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SYNTHESIS OF FIRE RETARDANT WITH PHOSPHORUS AND METAL FOR PRESERVATION AND REACH OF REDUCTION OF FLAMMABILITY OF TEXTILE MATERIALS

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Abstract. *In this article, the optimal conditions for synthesizing a new flame retardant based on pentaerythritol, phosphoric acid, and magnesium hydroxide are studied. In this case, the molar ratio of the initial substances was 3.64:1, and the synthesis duration was four hours. After the completion of this process, different (1, 3, and 5%) solutions of magnesium hydroxide were added at a temperature of 90 ºC for 3 hours. The yield of this reaction was 85%. Applying this obtained flame retardant to textile materials increases the fire resistance of textile materials. The composition of synthesized antiperine and antiperine applied textile fabrics was studied by IR spectrum, and the thermal decomposition of antiperine applied fabric was investigated by TGA and DTA analysis, and 100 μm sections of flame retardant treated fabrics were measured by scanning electron microscope. The fire resistance and physical mechanical properties of these materials were studied based on the requirements of GOST 50810-95. The oxygen index of the fabric treated with synthesized flame retardant was 35.6%.*

Keywords*: Flame retardant, phosphoric acid, pentaerythritol, textile materials, TGA and DTA analysis, IR spectrum.*
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Introduction

The production of textile materials and clothes from them is one of the oldest technologies [1]. Textile materials (TM) used in apparel, home improvement, and technology are made from natural and chemical fibers. Light industrial products have a big drawback - they are easily flammable, and therefore, in most cases, their use causes fires [2].

In recent years, increasing the fireproof properties of natural textile materials has become more and more relevant from year to year. This is because these textile materials are distinguished as combustible materials by including factors such as rapid flare, flame spread, and the release of various fumes and gases [3]. Nevertheless, textile materials are widely used in everyday life, in buildings and structures, as transport, and special means of protection [4]. The main characteristic that distinguishes organophosphorus flame retardants from other classes of flame retardants is their secondary properties, which provide additional advantages for processing and obtaining finished products [5, 6]. Therefore, they are often used as processing agents (for example, to reduce the viscosity of polyols, plastisols, and resins), plasticizers (for PVC and acrylates), non-toxic and almost non-volatile

solvents, and wetting agents (in production). The fire resistance of compounds containing phosphorus has been studied in many works [7, 8]. The method of introducing fire extinguishers depends on the type of material to be protected [9]. Of the inorganic antipyrine: aluminum hydroxide, magnesium hydroxide, ammonium polyphosphate, red phosphorus, etc. - about 50% of the world production of flame retardants, about 25% of the world production of flame retardants containing halogen (containing chlorine and bromine) - flame retardants, about 20% of the world production of refractory phosphoric compounds, mainly ether derivatives; inorganic refractory substances can also contain bromine or chlorine atoms. Nitrogen-containing antipyretic substances are used for a limited number of polymers [10]. Refractory substances prevent or suppress combustion processes. Nitrogen-containing compound antipyrine is less commonly used for certain types of materials [11]. However, they are effective flame retardants, with chemical or physical effects on their combustion in a gaseous or condensed phase while preventing the process [12]. Magnesium and aluminum hydroxides are often used as flame retardants[13]. In recent research, interest in antipyrine, which includes combinations of small molecules, is increasing with a new approach that hopes to obtain an important class of refractory materials, including phosphorus, nitrogen, and sulfur[14].

A new Schiff base of melamine, used as a refractory hardener for epoxy resins, is the product of the condensation of 4-hydroxybenzaldehyde with melamine with the addition of 9,10-dihydro-9-OXA-10-phosphafenanthrene 10-oxide (dopo) to the derived imine bond [15], a compound containing phosphorus and nitrogen (2-dimethylaminoethylamphenylhydroxyethylacrylate phosphate) and its oligomer (poly(2-dimethylaminoethylphenylhydroxyethylacrylate phosphate), pdf) [16], acrylate oligomers and phosphorus as raw materials, using functional monomers containing nitrogen and sulfur elements, a fire-resistant coating was obtained for new wood and applied to the wood surface based on the thiol-enol pressing reaction [17].

The main aims of the present study are the obtaining of a new flame retardant based on pentaerythritol, phosphoric acid, and magnesium hydroxide and determine their flame retardant efficiency by the oxygen index method.

Experimental part

Materials. In this research work, substances such as phosphoric acid (85%), pentaerythritol, and magnesium hydroxide were used to synthesize a new composition of antipyrine, and the synthesized new composition of antipyrine was used on cotton fabrics of different sizes (30 cm and 8 cm). All chemical reagents were purchased "chemically pure" from “Merit Chemicals” company. Cotton fabrics were purchased from "Surkhan ZIP TEXTILE" LLC.

Methods. The composition of antiperine synthesized in this research and the cotton fiber used with antiperine was determined by Infrared spectroscopy (IR). IR spectra of initial and synthesized compounds were provided by IR-Fourier spectrometer IR Tpacep-100. Analyzes have been made on Shimadzu (Japan) at the temperature range 100-1000°C, and a scanning electron microscope. The composition of these modified polymers depends on their uniform distribution with the help of an electron microscope from Japan JSM-6460LA (Jeol Interactive Corporation), and it has the following technical indicators: allowed: 4.0 Nm (at 30 KV); voltage acceleration: 0.1 to 4.9 KV (with 10 v step voltage), 5 to 30 KV step voltage (100 V); increase: x8 to X 300,000.

Flame retardant synthesis.

Phosphoric acid (92.24 g, 0.8 mol) was added to the flask, and pentaerythritol (29.95 g, 0.22 mol) was slowly added and heated at 1050 C for 4 h on a magnetic stirrer. After that, magnesium hydroxide (1, 3, 5%) was added to the mass of the obtained substance, and the reaction between these substances continued for 3 hours at a temperature of 900 ºC. As a result of the reaction, a white viscous water-soluble substance was formed. The reaction equation is shown in Figure 1.

The synthesis of oligomeric flame retardants and the optimal modes of their

production (temperature, the ratio of initial components, and their IR spectra) were studied. The efficiency of the reaction was determined to be dependent on temperature and time, and the reaction achieved 85% efficiency at a temperature of 90-100 °C for 3.5 hours.

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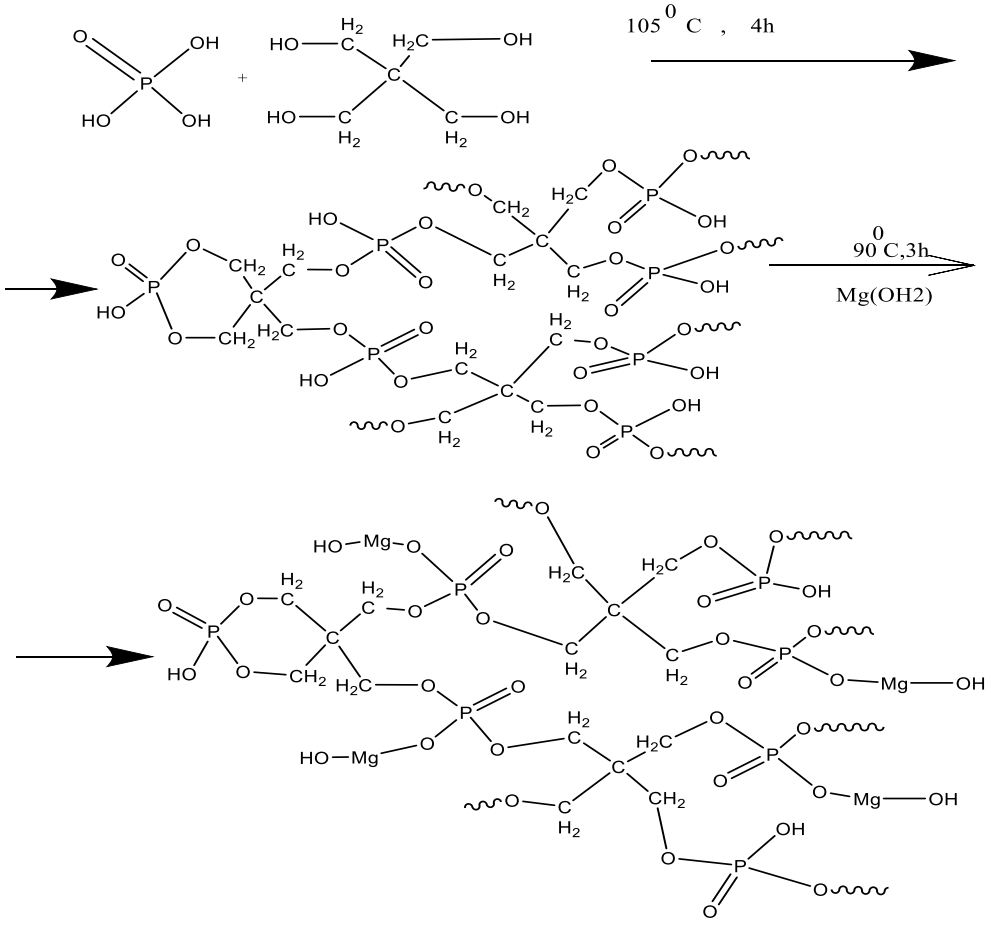


Fig. 1. Reaction equation for obtaining flame retardant

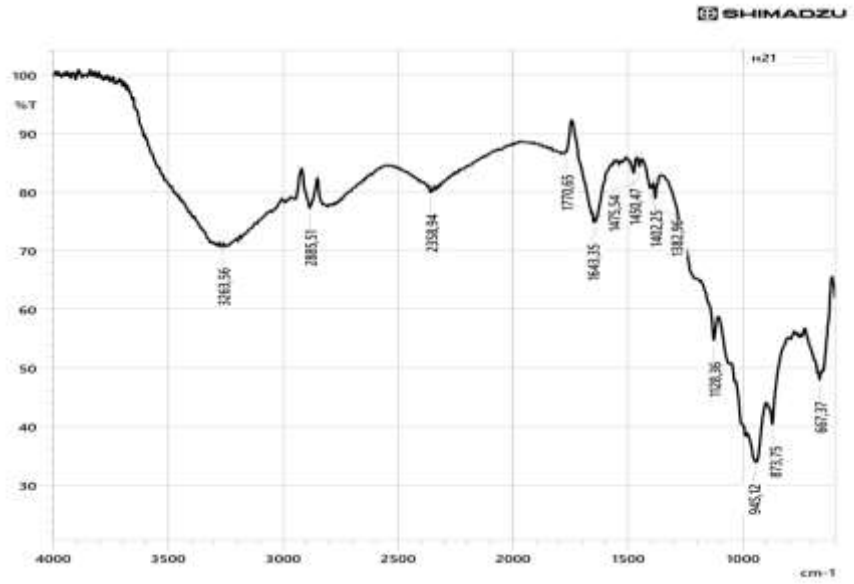


Fig. 2. IR-spectrum of flame retardant

This obtained substance has the following physicochemical properties: appearance, solid, white color, pH 6.2-6.5, Density 31.3 g/cm (25°C), Solubility 40^{C} soluble in water at room temperature.

IR analysis.

The IR spectrum of the obtained flame retardants (Fig. 2) shows that, a broad peak appears near 3500−3000 cm^{-1}, which belongs to -POH for flame retardant; the peak at 2885 cm^{-1} represents the deformation vibration of the -CH_2 bond; the peak at 1128 cm^{-1} and 1410 cm^{-1} corresponds to the -CH_2 bond, respectively; the characteristic absorption peaks of P-O appear at 1382 cm^{-1}; and peaks at 945-667.3 cm^{-1} represent P-0-C and P-O-Mg bonds.

Results and discussion

Flame retardant and fabric modification.

From the analysis of the results of the experiment, special attention is paid to the fact that in the modification of cellulose-based natural textile materials with phosphorus, nitrogen and metal-containing fire retardants, the modification process is partially realized due to the activity of phosphorus and nitrogen bonds with hydroxyl groups.

Thermogravimetric Analysis (TGA and DTA).

In thermogravimetric studies, the mass loss of oligomeric flame retardant, flame retardant-impregnated fabric and non-flame retardant treated fabric obtained as a result of the interaction of phosphoric acid, pentaerythritol and magnesium hydroxide was analyzed in the temperature range of 50-600 ^{o}C. Figure 3 shows 0.05mg of mass loss due to the evaporation of flame retardant water in a 3mg sample in TGA spheres up to 100 ºC (blue line in Fig. 3). According to the results of TGA analysis, 0.10mg of mass was lost in the temperature range of 100-200 ºC, 0.20 mg of high-level mass was lost in the temperature range of 200-300 ^{o}C, and 0.66mg of mass was lost in the temperature range of 300-450 ºC, which is the highest range. level mass loss was analyzed. At temperatures of 450-543 ºC, 50% of the total mass was thermally decomposed and 0.50 mg (i.e. 1.5 mg was decomposed from 3 mg) [18].

DTA analysis curve (red line in Fig. 3) for these samples are mainly characterized by 3 endothermic processes, i.e., heat absorbing. Three endothermic processes are in the temperature range from 238 ^{o}C to 323-378 ^{o}C. Thermal analysis of flame retardants has been found through experiments to have higher thermal stability compared to textile materials and textile materials treated with flame retardants.

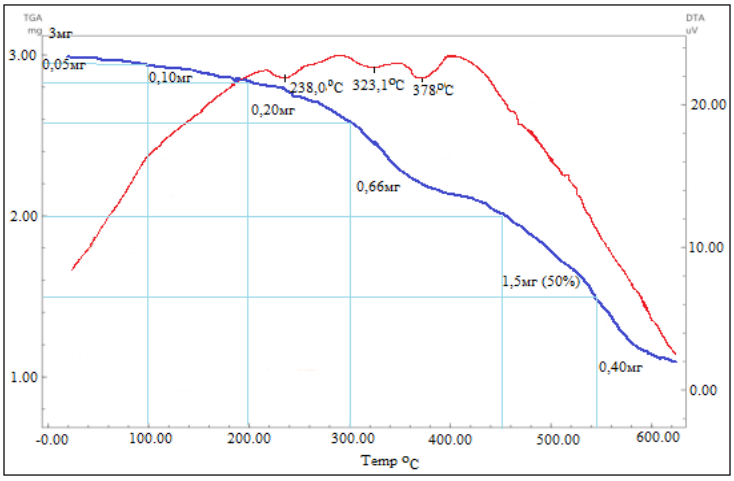


Fig. 3. Thermal analysis study of synthesized flame retardant: thermogravimetric analysis curve (blue); differential thermal analysis curve (red)

Thus, based on experimental data obtained on the kinetics of processes in the temperature range from 50 to 600 ºC, the properties of thermal-oxidative degradation of FM brand flame retardant were studied.

Thermal degradation of textile materials is presented, including TGA and DTA curves (Fig.4). The mass loss of the sample was studied by TGA, and it was found that the sample with a total mass of 2.42 mg emits water vapour at temperatures of 100 ºC. Then, it was investigated that thermal decomposition occurred in the structure of textile materials in the range of 100-200 ºC, resulting in a mass loss of 0.21mg (8.70%). Furthermore, the temperature range of 200-280 ºC produced the highest thermal degradation for these natural textile materials, resulting in 1mg mass loss and 50% total mass degradation. At the next stage, 0.9 mg of mass was lost in the temperature range of 280-400 ºC, and 0.1 mg of mass was lost in the range of 400-600 ºC, and about 0.2-0.3 mg of coke was formed. The DTA analysis found that one endothermic absorption was formed at a temperature of 292 ºC as a result of the thermal effect [19, 20].

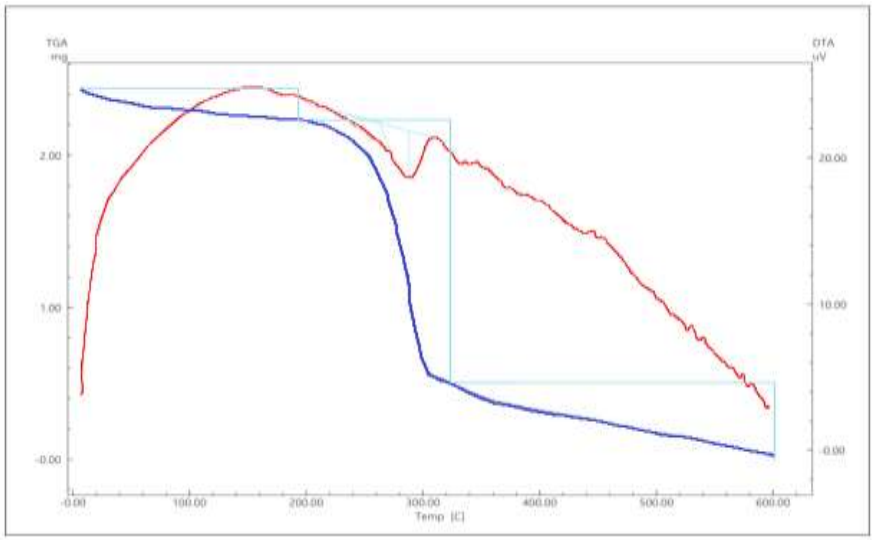


Fig. 4. Thermal analysis study of non-flame retardant treated textile material (TM): thermogravimetric analysis curve (blue); differential thermal analysis curve (red)

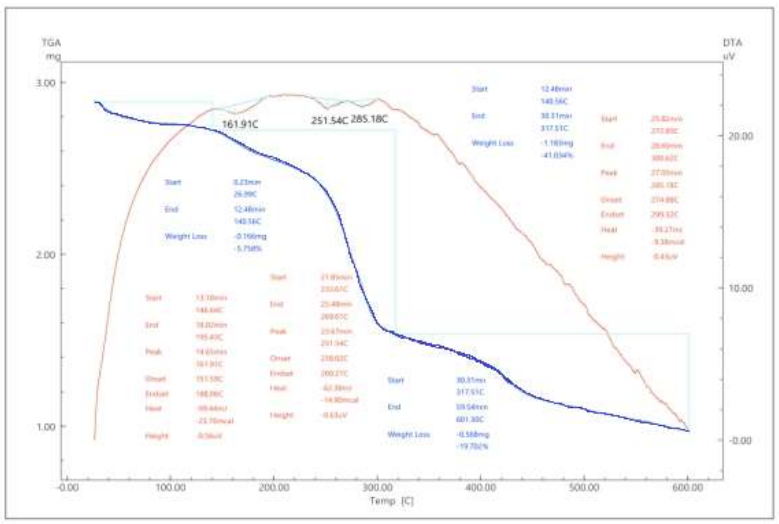


Fig. 5. Thermal analysis study of a sample of FM flame retardant fabric: thermogravimetric analysis curve (blue); differential thermal analysis curve (red)

Thus, based on experimental data obtained on the kinetics of processes in the temperature range from 50 to 600 ^{o}C, the properties of thermal-oxidative degradation of TM were studied.

Derivatogram of a flame retardant fabric sample

Thermal analysis study of a flame retardant fabric sample is presented in Fig. 5.

The TGA curve (Fig. 5) shows that the intense decompositions occur in the temperature range of 26.99-140.56 ºC, temperature of 140.56-317.51 ºC and temperature of 317.51-601.3 ºC. The obtained data indicate that, an active decomposition process takes place in the temperature range of 140.56-317.51 ºC and 41.034% of the total mass is decomposed. Three endothermic absorptions were determined in the DTA curve, at temperatures of 161ºC, and 251-285 ºC. Based on the results obtained from DTA and TGA analysis, kinetic parameters were determined for different temperature ranges of the process. Its advantage is that the kinetic properties of the reaction over the entire temperature range were calculated from a series of measurements and a single sample. The degree of mass loss (*v*m) was determined by the method of graphical differentiation of the TGA curve:

$$vm = Dm/Dt$$

here, Dm—mass loss, mg; Dt - time interval, min.

A detailed analysis of the TGA and DTA curves is given in the Table 1 below.

Table 1. Effect of temperature on weight loss of FM brand flame retardant fabric.

№	dw 2.88	1/T	dw/dт	M.g	Minut	T^0+K
1	2.81	0.0030	0.0269	0.07	2.6	323
2	2.75	0.0026	0.0170	0.13	7.61	373
3	2.71	0.0023	0.0134	0.17	12.61	423
4	2.56	0.0021	0.0181	0.32	17.61	473
5	2.42	0.0019	0.0203	0.46	22.6	523
6	1.70	0.0017	0.0427	1.18	27.61	573
7	1.47	0.0016	0.0432	1.41	32.63	623
8	1.37	0.0014	0.0401	1.51	37.61	673
9	1.20	0.0013	0.0394	1.68	42.61	723
10	1.11	0.0012	0.0371	1.77	47.61	773
11	1.05	0.0012	0.0347	1.83	52.6	823
12	0.99	0.0011	0.0328	1.89	57.6	873
13	0.96	0.0011	0.0322	1.92	59.6	893

The values of the activation energy of this process are listed in Table 2 for a sample of flame retardant dampened fabric of the FM brand.

Table 2. Results of thermal-oxidation analysis of a sample of flame retardant fabric of FM brand

№	dw 2.88	Ln (W_1/W_2)	1/T *10^{-3}
1	2.81	0.0246	3.0
2	2.75	0.0461	2.6
3	2.71	0.0608	2.3
4	2.56	0.1177	2.1
5	2.42	0.1740	1.9
6	1.70	0.5271	1.7
7	1.47	0.6725	1.6
8	1.37	0.7429	1.4
9	1.20	0.8754	1.3
10	1.11	0.9534	1.2

11	1.05	1.0090	1.2
12	0.99	1.0678	1.1
13	0.96	1.0986	1.1

From above thermal studies, it is seen that the main mass loss occurs in the 1st decay in the range of 26.99-140.56 ºC where 5.758% of the main mass is lost. Decomposition 2 occurs at 140.56-382.51 ºC, where 44.034% of the mass is lost. Third decomposition takes place at 317.51-601.3 ºC, during which 19.702% of the mass is lost.

Thus, based on the experimental data obtained on the kinetics of processes in the temperature range from 293 to 943 K, the properties of the thermal-oxidative degradation of the flame retardant fabric of the FM brand were studied.

SEM analysis

As a result of scanning electron microscope analysis of 100 μm sections of flame retardant treated fabrics, it was found that there is a uniform layer between the fibers of modified natural textile materials, and this layer ensures improvement of physical and mechanical properties of the material (Fig. 6).

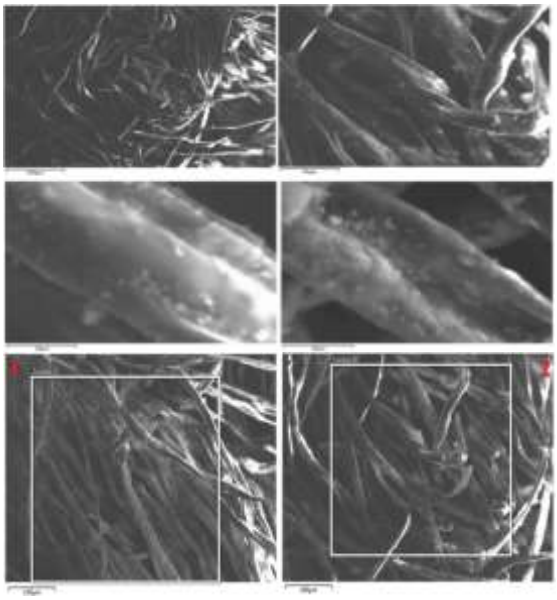


Fig. 6. Scanning electron microscope images of the proposed modifiers

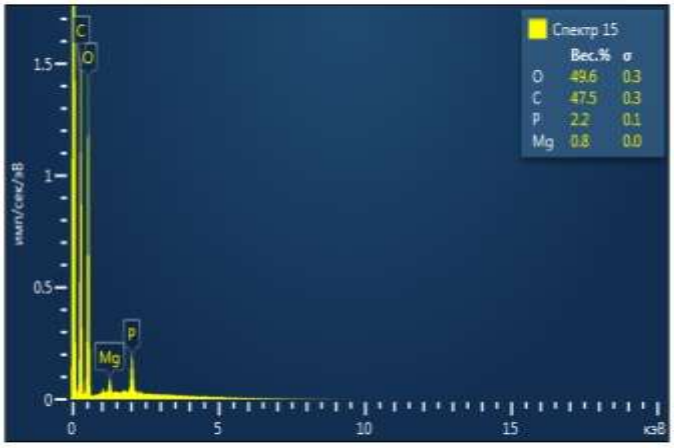
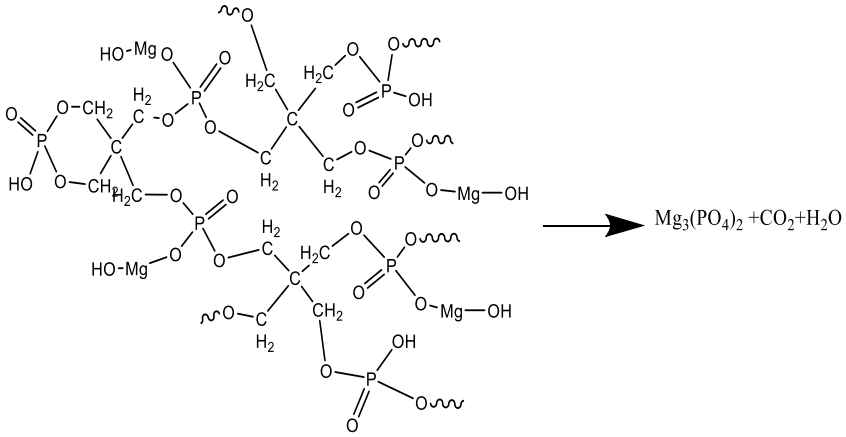


Fig. 7. Elemental analysis of flame retardant-treated fabric

In addition, elemental analysis of chemical substances in modified natural textile materials was carried out. Elemental analysis shows the carbon, oxygen, phosphorus and metals in the flame retardants associated with cellulose (Fig. 7).

Fire resistance mechanism.

The formation of water vapour due to the alkali groups of oligomeric flame retardants under the influence of temperature prevents the temperature from increasing and slows down the ignition. A small amount of metal groups in the flame retardant composition acts as a catalyst in nitrogen and phosphorous mixtures and accelerates the reaction of them with oxygen. In addition, the synergistic effect of metal groups can be theoretically justified by the formation of coke layers based on high-temperature stable salts with carbon residues and phosphorus while increasing the temperature stability of textile materials. The change in flame retardants caused by the high temperature caused by the flame is shown in the reaction in below.



As a result of the synergistic effect of the proposed flame retardants, coke formation increases relatively under the influence of temperature (Fig.8).

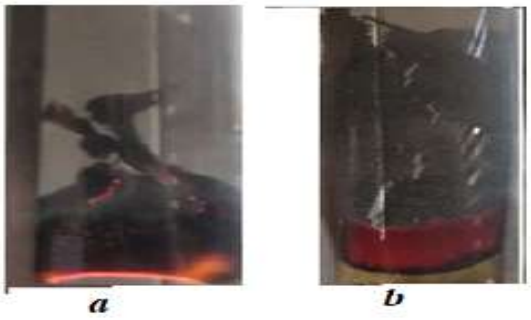


Fig. 8. Temperature-induced coking of (b) and untreated fabrics (a) with the proposed flame retardant

Table 3 shows the dependence of the temperature and time of the ignition of the materials on the temperature treatment of the modified natural textile materials in experimental tests at 120-130 ºC.

Table 3. Temperature and time dependence of combustion of natural textile materials modified with flame retardants

Temperature and time dependence of combustion of natural textile materials modified with flame retardants	Sample burning time, (seconds)	Active continuation of burning of the sample, (sec).	Independent burning time of the sample, (seconds).	Mass loss, %
Unmodified sample	2.8	9.0	53	97.6
Flame retardant treated fabric				
120-130 ºC, 3-4 minut.	9	not burn	not burn	21.5
120-130 ºC, 3-4 minut.	8	not burn	not burn	20.5

We can analyses the optimal time and temperature for the reaction of oligomeric flame retardants containing phosphorus and nitrogen with hydroxyl groups in cellulose during the modification of the processed sample at 120-130 °C for 3-4 minutes with natural textile materials. Therefore, it was taken into account that the modification of the sample at a temperature of 120-130 ºC showed high efficiency of independent burning time and was found to be effective.

Determination of oxygen index.

The flammability of textile materials modified with oligomeric flame retardant was studied based on GOST 12.1.044-89, oxygen index. The oxygen index indicator is specialized in the study of the spontaneous ignition of the material. It was determined through the experimental results that the oxygen index (OI) of the composites formed by modifying with oligomeric flame retardant in the amount of 5% to 20% is 35.6%.

When the obtained results were compared with analogues, it was found that the properties were close to them. That is, the oxygen index of the analogue (kvekadur DM-70) at a concentration of 5-15% was 34.0%, and the oxygen index of the analogue (Antipyren "MS")) at a concentration of 10-15% was 35.2% (Fig. 9). These data are close to the results of our proposed flame retardants and are economically superior.

Fig. 9. Procedure for determination of oxygen index of modified textile materials

Thus, it shows that the development prospects of modified natural textiles are high.

Physico-mechanical properties.

The fire resistance of the proposed flame retardants was increased by adding 5-20% to the content of textile materials, and the fire resistance and physical-mechanical properties of these materials were studied based on the requirements of GOST R 50810-95 (Table 4). Flame retardants were soaked in textile materials for 1-2 minutes at 30-40 ºC, then dehydrated by spinning over a special two-shaft device. Then the modified material was dried at 100-120 ºC, 120-130 ºC and 130-150 ºC for 9-12 minutes. Fire-resistant modifiers that increase the fire resistance of textile materials are analyzed by comparing their properties with the analogues of fire-retardant "MS", DM-70 and Apyrol CEP. The tensile strength (N) in the unmodified material was 200-210 (N). In modified materials, the breaking force (N) was 200-185 (N). These experimental test

procedures were carried out by GOST R 50810-95, fire resistance of textile materials modified with flame retardants.

Table 4. Fire resistance of natural textile materials modified with oligomeric flame retardants and physical and mechanical properties

Amount of oligomer flame retardant, g/l	The length of the area affected by the flame of the textile material, mm			Physical and mechanical properties of textile materials (force at breaking, N)		
	modification temperature, ^{o}C					
	100-120	120-130	130-140	100-120	120-130	130-140
Not modified with flame retardant	218	218	218	215	215	215
Treated with flame retardant (150 g/l)	117	118	122	200	199	189

Thus, the length of the area affected by the flame of the natural textile materials modified with 5-20% flame retardant that we offer is 112-125 mm at a temperature of 100-120 ^{o}C, 108-118 mm at a temperature of 120-130 ^{o}C, and 114-124 mm at a temperature of 130-140 ^{o}C. 123 mm, it was found that all offered oligomeric flame retardants meet the requirements of GOST. In addition, it was found that the physico-mechanical properties (tensile strength, N) of the textile materials modified with the proposed oligomeric flame retardants meet GOST requirements and are competitive with analogues. Based on this, it was studied that the physical and mechanical properties of natural fire-resistant textile materials are similar to unmodified materials. Modification of natural textile materials with oligomeric flame retardants containing phosphorus, nitrogen and metal has been determined by experimental tests to be more effective at a temperature of 120-130 ^{o}C than others.

Conclusion

(I). The flame retardant properties of textile materials treated with phosphorus-containing fire retardants were studied by determining their thermal stability. It was studied that the decrease in the rate of decomposition of cellulose in textile materials from 150 ^{o}C to 350 ^{o}C has a positive effect on the increase in the number of cellulose dehydration reactions.

(II). From the SEM analysis of the composites that formed textile materials with a new type of oligomer flame retardants, it was found that flame retardant molecules are uniformly distributed on the surface of fabric samples treated with flame retardant solution, and when the fabric is treated with a new type of environmentally friendly flame retardant solution, it was found that the fabric properties and fire resistance properties are improved.

(III). Increasing the concentration of phosphorus-retaining oligomeric flame retardant composites up to 20% has been studied to ensure a higher oxygen index in cellulosic textile materials. In addition, it was determined that modified refractory textile materials produce coke and lose mass, and as a result, mass loss of 97.6% for textile materials and 21.5-20.5% for modified materials were studied.

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Authors' Declaration

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are ours.

- Ethical Clearance: The project was approved by the local ethical committee in the Termez branch of Tashkent Medical Academy.

Authors' Contribution Statement

M.N.Sh: Writing – Original Draft. N.F.N: Reviewing and editing paper. Kh.Z.N: Reviewing and editing the paper. A.A: Software, Validation. B.MI and A.I.I: Writing – Original Draft, Conceptualization, Investigation, Visualisation.

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TEKSTİL MATERALLARININ ALIŞQANLILIĞININ QARŞISININ ALINMASI VƏ YA AZALDILMASI ÜÇÜN FOSFOR VƏ METAL TƏRKİBLİ YANĞINA DAVAMLI MATERİALLARIN SİNTEZİ

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Xülasə. Məqalədə pentaeritritol, fosfor turşusu və maqnezium hidroksid əsasında yeni yanğına davamlı materialların sintezi üçün optimal şərtlər araşdırılmışdır. İlkin maddələrin molyar nisbəti 3.64:1, sintez müddəti isə dörd saat təşkil etmişdir. Bu proses başa çatdıqdan sonra 90 ºC –də, 3 saat ərzində maqnezium hidroksidinin müxtəlif qatılıqlı (1, 3, 5%) məhlulları əlavə edilmişdir. Bu reaksiyanın çıxımı 85% təşkil etmişdir. Alınmış oddan-qoruyucu maddələrin tekstil materiallarına

tətbiqi onların odadavamlılığını artırır. Alınmış antiperin və antiperin tekstil parçalarının tərkibi IQ spektroskopiya ilə, termal parçalanması isə termal qravimetrik analiz (TQA) və differensial termiki analiz (DTA) üsulları ilə tədqiq edilmişdir. Yanğına davamlı maddələrlə örtülmüş tekstil parçaların 100 mkm ölçülü kəsikləri skanedilmiş elektron mikroskopiya üsulu ilə öyrənilmişdir. Bu materialların yanğına davamlılığı və fiziki mexaniki xüsusiyyətləri GOST 50810-95 tələbləri əsasında öyrənilmişdir. Sintez edilmiş yanğınadavamlı materiallarla işlənmiş parçaların oksigen indeksi 35.6% təşkil etmişdir.

Açar sözlər: yanğına davamlı materiallar, fosfat turşusu, pentaeritritol, tekstil materialları, TGA və DTA üsulları, İQ spektri.

СИНТЕЗ ФОСФОР- И МЕТАЛЛ-СОДЕРЖАЩИХ ОГНЕЗАЩИТНЫХ МАТЕРИАЛОВ ДЛЯ ПРЕДОТВРАЩЕНИЯ И СНИЖЕНИЯ ГОРЮЧЕСТИ ТЕКСТИЛЬНЫХ МАТЕРИАЛОВ

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Резюме: В данной статье изучены оптимальные условия синтеза нового антипирена на основе пентаэритрита, фосфорной кислоты и гидроксида магния. При этом мольное соотношение исходных веществ составляло 3,64:1, а продолжительность синтеза – четыре часа. После завершения этого процесса добавляли разные (1, 3 и 5%) растворы гидроксида магния при температуре 90°C в течение 3 часов. Выход этой реакции составил 85%. Нанесение полученного антипирена на текстильные материалы повышает огнестойкость текстильных материалов. Состав синтезированных антипериновых и антипериновых накладных текстильных тканей изучали методом ИК-спектроскопии, а термическое разложение антипериновых накладных тканей исследовали методами термогравиметрического анализа (ТГА) и дифференциально-термического анализа (ДТА)анализа. Срезы обработанных огнезащитным составом тканей толщиной 100 мкм изучали с помощью сканирующего электронного микроскопа. Огнестойкость и физико-механические свойства этих материалов исследовали на основании требований ГОСТ 50810-95. Кислородный индекс ткани, обработанной синтезированным антипиреном, составил 35.6 %.

Ключевые слова: антипирен, фосфорная кислота, пентаэритрит, текстильные материалы, анализ ТГА и ДТА, ИК-спектр.