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CREATION OF A HEAT-RESISTANT POLYMER COMPOSITION BASED ON HYDROLYZED POLYACRYLINITRILE AND ORGANIC SILICON COMPOUNDS CHEMICAL METHODS

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Abstract

An effective method for producing fire-resistant and heat-insulating highly dispersed fillers based on HIPAN and multifunctional components, and the study of the resulting polymer composition through viscosity changes. The properties and structure of the substance were analyzed using differential thermal analysis and IR spectroscopy. The physical state, structure, and chemical composition of the polymer were investigated under the influence of external fields (ultrasound, heat) and chemical reagents, and the given thermal and physical parameters, as well as the average molecular weight, of fire-resistant compositions based on HIPAN were studied.

Keywords

Polymer, HIPAN, hydrolyzed polyacrylonitrile, tetraethoxysilane, organosilicon compound, thermostability, crosslinking, viscosity, solution, thermal stability.

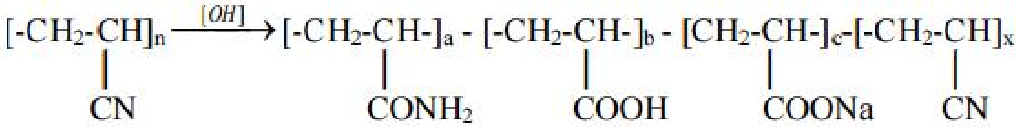
Introduction. Synthetic and natural polymers are an essential part of modern life and are currently used in almost all fields. Today, traditional synthetic polymer materials and natural polymer materials are widely utilized. However, a drawback of such polymer materials is their higher flammability compared to other materials. Therefore, for many end products containing polymers (e.g., cables, carpets, furniture cabinets, various fabrics, etc.), there is a need to create and use new compositions of heat-insulating polymers with a high degree of fire resistance to ensure public safety from fires. Obtaining highly dispersed fire-resistant and heat-insulating coatings based on organosilicon compounds, studying their properties, and the production of such organosilicon materials is also of theoretical importance.

Literature Review. One of the most common and effective methods for reducing the flammability of polymer materials is the use of flame retardants. According to their mechanism of action, thermostable substances can be divided into different groups, such as inhibitors of the combustion process and catalysts for the formation of coke on the polymer surface [1-2]. Selecting a thermostable substance for a specific product is a complex task, as it requires knowledge of the effect of each component on the polymer and the mechanisms of their interaction with each other.

Experimental part: Highly dispersed fire-resistant and heat-insulating fillers based on the organic compounds HIPAN and tetraethoxysilane are used to increase the fire resistance of wood and construction materials, as well as for their multi-stage protection against fire exposure.

HIPAN, which is an industrial waste product, is obtained by hydrolysis. Depending on the hydrolysis conditions (temperature, type of catalyst, presence of an organic solvent), the amount of functional groups may vary [3].

The chemical composition of HIPAN can be schematically represented as follows:



$$[-CH_2-\underset{\substack{|\\CN}}{CH}]_n \xrightarrow{[OH]} [-CH_2-\underset{\substack{|\\CONH_2}}{CH}-]_a - [-CH_2-\underset{\substack{|\\COOH}}{CH}-]_b - [CH_2-\underset{\substack{|\\COONa}}{CH}-]_c-[-CH_2-\underset{\substack{|\\CN}}{CH}]_x$$

Here, a, b, c, x depend on the hydrolysis conditions and duration.

In the presence of solutions of mineral acids, the hydrolysis of tetraethoxysilane ($Si(C_2H_5O)_4$) occurs with the elimination of ethanol and subsequent condensation of hydroxyl groups.

50 mL of HIPAN was placed into a reactor equipped with a thermometer, and tetraethoxysilane (TEOS) was added dropwise slowly (in the presence of emulsifiers) at a temperature of 30–60 °C and (in some cases) under UV irradiation with vigorous stirring. Thereafter, the solution was stirred at room temperature for several hours, yielding a viscous polymer.

The reactions were carried out in various ratios: HIPAN:TEOS = 50:1, 50:2, 50:3, 50:4, 50:5, with constant stirring.

The reaction was carried out with different ratios of starting reagents (from 10:1 with increasing HIPAN ratio) at temperatures ranging from 30 to 80 °C. With increasing temperature and TEOS amount, the resulting solid mass becomes insoluble in solvents due to extensive crosslinking, which is likely attributed to the complete crosslinking and bonding of the reacting substances. This is explained by the fact that as the degree of transition of the linear form of HIPAN to a network structure increases, the solubility of the resulting polymer decreases, forming a solid mass [4].

Table 1

Properties of the polymer obtained based on hydrolyzed polyacrylonitrile and tetraethoxysilane in various ratios at a temperature of 30 °C

No.	HIPAN (ml)	$Si(C_2H_5O)_4$ (ml)	Temperature (°C)	Viscosity (N·mm/s)
1	50	1	30	34,55
2	50	2		35,60
3	50	3		36,30
4	50	4		39,67
5	50	5		40,48
6	50	10		solid mass
7	50	20		solid mass

An increase in temperature leads to an acceleration of the process and an increase in the polymer crosslinking yield. An increase in temperature above 50 °C subsequently led to complete crosslinking and the formation of a solid mass. It should be noted that the resulting product turns into a solid mass [5]. The effect of the ratio of the reacting components on the resulting product was studied (Table 1).

As can be seen from Table 1, with increasing amount of tetraethoxysilane in different ratios of hydrolyzed polyacrylonitrile and tetraethoxysilane, the viscosity increases due to the increase in the number of crosslinks in the resulting polymer. When the component ratio is 50:1, crosslinking becomes excessive, resulting in a solid mass. Based on the above, the optimal ratio was taken as 10:1 (50:5), and further research was continued.

Additionally, the kinetics of polymer crosslinking at different temperatures were studied (Table 2).

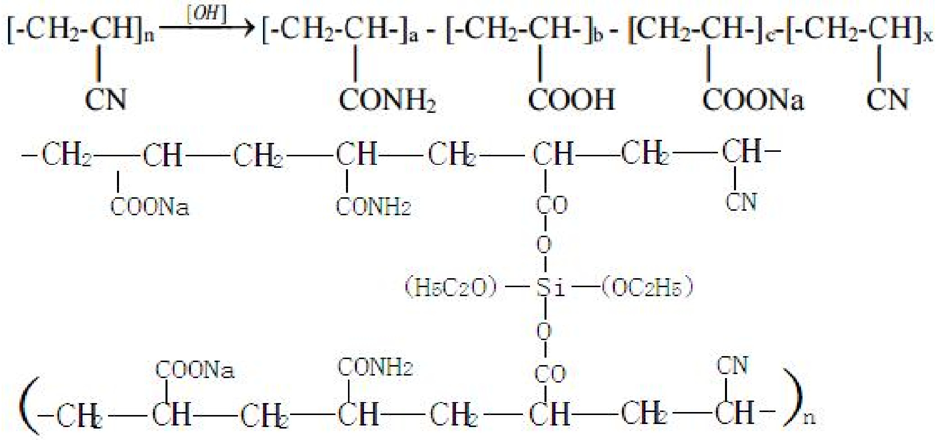
Table 2

Properties of the polymer obtained based on hydrolyzed polyacrylonitrile and tetraethoxysilane in a 50:5 ratio

No.	HIPAN (ml)	$Si(C_2H_5O)_4$ (ml)	Temperature (°C)	Viscosity (N·mm/s)
6	50	5	30	40,48
7	50	5	40	38,50
8	50	5	50	34,10
9	50	5	60	29,70
10	50	5	70	26,40

When studying the crosslinking process of the components obtained based on the optimal ratio at different temperatures (Table 3.2), it was observed that with increasing temperature, the viscosity of HIPAN decreases (at 30 °C, the solution viscosity is 40.48 N·mm/s, while at 70 °C it is 26.40 N·mm/s).

Based on the experiments conducted, the interaction of functional groups in hydrolyzed polyacrylonitrile with tetraethoxysilane can be represented by the following scheme.

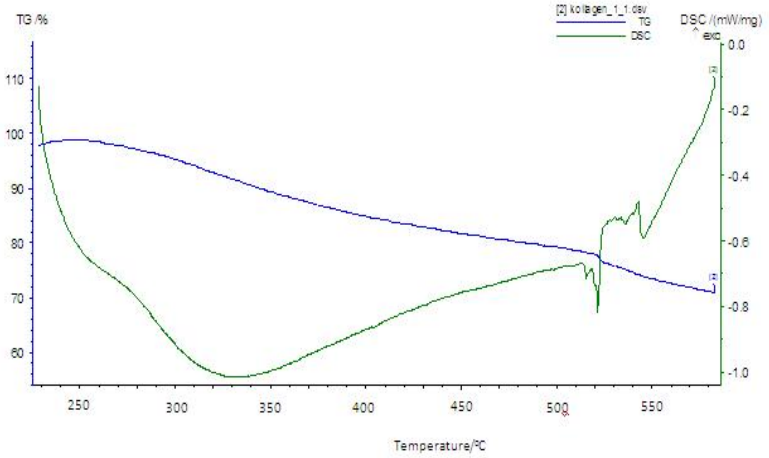


+ $Si(C_2H_5O)_4$ →

The amount of functional groups b (in the schematic representation of hydrolyzed polyacrylonitrile given above) and the degree of crosslinking of CH–COO determine the viscosity of the resulting polymer. Exceeding this value beyond the 10:1 ratio leads to the transformation of the polymer into a solid mass (via a rubbery mass).

Furthermore, to study the influence of physical processes on the crosslinking process, the effect of UV radiation was investigated. The increase in viscosity of the reaction mixture under UV irradiation indicates an acceleration of the process. Under UV radiation, the formation of active centers in the mixture is observed [6].

Study of the thermal stability of polymers and their components. The factor that ensures the fire resistance of materials treated with the obtained polymer is their thermal stability [7]. Therefore, the thermal stability of the polymer was analyzed using DTA (differential thermal analysis).



Differential thermal curve of the organosilicon polymer obtained based on hydrolyzed polyacrylonitrile and tetraethoxysilane. Figure 2.

Analysis of the differential thermal analysis results: In the temperature range of 20–522 °C, the sample slowly loses mass without any pronounced thermal processes — this is due to the gradual loss of moisture (both hydrated and non-hydrated). At 522 °C, a strong thermal emission is observed — an exothermic reaction associated with the pyrolytic decomposition of the polymer. The total weight loss of the sample in the measurement range is 27.4%. At 548–550 °C, destruction begins corresponding to the length of ordinary C–C bonds (in accordance with bond enthalpy), and the process continues until the formation of ash (until Si–O bonds remain).

Additionally, the average molecular weight and elemental composition of various organosilicon (oligo)polymers were analyzed (Table 3).

Table 3

Analysis of the average molecular weight and elemental composition of the polymer obtained based on hydrolyzed polyacrylonitrile and tetraethoxysilane

HIPAN (mL)	TEOS (mL)	Average molecular weight	Elemental composition, %				
			C	H	Si	N	O
50	1	11716	49,19	5,10	0,24	9,55	26,6
	2	11832	49,49	5,20	0,48	9,46	27,1
	3	11948	50,41	5,25	0,70	9,37	27,4
	4	12064	51,33	5,30	0,92	9,28	27,9
	5	12180	52,26	5,35	1,15	9,20	28,6
	10	12760	53,96	6,01	2,20	7,77	29,5
	20	13040	55,03	6,30	2,60	6,40	30,3

If we pay attention to the results in Table 3, the increase in the degree of crosslinking with an increasing amount of the crosslinking agent fully corresponds to the laws of polymerization reactions. This, in turn, is also reflected in the results of the elemental analysis of each oligomer. In the same situation, an increase in the amount of HIPAN (rather than the crosslinking agent) has an inversely proportional effect, which indicates a sharp decrease in crosslinking.

Conclusion: Based on the results of thermal analysis of the obtained organosilicon polymers, it was determined that thermal stability increases with an increase in the number of organosilicon bridges in the HIPAN molecule (the presence of –Si–O–Si– bonds determines the thermal stability of the polymer). By taking kaolin and liquid glass in a mass ratio of 40:60 for the polymer prepared based on organosilicon polymers, and adding 6% of the polymer relative to their mass, we have the opportunity to create a composition with specified properties.

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