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POLYMERIC FRAMETERING FOR REDUCING THE FLAMMABILITY OF CELLULOSE MATERIALS

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ABSTRACT

The article deals with some issues of creating new polymer flame-retardants for cellulosic materials. It has been established that the combustion of modified cellulose and nonwoven materials is accompanied by the formation of compounds that catalyze the dehydration reaction during pyrolysis and intensifies the formation of coke. The mechanisms of fire-retardant characteristics of new polymers are revealed.

KEY WORDS: *fire-retardant, polymer, flammability, cellulose, combustion, coke remainder, condensation, departure, recuperation.*

Despite the rapid development of the production of synthetic polymer materials, plastics and composite materials, there is a shortage of functional materials with a complex of valuable properties, such as fire resistance, thermal stability, etc. The most promising high-molecular compounds of this type are polymers containing functional phosphorus-containing groups. In addition, in the chemistry of high-molecular compounds, works devoted to the synthesis and research of polymers containing ammonium groups have received some interest due to the possibility of their wide application in many areas of the national economy [1].

By analogy with these studies, we have attempted to develop a technology for producing phosphorus-containing polymers by similar methods, since it is known from the literature that tertiary phosphorus-containing compounds easily react with nucleophilic substitution with such negative centers as halogens, oxygen, sulfur. The latter predetermined the possibility to investigate the behavior of tertiary phosphorus-containing compounds obtained from local resources in nucleophilic substitution reactions with monomers containing in their structure both one negative center (allyl bromide, methacrylyl chloride, propargyl bromide) and two negative centers (epichlorohydrin) in order to obtain high-molecular compounds based on the products of these reactions [2].

To obtain phosphorus-containing polymers, triphenylphosphite (TFFIT) and sodium dihydrophosphate (NDGF) were selected as the most affordable, less toxic and widely used of trivalent phosphorus compounds. Preliminary studies have revealed that during the interaction of the above monomers with NDGF and TFFIT, instead of monomeric quaternary salts, high-molecular substances that do not contain free monomer molecules are released, i.e. spontaneous polymerization occurs.

With an excess of a halide-containing monomer, polymer formation stops simultaneously with the consumption of a nucleophilic agent (phosphate or phosphite), an unreacted monomer remains in the system. This indicates a close relationship between salt formation and polymerization and indicates that only molecules of halide-containing monomers that have entered into a quaternization reaction participate in polymerization. To clarify the nature of the interaction of TFF and TFFIT with the above monomers, UV and IR spectra of the initial and final products, as well as PMR spectra of the initial components were studied. It was found that in the IR spectrum of the polymer obtained on the basis of the interaction of TFF with ECG, the band corresponding to the valence vibrations of the P-Cl bond is shifted to the low-frequency region up to 1350 cm-1, compared with that in the TFF spectrum. The valence oscillation of the C-SI bond (850-800 cm–1) belonging to the ECG group disappears due to the formation of a new chemical bond $_{P+\delta \ldots . C1-\delta}$ in the area of 1350 cm-1. At the same time, new intense absorption bands are also formed in the region of 1050-1100cm-1, related to asymmetric oscillations of the simple ether bond (-C-O-C-) due to the opening of the epoxigroup (1250, 930 cm-1) of the ECG during interaction with the TFF. The synthesized polymers are brown powders, resistant to long-term storage, and dissolve in many organic solvents.

Further, the study of phosphorus-containing polymers synthesized on the basis of the interaction of TFFIT and NGF with ECG and MAX as a retarder burning cellulose and nonwovens was of interest.

Samples of fire-proof cellulose and nonwovens were obtained by a known method [3]. Table 1 shows the main thermal parameters of fire-proof samples of cellulose and nonwovens. As can be seen from Table 1, the thermal stability of fire-proof samples increases with an increase in the amount of polymer flame retardant. At the same time, coke residue yields and oxygen index values also increase. During the thermal decomposition of fire-protected cellulose, an increase in the amount of released water, coke residue and a decrease in the yield of levoglucosan is observed. It should be noted that the resulting fire-proof cellulose and non-woven materials retain almost all the applied properties of the source material. Further, it was of interest to study the effect of polymer flame retardants and a low-molecular-weight analog on the physico-mechanical and other applied properties of modified cellulose and nonwovens. Studies of the flammability of modified cellulose and nonwovens by the "fire tube" method and the determination of the rate of ignition have shown that the polymer phosphorus-containing flame retardant has a higher flame retardant effect, contributing to the transfer of the combustible material to the group of difficult-to-burn. The charring observed in this case, characteristic of any organic substance, is limited by the area of action of the flame Burning modified cellulose and nonwovens is accompanied by the formation of compounds that catalyze the dehydration reaction during pyrolysis and intensifies the formation of coke. At the same time, the coke yield and the smoke generation coefficient undergo a correlation change. The process of decomposition of modified tissue samples mainly proceeds in the condensed phase, which also explains the low smoke emission during their burning. Further decomposition of the flame retardant is accompanied by the release of inert incombustible gases that prevent flaming burning and smoldering protected material. Studies of the flammability of modified cellulose and nonwovens by the "fire tube" method and the determination of the rate of ignition have shown that the polymer phosphorus-containing flame retardant has a higher flame-retardant effect, contributing to the transfer of the combustible material to the group of difficult-to-burn. The charring observed in this case, characteristic of any organic substance, is limited by the area of action of the flame.

Table 1.
Some thermal parameters of fire-proof samples

Samples	Antiren, %	The temperature of the beginning of decomposition, T, K.	The temperature of the beginning of intensive decomposition, T, K.	Amount of coke residue, %	KI, %
Cellulose	0	473	500	0	18,0
	НА-2,0	488	509	11	22,2
	ПА-0,5	493/495	510/512	13/15	20,6/21,0
	ПА-1,0	493/497	513/514	16/18	24,2/25,2
	ПА-2,0	493/497	523/525	19/21	30,4/32,6
	ПА-3,0	497/500	543/550	22/24	34,6/36,2
Non-woven fabric	0	453	493	0	17,5
	НА-2,0	478	501	14	22,6
	ПА-0,5	473/475	513/515	18/19	21,4/22,0
	ПА-1,0	497/499	518/521	20/21	26,4/27,0
	ПА-2,0	523/530	543/550	24/26	30,6/32,6
	ПА-3,0	547/552	573/578	28/31	32,4/34,0

Note. NA-low molecular weight flame retardant, PA-polymer flame retardant. In the denominator - data for the polymer based on TFFIT-MAX, in the numerator- data for the polymer TFFIT-ECG.

Table 2
The effect of flame-retardant content on the parameters of thermal degradation of samples

Samples	content. antipyrean, %.	The temperature of the beginning of decomposition, T, К	Temp-re of the max speed, T.К	Activation energy, KJ	Temp-re of flam-ty, T, К	K.I., %
Cellulose	0	550	593	160,9	673	18
	НА-2,0	525	564	164,6	713	21,6
	ПА-1,0	540/544	578/585	163,5/164	900/930	24,2/25
	ПА-3,0	520/530	538/552	188,4/192	949/962	24,6/25,6
non-woven fabric	0	473	503	94,8	979	17,5
	НА-2,0	466	499	97,4	995	22,4
	ПА-1,0	453/460	493/495	96,4/98,4	1003/1009	26,2/27
	ПА-3,0	450/465	487/499	108/111,6	1042/1056	28,4/29

Note. NA-low molecular weight flame retardant, PA-polymer flame retardant. In the denominator- data for the polymer based on FIRMS, in the numerator- data for the polymer NDGF-ECG.

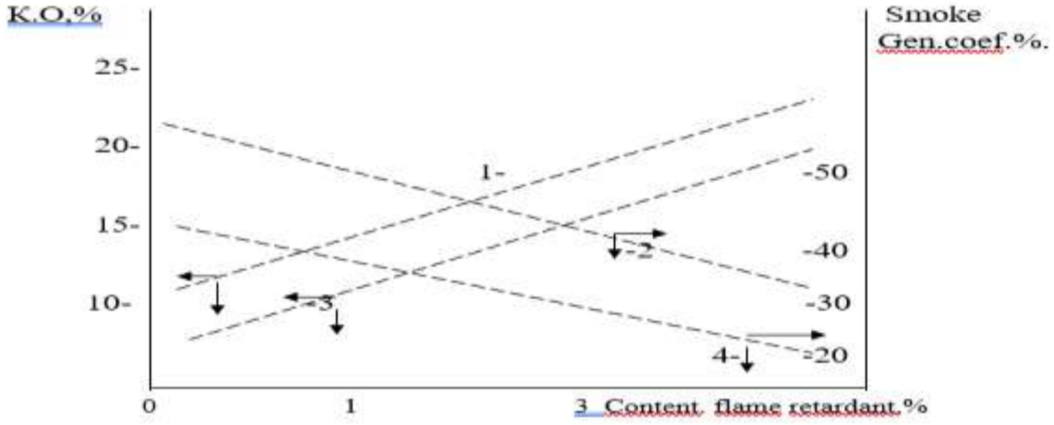

Fig. 1. The effect of a polymer flame retardant based on TFFIT: ECG for the output of coke residue and for the smoke generation coefficient: 1,3-modified cellulose material, 2,4-non-woven material.

Flammability of the modified cellulose and nonwovens is accompanied by the formation of compounds that catalyze the dehydration reaction during pyrolysis and intensifies the formation of coke. At the same time, the coke yield and the smoke generation coefficient undergo a correlation change (Fig. 1). As is known, the presence of oxygen atoms in the structure of cellulose materials enriches volatile pyrolysis products with oxygen-containing substances. The source of additional oxygen release in the flame slows down the process of soot formation.

The study of the IR spectra of the obtained coke residues showed that all of them are characterized by the presence of a very intense absorption band in the region of 1235 - 1275 cm–1, which indicates the presence of a P = O bond in cokes. Characteristic absorption bands are observed in the regions of 1020-1030 cm-1, corresponding to the P-O-C group. The IR spectrum of coke formed as a result of combustion of samples modified with a polymer flame retardant shows a strong absorption in the region of 1714 cm-1, characteristic of the carbonyl group, absent in the IR spectrum of samples modified with tricresyl phosphate. Burning Consequently, polymer flame retardant accelerates the carbonization process, i.e. soot burning during combustion of nonwovens, and the possibility of carbonation of the latter low-molecular flame retardant is excluded. The results obtained once again confirmed the effectiveness of polymer flame retardants over low molecular weight analogues.

Consequently, during pyrolysis of cellulose and nonwovens samples modified with phosphorus-containing polymer flame retardant, their propensity to capture electrons and form negative ions, the recombination of which with positive phosphonium ions in the flame by the nuclei of soot particles increases. In this regard, burning modified cellulose and nonwovens is characterized by a greater tendency to structuring and the formation of a significant amount of coke.

The process of decomposition of modified tissue samples mainly proceeds in the condensed phase, which also explains the low smoke emission during their burning. Further decomposition of the flame retardant is accompanied by the release of inert incombustible gases that prevent flaming burning and smoldering protected material. IR absorption spectra of coke residues confirmed this assumption.

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