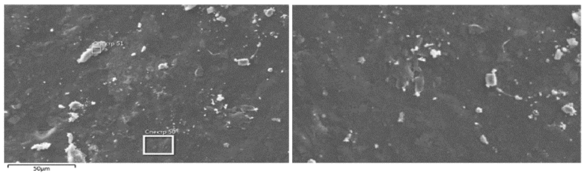

Fig. 3. SEM analysis of modified polyethylene with a fire-resistant additive of the PPA brand

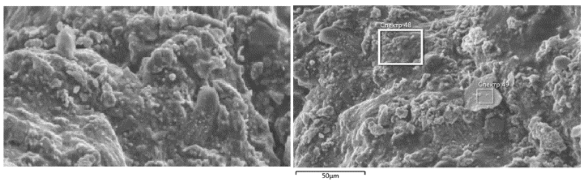

Fig. 4. SEM analysis of modified polyethylene with a fire-resistant additive of the PPK brand

From the obtained images it is clear that on the surface of polyethylene samples to which fire-resistant fillers are added, the filler molecules are distributed evenly. It has been revealed that when fire-resistant fillers of the PPA, PPK, and PPM brands are added to polyethylene, they retain their properties and fire resistance.

To ensure high bond strength between components, it is necessary to completely wet the metal compounds or fiber modifiers, which is achieved by applying a liquid binder to the surface of the fiber, and the energy of the fiber surface must be greater than the surface tension of the liquid matrix. Modification of polymers led to changes in their physical and mechanical properties.

TABLE 5 THE FLAMMABILITY CATEGORY OF VERTICALLY INSTALLED SAMPLES OF COMPOSITES OF MODIFIED PE WITH FIRE-RESISTANT ADDITIVES OF THE PPA, PPK, AND PPM BRANDS ACCORDING TO GOST – 28157

Composite compositions,%	Characteristics
100%, PE (polyethylene F-0220)	PV-2
90%PE + 10%PPA	PV-1
80%PE + 20%PPA	PV-0
70%PE + 30%PPA	PV-0
90%PE + 10%PPK	PV-0
80%PE + 20%PPK	PV-0
70%PE + 30%PPK	PV-0
90%PE + 10%PPM	PV-0
80%PE + 20%PPM	PV-0
70%PE + 30%PPM	PV-0
90% PE + 10% Fire retardant-EcoPirenes (analogue)	PV-1
80% PE + 20% Fire retardant-EcoPirenes (analogue)	PV-0
70% PE + 30% Fire retardant-EcoPirenes (analogue)	PV-0
90%PE + 10%Pirilax (analogue)	PV-1
80%PE + 20%Pirilax (analogue)	PV-0
70%PE + 30%Pirilax (analogue)	PV-0

Fire-resistant modifiers were obtained from composites of the PPA (fly ash, vermiculite, ammonium sulfate, polysulfide rubber), PPM (fly ash, vermiculite, silica, polysulfide rubber), PPM (fly ash, vermiculite, montmorillonite, polysulfide rubber) composites and studied the effect of fire-resistant fillers on polymer materials and mechanisms for increasing their fire resistance. Multifunctional fire-resistant modifiers of the PPA, PPK, and PPM brands were added to the polymer (polyethylene F -0220) up to 10 - 30%, and the most important physicochemical properties of this composite were studied. The experimental results on physical and mechanical properties and fire resistance are given in the table. 5 [11, 12, 13, 14, 15].

The physicochemical and fire-resistant properties of polymer materials with fire-resistant fillers PPA, PPK, and PPM were studied. It has been established that metal groups in the fillers during combustion form a coke layer on the surface of the polymer, which affects thermal conductivity and flame propagation.

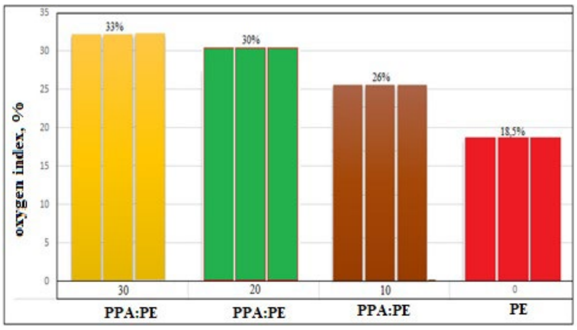

Fig. 5. Results of an analysis of the effect on the oxygen index of polymer materials modified with a fire-resistant additive of the PPA brand

As a result, this leads to a decrease in combustion duration in the polymer material. The introduction of the proposed additives significantly increases the oxygen index of polymer building materials, which is the main criterion for their non-flammability (Fig. 5–6) [5]. The parameters of the oxygen index according to GOST 12.1.044–18 also prove the effectiveness of the proposed additive, which has been studied to achieve the highest value when used in thermoplastics such as polyethylene [11].

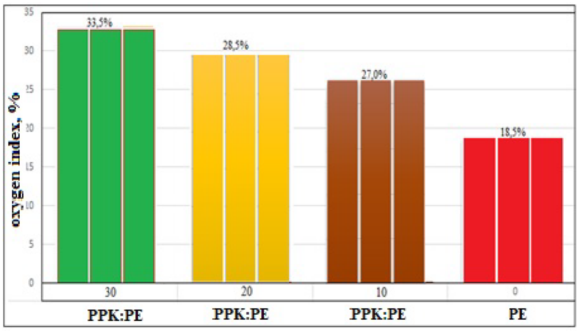

Fig. 6. Results of an analysis of the effect on the oxygen index of polymer materials modified with a fire-resistant additive of the PPK brand

Along with the creation of composites in optimal proportions from polyethylene and polypropylene with fire-resistant compositions based on polysulfide polymer binders, their physical and mechanical properties were also studied. The composition of additives has been fully studied using the most modern physicochemical methods (infrared, ultraviolet, elemental analysis, X-ray diffraction analysis, electron microscope, granulometric analysis, etc.), and their advantages have also been studied.

Fire-resistant compositions of the PPA, PPK, and PPM brands, proposed to increase the fire resistance of polymer compositions, have the same properties as their analogues, and, due to the use of domestic raw materials, they are determined to have higher economic efficiency.

IV. CONCLUSIONS

Based on the results obtained, it was determined that the introduction of additives into the fly ash fillers of the Shirin Thermal Power Plant and Uzmetkombinat JSC, as well as phosphogypsum waste from Maxam-Ammofos JSC, leads to an increase in the thermal stability of polymer materials. The main reason for this is explained by the fact that the composition of fly ash and phosphogypsum waste introduced into the polymer composite contains metal oxides, silicon oxide, and phosphorus compounds, which are resistant to temperatures and have fire-retardant properties.

During experimental tests, it was revealed that from the fire resistance properties of the proposed polymer-containing building materials modified with fire-resistant compositions of the PPA, PPK and PPM brands, consisting of polymer binders, fire-retardant additives and fillers, the oxygen index increased from 18.5 to 33.0%, while the weight loss during thermal oxidation was reduced from 97.5 to 73–75.5%, and the flammability level according to GOST 28157 was changed from PV-2 to PV-0..

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