ЕҢБЕК ҚЫЗЫЛ ТУ ОРДЕНДІ
«Ә. Б. БЕКТҰРОВ АТЫНДАҒЫ
ХИМИЯ ҒЫЛЫМДАРЫ ИНСТИТУТЫ»
АКЦИОНЕРЛІК ҚОҒАМЫ

Қазақстанның Химия Журналы

Химический Журнал Казахстана

Chemical journal of Kazakhstan

АКЦИОНЕРНОЕ ОБЩЕСТВО
ОРДЕНА ТРУДОВОГО КРАСНОГО ЗНАМЕНИ
«ИНСТИТУТ ХИМИЧЕСКИХ НАУК
им. А. Б. БЕКТУРОВА»

4 (64)

ОКТЯБРЬ – ДЕКАБРЬ 2018 г.

ИЗДАЕТСЯ С ОКТЯБРЯ 2003 ГОДА

ВЫХОДИТ 4 РАЗА В ГОД

АЛМАТЫ
2018

UDC 677.027.62

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SOL-GEL COMPOSITION ON THE BASIS OF SODIUM SILICATE AND AMMONIUM POLYPHOSPHATE FOR OBTAINING FIRE-RESISTANT CELLULOSE TEXTILE MATERIALS

Abstract. The article gives a new method for producing fire-resistant cellulose textile materials using the sol-gel method, where the sol-gel precursor is sodium silicate. A new sol-gel method is proposed for the formation on a fiber of a functional coating based on sodium silicate and nitrogen-phosphorus-containing compounds. The effect of processing parameters on the fire resistance and on the discontinuous characteristics of the obtained materials was studied. The method of scanning electron microscopy was used to study the structure of the surface of treated fibers. The thermal stability and index of the limiting oxygen index of control and processed cellulose textile materials with a sol-gel composition were studied.

Key words: fire resistance, sol-gel, sodium silicate, cellulose textile materials.

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].

The problem of imparting fireproof properties to textile materials of various nature and purpose has become increasingly important in recent years [2]. This is due to the fact that they are a serious source of danger during fires, it is easily ignited, promotes the spread of the flame and, when burning, emits a large amount of smoke and gases. One of the urgent tasks is the replacement of traditional technologies of making fireproof properties of textile materials cheaper and environmentally safe [3]. With the traditional technology of final finishing of cellulose cotton textile materials, it is possible to impart certain properties to them by modification with special dressing agents. In connection with this, it is important to develop new methods for imparting fire resistance to textile matrials, which would significantly increase the stability of the modifying effects. To modify cellulosic materials and impart technical properties to textile materials, a sol-gel method was used in the work [4]. The main advantage of the sol-gel method over others is that it allows you to control the structure of the resulting materials, the particle size, the pore volume and volume, the surface area of the films, in order

to obtain a material with desired properties. This method does not require unique equipment and expensive initial reagents and therefore is a relatively cheap method of synthesis. Coatings obtained by the sol-gel method are a suitable tool for the modification of a large number of materials, such as glass, paper, synthetic polymers, wood, metal and textiles [5].

The purpose of this study is to produce textile materials with flame retardant properties using sol gel technology based on sodium silicate and ammonium polyphosphate.

Objects and methods of research. As an object of study in the work was: cotton fabric article 1030, sodium silicate, ammonium polyphosphate, thiourea. Samples of cotton linen weave are treated with an aqueous solution of sodium silicate, the hydrolysis catalyst is 70% CH3COOH impregnation at 25-300C for 1 minute, then spinning on a two-shaft plus with a degree of 90%. The fabric was then dried at a temperature of 750C for 8-10 minutes. Next, the treated fabric was heat treated at 110, 130, 150 °C for 1 minute, in the second stage impregnated with an aqueous solution of ammonium polyphosphate and thiourea, after pressing 90%, drying at 75 °C for 3 minutes in a oven, followed by washing in distilled water and dried at room temperature.

Characterization techniques. Tests of fire-retardant effectiveness of the developed compositions were carried out in accordance with GOST R 50810-95, which establishes a method for determining the ability of textile materials (fabrics, non-woven fabrics) to resist ignition, sustainable burning, and also to assess their fire retardancy (table 1). The standard is applied to all combustible decorative textile materials supplied to the consumer.

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		
		Na_2SiO_3	CS $(NH_2)_2$	flamee-tardant	Heat treatment temperature, °C					
					110	130	150	110	130	150
1		Control sample			220	220	220	202	202	202
2	cot_si-5	50	60	200	116	144	136	205	206	219
3				300	109	115	128	212	209	222
4				400	98	111	107	218	212	219
5	cot_si-10	100		200	123	138	137	206	214	223
6				300	114	110	113	209	206	217
7				400	115	131	104	217	217	219
8	cot_si-15	150		200	115	108	124	220	209	209
9				300	94	90	90	219	211	211
10				400	90	99	94	223	216	219

The surface morphology of the treated samples was studied an auto-emission 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 (figure 1).

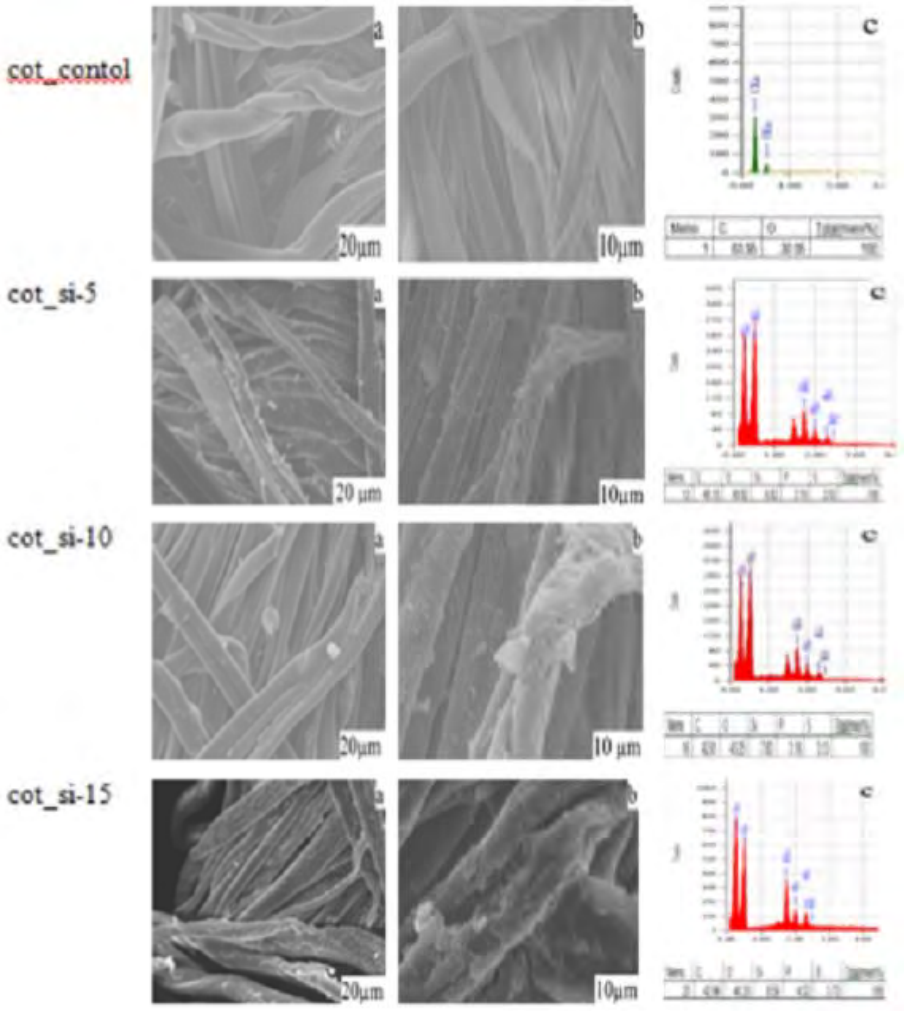

Figure 1 – Electron microscopic images of cotton fabric and treated with flame retardant composition with different resolution and energy dispersive microanalysis

The thermal stability of the fabrics was evaluated by thermogravimetric (TG) analyses from 50 to 600 ^{0}C with a heating rate of 10 ^{0}C/min, both in nitrogen and in air (figure 2 and table 3). To this aim, a TAQ500 thermogravimetric balance was used, placing the samples in open alumina pans (ca. 10 mg). The experimental error was 0.5% on the weight and 1 °C on the temperature.

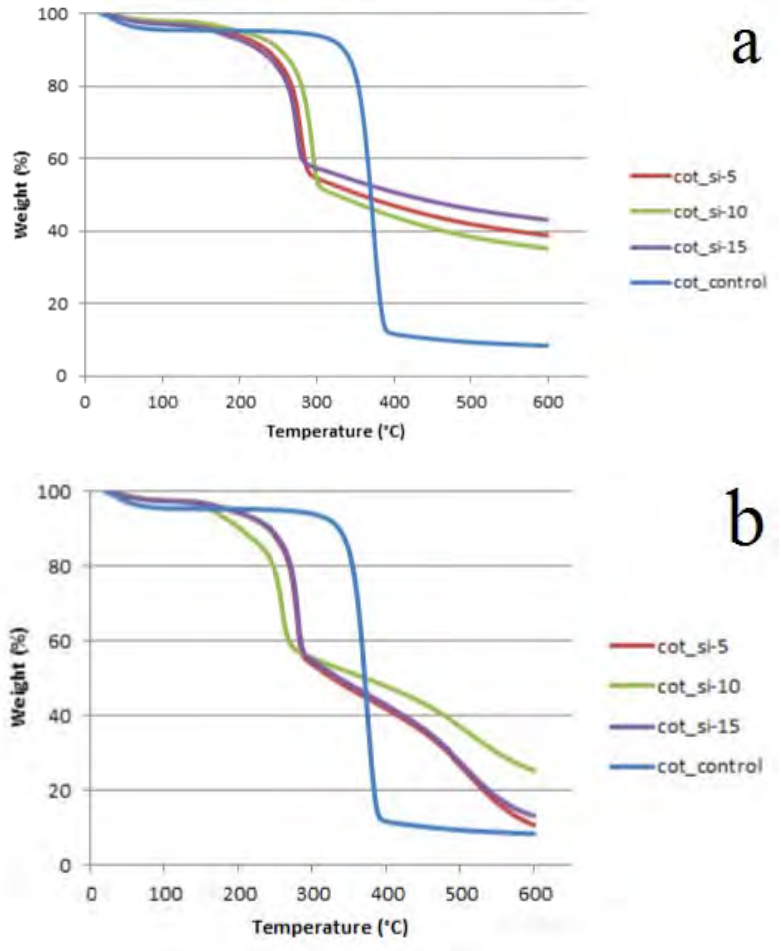

Figure 2 – Comparison between TG curves of cotton control and sol-gel treated cotton fabrics in nitrogen and air

Table 2 – Average elemental composition of untreated and modified cotton fabrics, obtained on the basis of the results of energy-dispersive microanalysis

#	Sample	Mass fraction, %				
		C	O	Si	P	S
1	cot_control	54.95	45.05			
2	cot_si-5	48.15	40.82	6.82	2.18	2.03
3	cot_si-10	42.81	43.25	7.63	3.18	3.13
4	cot_si-15	42.96	40.20	8.59	4.52	3.73

The determination of the Limiting Oxygen Index (LOI) is the minimum concentration of oxygen, expressed as a percentage, that will support combustion of a polymer. It is measured by passing a mixture of oxygen and nitrogen over a burning specimen, and reducing the oxygen level until a critical level is reached. The determination of the Limiting Oxygen Index (LOI) (table 4) was carried out according to ASTM D2863-09 standard, using an OXINDEX apparatus, built by MÜKI.

Results and discussion. The results of the test of flame retardant efficiencyare 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.

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 1). Figure 1 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.

According to electron-scanning microscopy and conducted energy-dispersive microanalysis (table 2), pure cotton fabric contains C-69.95% O-30.05%. After the modification, the particles formed on the surface of the treated fabric: Si – 6.82%, P – 2.18%, S – 2.03% which are distributed rather unevenly. It is shown that with the increase in the concentration of the flame retardant in the modified composition in the treated samples, the content of phosphorus and sulfur increases to 8.59% and 3.73%, respectively.

The thermal and thermal oxidation stability of sol-gel treated samples was evaluated by thermogravimetric analysis and compared with the thermal stability of untreated cotton. Tables 3 collected data on nitrogen and air; build the TG curves of the samples in both atmospheres. As already demonstrated, thermal degradation of a honeycombton in nitrogen occurs in one stage during which maximum weight loss is registered in 390 ºC.

Table 3 – TGA data of untreated and sol-gel treated cotton fabrics in nitrogen and air

Sample	nitrogen				air			
	T_{oneset} (^{0}C)	T_{max} (^{0}C)	Residue at T_{max} (%)	Residue at 600 ^{0}C (%)	T_{oneset} (^{0}C)	T_{max} (^{0}C)	Residue at T_{max} (%)	Residue at 600 ^{0}C (%)
cot_control	345	390	10	8	350	390	12	10
cot_si-5	275	300	55	39	290	300	55	12
cot_si-10	295	310	50	35	250	290	57	23
cot_si-15	275	295	58	44	290	300	55	13

The limiting oxygen index was also studied, the results of which are given in Table 4. As shown in the table, the untreated tissue has a low oxygen index, compared to the processed samples with sol-gel composition.

Table 4 – LOI data of untreated and sol-gel treated cotton fabrics

#	Sample	LOI(%)
1	cot_control	19
2	cot_si-5	34
3	cot_si-10	35
4	cot_si-15	36

Conclusion. A sol-gel composition based on sodium silicate and ammonium polyphosphate was first developed to impart flame retardant properties to cellulosic textile materials. The processed materials of the present composition have a high fire resistance and physical-mechanical properties. The processing parameters of cellulose materials have been determined. It is shown that the heat resistance and oxygen index of the treated textile samples are higher compared to the untreated ones.

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Резюме

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ОТҚА ТӨЗІМДІ ЦЕЛЛЮЛОЗАЛЫ ТЕКСТИЛЬ МАТЕРИАЛДАРЫН НАТРИЙ СИЛИКАТЕ МЕН ПОЛИФОСФАТ АММОНИЯ НЕГІЗІНДЕ ЗОЛ-ГЕЛЬ КОМПОЗИМЕН АЛУ

Мақалада натрий силикатын 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. Қолданылған электронды микроскоп әдісінің нәтижесі бойынша өңделген текстиль материалының бетінде функционалды жабын мен антипиреннің берік бекітілгенін көрсетті. Сонымен қатар пайда болған жабынның текстиль материалына зияны жоқ екені физико-механикалық көрсеткіштерді зерделеу барысында анықталды. Зерттеу нәтижелері толыққанды болу үшін текстиль материалдарының термотұрақтылығы мен оттегі индексінің мөлшнрі анықталды. Зерттеу нәтижелерін целлюлозалы текстиль материалдарын өңдеу өндірісінде қолдануға болады.

Түйін сөздер: целлюлозалы текстиль материалдары, отқа төзімділік, золь-гель, натрий силикаты, антипирен.

Резюме

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ЗОЛЬ-ГЕЛЬ КОМПОЗИЦИЯ НА ОСНОВЕ СИЛИКАТА НАТРИЯ И ПОЛИФОСФАТА АММОНИЯ ДЛЯ ПОЛУЧЕНИЯ ОГНЕСТОЙКИХ ЦЕЛЛЮЛОЗНЫХ ТЕКСТИЛЬНЫХ МАТЕРИАЛОВ

В статье приведен новый способ разработки огнестойких целлюлозных текстильных материалов золь-гель методом на основе силиката натрия. Основным результатом проведенного исследования является разработанная экологически безопасная технология получения огнестойкой золь-гель композиции, которая протекает в двух стадиях. На первой стадии текстильный материал пропитывается в прекурсоре в течение 1 мин, а на второй стадии пропитывается в ванне с антипиреном. Исследовано влияние параметров обработки на эффективность огнезащитности на физико-механические свойства ткани. Применен метод электронной микроскопии с энергодисперсионным анализом на элементный состав необработанного и обработанных текстильных материалов, результаты которого доказывают наличие функционального покрытия и фиксации антипирена в его объеме. Так же методом термогравиметрического анализа изучена термостойкость необработанного и обработанных целлюлозных текстильных материалов. Результаты исследования могут быть применены в отделочном производстве текстильных целлюлозосодержащих материалов.

Ключевые слова: огнестойкость, золь-гель, силикат натрия, целлюлозные текстильные материалы, антипирен.