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FIRE RETARDANT OF CELLULOSE TEXTILE MATERIALS BASED ON SOL-GEL COMPOSITION

Abstract. The article describes using the tetroetokisilane, thiourea and fire retardant to impart fireproof properties to cellulose textile materials. The influence of concentration of the initial components, temperature and duration of heat treatment on retardant properties was studied. The morphology of the surface of fibers of textile materials and elemental microanalysis of the fiber surface structure is studied.

Key words: cellulosic textile materials, fire resistance, sol-gel, tetro-ethoxysilane, thiourea, fire retardant.

Introduction. At present, a large number of easily combustible fabric materials, representing a significant fire hazard, are used in living quarters. Investigations of the causes of fires show that the interior elements of textiles (curtains, upholstery fabrics, carpeting) not only contribute to the rapid spread of fire, but also are a source of a number of asphyxiating gases in a fire. Reduction of fire danger is possible with the help of measures carried out by chemical methods of fire protection of soft and hard fabric materials. Preventing the development of fire, chemical means of fire protection facilitate firefighting, and in some cases exclude the possibility of a fire [1-3].

The actual problem of modern chemistry of polymers is the creation of materials of low combustibility, as well as the development of specific flame retardants, flame retardants, which in addition to high efficiency should show good compatibility with used polymers and meet environmental safety requirements [4-5].

The purpose of this study is to produce cellulose materials with flame retardant properties using sol gel technology.

Experimental part. The main component for the preparation of the sol is tetroethoxysilane, water and ethyl alcohol solvent, acetic acid hydrolysis catalyst, article 1030 cotton fabric with a surface density of 147g/m^2.

Tetroethoxysilane - is a volatile transparent colorless liquid with a characteristic spicy-sweetish, somewhat similar to an alcohol smell. It is well mixed with organic solvents, water, aqueous solutions of acids.

Ethyl alcohol - is a monohydric alcohol, under standard conditions a volatile, flammable, colorless transparent liquid.

Thiourea-CS $(NH_2)_2$ - thioglyclic acid diamide, white crystals of bitter taste, melting point 180-182 °C (with rapid heating, with slow decomposition); moderately soluble in water, methanol, pyridine, well in 50% aqueous pyridine.

Before carrying out the experimental work, the cotton bleached fabric of the coarse band art. 1030 was washed with a 2% non-ionic detergent at 40° C for 20 minutes and then washed with distilled water and dried. After drying, the desiccator was kept above dehydrated $CaCl_2$ for at least 24 hours to determine the exact sample weighed [6].

Methods of research. A sample of cotton fabric measuring 200 × 200 mm after determining the exact weight on an analytical balance was subjected to impregnation in a bath with tetroetoksisilan (at 1: 1: 8) for 1 minute, spin drying was 90%, followed by drying at 75 – 85 ^{0}C for 8 - 10 min, then treated fabric was subjected to heat treatment at 110 ^{0}C, 130 ^{0}C, 150 ^{0}C for 2 minutes, followed by rinsing in large amount of distilled water and then drying.

In the second stage after treatment with tetraethoxysilane, the samples were impregnated in aqueous solution of fire retardant and thiourea for 1 minute. After extraction 90%, drying at 75 ^{0}C for 3 min in an oven, followed by washing in distilled water and dried at room temperature.

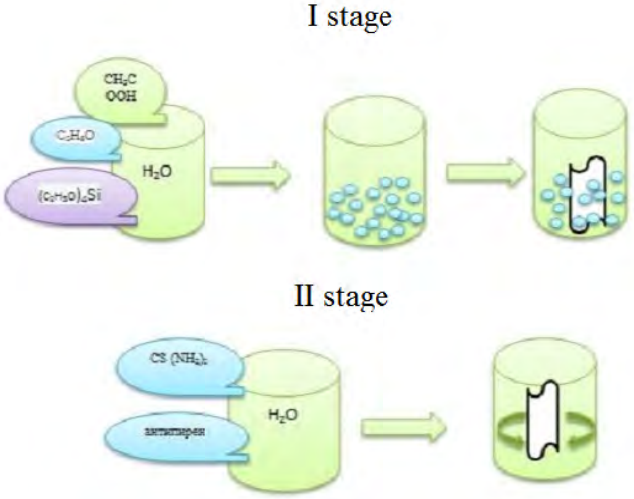

Figure 1 – Scheme for the production of fire-resistant composition

Results and discussion. The results of the test of flame retardant efficiency, tensile load and stability of impregnation after 5 washes are shown in table 1. As can be seen from the table, the sol-gel composition used provides a high degree of fire resistance and does not affect the physical and mechonical characteristics of cellulosic textile materials. This indicates the presence of SiO_2 coating and fixation of the fire retardant.

In order to fully elucidate the mechanism of interaction of the fire-resistant sol-gel composition with cellulose of cotton fiber, the IR spectra of samples of initial and processed tissues were studied in the work (figure 2). When analyzing the spectra shown in figure 2 and in table 2, it can be seen that the spectra of the cellulose treated with this composition compared to the original cellulose have

Table 1 – Results of the test of flame retardant efficiency and breaking load of treated cellulose materials

№	Concentration of substances, g/L			Length of the char section, mm			Breaking load, N			Length of the charred area (after 5 washes), mm		
	$(C_2H_5O)_4Si$	$CS(NH_2)_2$	flame etardant	Heat treatment temperature, ° C								
				110	130	150	110	130	150	110	130	150
1	Source sample			220	220	220	202	202	202	220	220	220
2	100	60	200	110	123	124	203	199	198	135	182	166
3			300	102	109	113	201	202	199	121	139	165
4			400	95	105	112	205	200	196	112	139	124

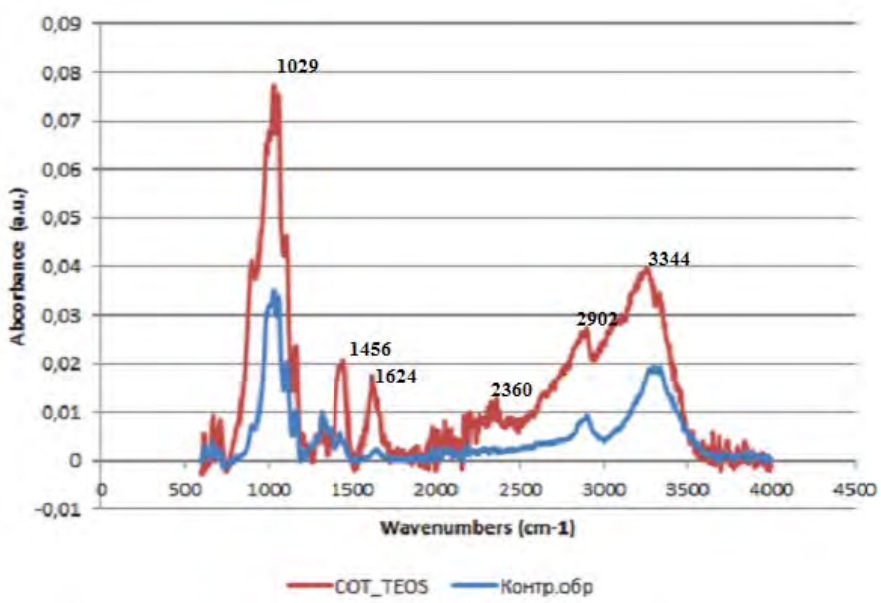

Figure 2 – IR spectra of a control sample and treated cellulose fibers with tetroethoxysilane

Table 2 – Basic vibration frequencies of processed cellulose textile materials

The oscillation frequencies (cm^{-1})		Oscillatory modes
Counter. arr.	Arr. with TEOS	
3336	3344	(NH_2)
2904	2902	(C-H)
	2360	(P-H)
	1624	(N-H)
	1456-1392	(C-N)
1031	1029	(Si-O- Si)

undergone significant changes. In the IR spectrum of the processed sample, all absorption bands are retained, characteristic of untreated cotton fiber. The absorption bands of the processed sample indicate the presence of Si-O-Si bond bonds in the 1029 and 1031 cm^{-1} regions, NH groups in the 1624 cm^{-1} region, and also the P-H groups in the 2360 cm^{-1} region, CN groups in the regions 1392-1456 cm^{-1}.

Based on the foregoing, it can be concluded that the interaction of the composition with the macromolecules of cellulose leads to a significant change in the absorption bands of the processed samples. Thus, taking into account the studies carried out, it can be concluded that when treating cellulose with this composition, chemical bonds are formed between the macromolecules of cellulose and sizing agents.

Investigation of the morphology of the surface of the fibers of textile materials and elemental microanalysis of the fiber surface structure used an autoemission scanning electron microscope JSM-6490LA (Japan) with the X-ray spectral analyzer system JED-2300 Analysis Station. Based on the result of scanning electron microscopy, a polymeric layer is formed on the surface of the treated tissues in the form of an oxide-silicon matrix (figure 3). Figure 3 clearly shows that fire retardant particles are present on the treated fiber on the surface of the fiber, fire retardant plates of various shapes are noticeable.

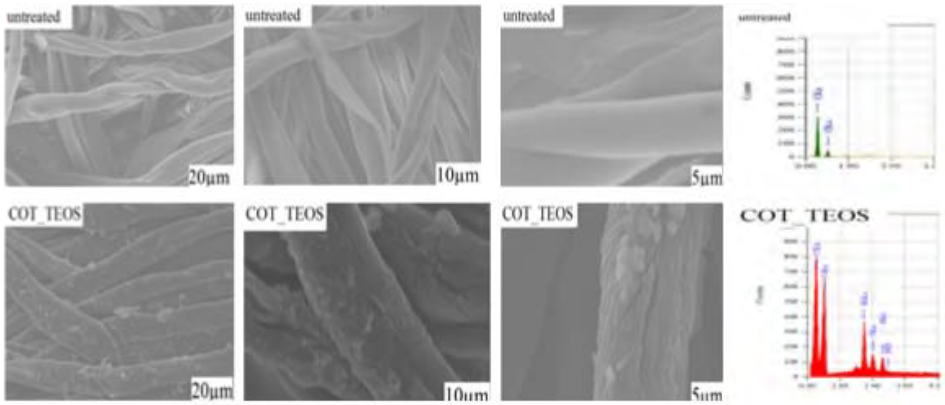

Figure 3 – Electron microscopic images of cotton fabric and treated
with flame retardant composition with different resolution and energy dispersive microanalysis

According to the energy-dispersive microanalysis, the figure shows the presence of carbon and oxygen, which is natural for cellulosic materials. After processing, particles of Si, P, S are formed on the surface of the treated fabric and are distributed rather unevenly.

Conclusion. Analysis of the results of the study allows us to draw the following conclusions:

It is shown that this method of impregnating cellulosic textile materials in a precursor, followed by drying and heat treatment and flame retardant, makes it possible to obtain a silica coating with a fixed flame retardant of high degree.

The IR spectra data confirm the appearance of a chemical bond between the cellulose macromolecules and the flame-resistant sol-gel composition.

Using the method of electron-scanning microscopy, it has been established that the treatment of tissues with the developed compositions leads to a change in the morphology of the surface of the fibers.

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Резюме

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ОГНЕСТОЙКОСТЬ ЦЕЛЛЮЛОЗНЫХ ТЕКСТИЛЬНЫХ МАТЕРИАЛОВ НА ОСНОВЕ ЗОЛЬ-ГЕЛЬ КОМПОЗИЦИИ

В статье приведен новый способ разработки огнестойких целлюлозных текстильных материалов золь-гель методом с применением тетроэтоксисилана. Основным результатом проведенного исследования является разработанная экологически безопасная технология получения огнестойкой золь-гель композиции, которая протекает в две стадии. На первой стадии текстильный материал пропитывается в прекурсоре в течение минуты, а на второй – в ванне с антипиреном. Исследовано влияние параметров обработки на эффективность огнезащитности, на физико-механические свойства ткани и на стойкость огнестойкой золь-гель композиции после 5-ти стирок. Выявлено, что наилучшей эффективностью огнезащитности обладают образцы, обработанные при концентрации тетроэтоксисилана равной 100 г/л и прошедшие термическую обработку при 150 °С. Данные ИК-спектров подтверждают появление химической связи между макромолекулами целлюлозы и огнестойкой золь-гель композиции. Также применен метод электронной микроско-

пии, результаты которой доказывают наличие функционального покрытия и фиксации антипирена в его объеме. Результаты исследования могут быть применены в отделочном производстве текстильных целлюлозосодержащих материалов.

Ключевые слова: целлюлозные текстильные материалы, огнестойкость, золь-гель, тетроэтокисилан, тиомочевина, антипирен.

Резюме

Е. Такей, Б. Р. Таусарова, А. Буркитбай

ЗОЛЬ-ГЕЛЬ КОМПОЗИЦИЯ НЕГІЗІНДЕГІ
ЦЕЛЛЮЛОЗАЛЫ ТЕКСТИЛЬ МАТЕРИАЛДАРЫНЫҢ ОТҚА ТӨЗІМДІЛІГІ

Мақалада тетроэтоксисилан қолдана отырып золь-гель әдісімен отқа төзімді целлюлозалы текстиль материалын алу технологиясы сипатталған. Зерттеу жұмысының негізгі нәтижесі болып экологиялық қауіпсіз отқа төзімді золь-гель алу әдісі болып табылады. Бұл әдіс екі деңгейде жүзеге асады, біріншісі: текстиль материалының үлгісін золь прекурсорында сіңіріп, екінші деңгейде антипиренмен өңдейміз. Өңдеу параметрлерінің отқа төзімділік пен үзілу жүктемесіне, сонымен қатар өңдеу композициясының тұрақтылығына әсері зерделенді. Ең жақсы нәтижелерді тетроэтоксисиланның 100 г/л мөлшеріндегі 150 °С термоөңдеу өткен текстиль материалдары көрсетті. Сонымен қатар зерделенген ИК-спектр нәтижесі бойынша текстиль материалындағы целлюлоза мен отқа төзімді золь-гель композиция арасындағы химиялық байланыстың бар екенін көрсетті. Қолданылған электронды микроскоп әдісінің нәтижесі бойынша өңделген текстиль материалының бетінде функционалды жабын мен антипиреннің берік бекітілгенін көрсетті. Зерттеу нәтижелерін целлюлозалы текстиль материалдарын өңдеу өндірісінде қолдануға болады.

Түйін сөздер: целлюлозалы текстиль материалдары, отқа төзімділік, золь-гель, тетроэтоксисилан, тиомочевина, антипирен.