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The Determination of Functionalized Textile Materials Durability Based on Copolymers of Acrylonitrile to Thermal and Thermal-Oxidative Degradation

Določanje obstojnosti funkcionaliziranih tekstilij na osnovi kopolimerov akrilonitrila na toplotno in toplotno-oksидativno razgradnjo

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Abstract

Acrylonitrile is widely used to produce carbon fibres, household textiles, artificial fur, etc. The modification of polyacrylonitrile fibres in an alkaline medium is extended in the production of textile sorbents. The scientific direction of fibre modification through surface activation using hydrogen peroxide is of scientific interest. However, the thermal and thermophysical properties of the samples are not examined. Therefore, interest arises when analysing the effect of polyacrylonitrile textile material functionalization on the resistance of fibres to thermal oxidation and thermal degradation. The study of thermogravimetry and differential thermogravimetry thermooxide of polyacrylonitrile – modified fibres is carried out using thermogravimetric analysis to research the thermal oxidation and thermal degradation of modified polyacrylonitrile fibre samples. A change in the rate of thermal oxidative-degradation in the process of functionalization was identified in this research. A change in the loss of mass of the sample under different conditions of functionalization was also identified. The total glass transition of the polyacrylonitrile was achieved by heating the polymer to 130 °C. The intensification self-regulation of the structure of the polymeric material through the formation of the mesophase was released at the temperature of around 100 °C. During the heating of the initial polyacrylonitrile textile material to the temperature of 70–80 °C, a weakening of intermolecular contacts was observed, which led to an increase in the mobility of macromolecular segments. The temperature of 78 °C was considered to be the initial glass transition temperature $T_{g,t1}$ and $T_{g,t2} - 121$ °C as the final temperature for the original sample. However, if the temperature exceeded 130 °C, chemical changes in the polymer occurred, in particular, the process of cyclization. The analysis of the thermogravimetry data of thermally oxidized samples resulted in the retention of the complex stepwise nature of decomposition inherent in the initial fibrous material based on acrylonitrile copolymers. The conducted analysis showed the absence of significant changes



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in the reasonable conditions of chemical modification and practical operation of textile materials.

The physical and mechanical properties of functionalized textile materials based on acrylonitrile copolymers were studied. As a result of surface functionalization, an insignificant strength reduction of functionalized textile materials occurred.

Keywords: superficial modification, polyacrylonitrile fibre, glass transition temperature, physical and mechanical properties of textile materials

Izvleček

Akrlonitril se na široko uporablja za izdelavo ogljikovih vlaken, gospodinjstkih tekstilij, umetnega krzna ipd. Modificirana poliakrlonitrilna vlakna so v alkalnem mediju razširjena za izdelavo tekstilnih absorbentov. Površinska aktivacija vlaken z vodikovim peroksidom je znanstveno zanimiva, pri čemer njihove toplotne in toplotno-fizikalne lastnosti niso raziskane. Zanimal nas je vpliv funkcionalizacije poliakrlonitrilnega tekstilnega materiala na odpornost proti toplotni oksidaciji in toplotno razgradnjo. Študij termogravimetrije in diferencialne termične analize termooksidiranih poliakrlonitrilnih – modificiranih vlaken je bil izveden s pomočjo termogravimetrične analize za proučevanje termooksidativne in termične razgradnje vzorcev modificiranih poliakrlonitrilnih vlaken. V tej raziskavi je bila ugotovljena sprememba hitrosti toplotne oksidativne razgradnje v procesu funkcionalizacije. Zaznana je bila tudi sprememba v izgubi mase vzorca pri različnih pogojih funkcionalizacije. Popoln steklast prehod poliakrlonitrila je bil dosežen pri segrevanju polimera do 130 °C. Pospešeno samourejanje strukture polimernega materiala s tvorbo mezofaze se je začelo pri temperaturi okoli 100 °C. Med segrevanjem izhodnega poliakrlonitrilnega tekstilnega materiala do temperature okoli 70–80 °C sta bila opažena oslabitev medmolekulskih interakcij in povečanje gibljivosti makromolekulskih segmentov. Temperatura 78 °C je veljala za začetek steklastega prehoda ($T_{g,T1}$) oziroma 121 °C za konec steklastega prehoda ($T_{g,T2}$) v izvornem vzorcu. Pri temperaturi nad 130 °C so nastopile kemične spremembe v polimeru, predvsem ciklizacija. Analiza termogravimetričnih meritev toplotno oksidiranih vzorcev je pokazala, da se je ohranila kompleksna postopna narava razgradnje, ki je značilna za začetni vlaknati material na osnovi kopolimernega akrilonitrila. Analiza je pokazala, da v sprejemljivih pogojih kemijske modifikacije in praktičnega manipulacije ni prišlo do bistvenih sprememb tekstilnih materialov. Proučene fizikalno-mehanske lastnosti funkcionaliziranih tekstilnih materialov na osnovi kopolimerov akrilonitrila niso pokazale pomembnega znižanja trdnosti funkcionaliziranih tekstilnih materialov zaradi površinske funkcionalizacije.

Ključne besede: površinska modifikacija, poliakrlonitrilno vlakno, temperatura steklastega prehoda, fizikalne in mehanske lastnosti tekstilnih materialov

1 Introduction

Polyacrylonitrile textile materials are among the most promising synthetic fibres. The creation of acrylonitrile textile materials with an improved set of properties that bring products closer to natural ones is an urgent and promising direction in the development of the textile industry.

The modification of polyacrylonitrile fibres in an alkaline medium is widely used in the production of textile sorbents [1-5]. To a large extent, the proper-

ties of sorbents are determined by a set of functional groups and the density of the spatial mesh caused by polymer crosslinking [3, 4, 6-9].

The reaction of polyacrylonitrile (PAN) fibres with alkali is characterized by the appearance of several types of functional groups. This is due to the successive transformations of nitrile groups into heterocyclic, amide and carboxylate-ionic groups [3, 5]. In this case, the reaction does not end with complete transformation. Some of the remaining intermediate structures have ionogenic properties [3].

It should also be noted that during modification, textile materials with functional features are obtained without changing the properties of the fibre bulk. Saponification of polyester and triacetate textile materials is a simple method for alkaline fibre surface treatment. As a result of saponification, carboxyl and hydroxyl groups are "liberated".

Surface alkaline hydrolysis can be applied to polyester materials prior to dyeing. Such processing improves the colour characteristics and quality of finished products [10-15]. Namely, the capillarity of the materials increases, the rigidity decreases, the drape is improved and the soiling of fabrics decreases [14]. A positive effect due to the hydrolysis of the polymer is achieved only when the mass of the textile material is reduced by 10–30% [10-14]. As a result, the mechanical strength of the fibre decreases.

In a scientific study conducted by the authors of the paper [14], information is provided on the modification of polyacrylonitrile fibres by activating the surface of the fibres with hydrogen peroxide. The scientific direction of fibre modification through surface activation using hydrogen peroxide is of scientific interest. A positive effect on the sorption properties of PAN fibres after their surface modification was shown. It is a known fact that polyacrylonitrile fibres are used mainly as a textile or raw material for the production of carbon fibres [15–18]. The latter can be used at elevated temperatures (1,600–2,000 °C). Therefore, interest arises from studying the effect of functionalization of a PAN-textile material on the resistance of fibres to thermal oxidation and thermal degradation. It is also possible to use modified fibres as technical textiles, where their thermal stability is necessary.

Amides are obtained through the partial hydrolysis of nitriles. The Radziszewski reaction mechanism provides for a nucleophilic attack of the nitrile group by a hydroperoxide anion. The result is an unstable intermediate that is an oxidizing agent. Subsequently, there follows the hydride transfer from the second water molecule to the intermediate formation of the hydroperoxide carboximide (Fig-

ure 1). In an alkaline solution, hydrogen peroxide restores the intermediate to the amide through the release of oxygen [14].

Mechanical properties represent some of the most important basic characteristics of fibres as textile materials. During the process of fibrous material functionalization based on acrylonitrile copolymers, a change in the surface occurs due to the formation of amide and carboxyl groups. This leads to the rapid and intense degradation of the surface layers of the polymer material. This work determined the relative strength of the textile material by comparing the strength of the original sample and samples after functionalization.

2 Materials and methods

For the manufacture of samples for functionalization, nitron D yarn ("Polymir" plant; Republic of Belarus) was used. The linear density of yarn was 15 tex. Samples of knitted fabrics were made on a class 10 flat knitting machine. The composition of the copolymer was: acrylonitrile 91%, methyl acrylate 8% and AMPS (2-acrylamide-2 methyl-propanesulfonic acid) 1%. A hydrogen peroxide solution (35%) in presence of tetraborate (pH 8.0) buffer systems was used for the processing of fibre material.

Assuming that the PAN sample before functionalization was designated as PAN-0, the concentration of hydrogen peroxide would then be as follows: 50 g/l - PAN-50, 100 g/l - PAN-100, 120 g/l - PAN-120 and 180 g/l - PAN-180.

The functionalization of the test samples was carried out according to the Radziszewski reaction. The concentration of hydrogen peroxide (35%) varied accordingly: 50 g/l, 100 g/l, 120 g/l and 180 g/l. The temperature was 98 °C, while the material to liquor ratio was 1:20. The duration of processing was 60 minutes. Samples of textile materials: nitron D (Polimир plant; Republic of Belarus), with a direct linear density of 15 tex. The composition of the copolymer was: acrylonitrile – 91%, methyl acrylate – 8% and

AMPS (2-acrylamide-2-methyl-propanesulfonic acid) – 1%.

The thermophysical properties of the samples were studied using differential scanning calorimetry (DSC). A TA Instruments Q100 calorimeter was used for this purpose. It allows measurements to be made in a nitrogen atmosphere in a temperature range from -55 to 240 °C and a heating rate of 20 °C/minutes. To do this, mass of samples 0.01–0.015 g were placed in aluminium capsules, which were then hermetically sealed.

The heating-cooling-heating scanning mode was used for the studied samples. In this case, for the analysis of thermophysical properties, as a rule, DSC curves obtained during reheating were chosen. The middle of the endothermic transition on the curve of the temperature dependence of the heat capacity $C_p = f(T)$ corresponded to the glass transition temperature ($T_{g,t}$) of the textile material based on the acrylonitrile copolymer.

The resistance of textile materials to thermal (in an atmosphere of dry nitrogen) and thermal-oxidative (in air) degradation was determined in dynamic mode using a Setaram TG TDA92 instrument in an inert atmosphere and in air in the temperature range of 20 °C to 700 °C and a temperature increase rate of 10 °C/minute with the constant removal of degradation products. The mass of the samples was 0.01–0.015 g. For each sample, thermogravimetry (TG) and differential thermogravimetry (DTG) curves were simultaneously recorded. The accuracy of measuring the temperature parameters of degradation was ± 3 °C, while the error in determining the mass loss was $< 1.0\%$.

All used methods are defined by the current international or national standards of Ukraine for the relevant textile products.

To assess the mechanical properties of yarn, which determine their behaviour during operation, a range of indicators was used, including breaking force and elongation at break [19].

In this work, physical and mechanical indicators were determined using a pendulum-type tensile

testing machine RM-3-1. The clamping length of the sample was 500 mm, the lowering speed of the lower clamp was 500 mm/min, the pre-tension force (based on the nominal linear density of the yarn is 15 tex) was 10 cN.

The results of the research are presented in relative units in order to compare and contrast the influence of functionalization conditions on the physical and mechanical properties of fibrous materials, which made it possible to limit the influence of instrument error and yarn thickness variation, as well as other systematic factors. The confidence interval was 95%, with 30 measurements for each sample.

3 Results and discussion

The analysis of thermo-oxidative degradation indicators shows the possibility of expanding the scope of the modified fibre for technical use.

Mechanical properties represent important characteristics of fibres and textile materials. The specific feature of fibres as polymeric materials is that they represent oriented systems. One of the most important properties of fibrous materials is the anisotropy of their mechanical behaviour due to the specificity of fibre structure. The elementary units of macromolecules are bonded by covalent chemical bonds. The interaction between the units of two neighbouring macromolecules is caused by weaker intermolecular bonds. As a result, the work required to break chemical bonds exceeds the work required to break intermolecular bonds by several times. Fibrous materials based on acrylonitrile copolymers have a rather high strength. During the functionalization of fibrous material based on acrylonitrile copolymers, there is a change in the surface due to the generation of amide and carboxyl groups.

Data regarding changes in breaking force and the elongation of fibrous material are shown in the table below Table 1.

Table 1: Effect of functionalization conditions on changes in breaking force and the elongation of PAN-based fibrous material

№	Functionalization conditions (of 35% H ₂ O ₂) (g/l)	Relative strength in relation to the original sample, relative units	Relative root-mean-square error (%)	Relative extension (%)	Relative root-mean-square error (%)
1	Original sample	1.0	11.8	1.0	15.5
2	50	0.937	14.3	0.998	19.3
3	100	0.995	15.4	0.940	20.3
4	120	0.873	14.1	0.938	21.2
5	180	0.911	15.0	1.11	21.0

However, with an increasing concentration of hydrogen peroxide, the loss of strength characteristics does not exceed 5–8%. The formation of this kind of “threshold” can presumably be caused by changes in the internal structure of the fibrous material. With a further increase in concentration, it is possible to form “semi-cross-linked” structures due to further transformations of amide groups. Since the changes directly on the surface of the fibrous material are of interest, functionalization using hydrogen peroxide concentrations of up to 100 g/l is recommended for the textile industry with the least loss of breaking strength. For samples of fibrous materials based on acrylonitrile copolymers, functionalized at pH = 8.0 for samples № 3, 4 and 5 (Table 1) in relation to the original sample, a slight increase in elongation is observed. Thus, the changes could be determined by changes within the structure of the fibrous material. Therefore, surface functionalization is associated with an insignificant change in strength characteristics, which does not lead to a change in the consumer properties of the fibrous material.

For a two-phase textile material based on acrylonitrile copolymers, two temperature transitions (glass transitions) $T_{g,T1}$ and $T_{g,T2}$ are inherent. The $T_{g,T1}$ transition occurs at a temperature of ~100 °C, above which the vitrified mesophase turns into a mesophase melt. The $T_{g,T2}$ transition at a temperature of ~150 °C is the transition of a glassy amorphous phase to an elastic state. The transition of the ordered phase from the glassy state to a fluid state occurs at a lower temperature than the softening of the amorphous phase.

Similar to other linear polymers, fibre-forming acrylonitrile copolymers are characterized with several stages of polymer at the initial stage of fusion, which is associated with the manifestation of mobility or substitution of functional groups, i.e. conformational transitions. Based on the well-known Flory-Huggins model, the glass transition temperature should be directly proportional to the energy required to change the mobility of a segment.

The purpose of this study was to investigate and compare the changes in indicators of physical and mechanical properties and glass transition temperature of textile materials based on thermogravimetry (TG) and differential thermogravimetry (DTG) curves using the differential scanning calorimetry (DSC) method. In Figure 1, DSC curves characterizing heat release during heating of the original PAN fibre samples (curve 1) and after their functionalization (curve 2) are depicted. Summary parameters are shown in Table 2.

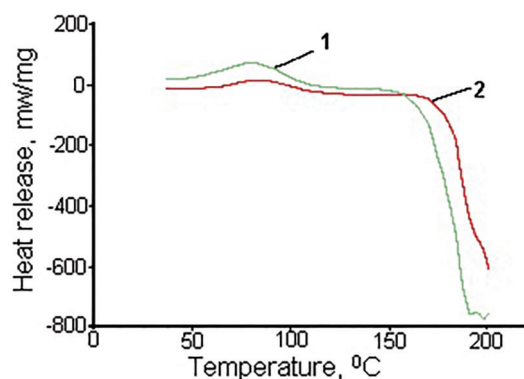


Figure 1: DSC thermograms for PAN: curve 1 is PAN-0; curve 2 is PAN-180

When PAN was heated to 130 °C, the polymer completely fused. At a temperature of around 100 °C, self-ordering of the structure of the polymeric material was intensified with the formation of a mesophase.

When the sample PAN-0 was heated to the temperature of 80 °C (Figure 1, curve 1), the significant weakening of intermolecular contacts was observed. This caused an increase in the mobility of macromolecules segments.

The temperature of 78 °C can be considered the

initial glass transition temperature $T_{g,T1}$. Accordingly, $T_{g,T2} - 121$ °C can be considered the final temperature for the initial sample (Table 1). When heated above the temperature of 130 °C, chemical changes in the polymer occur, i.e. the process of cyclization.

When functionalized PAN-180 is heated, the temperature of 82 °C can be considered the initial glass transition temperature $T_{g,T1}$, while $T_{g,T2} - 132$ °C can be considered the final temperature (Table 1). The glass transition temperature is 92 °C.

Table 2: Thermophysical characteristics for samples of textile material based on acrylonitrile copolymers

Samples	$T_{g,T1}$ (°C)	$T_{g,T2}$ (°C)	$\Delta T_{g,T}$ (°C)	$T_{g,T}$ (°C)	ΔC_p (Wg ⁻¹ K ⁻¹)
PAN-0	78	121	43	85	0.398
PAN-50	76	125	44	84	0.404
PAN-100	75	130	43	82	0.385
PAN-120	78	128	48	85	0.432
PAN-180	82	132	50	92	0.447

Thus, the value of $T_{g,T}$ is shifted to higher temperatures. In this case, the temperature range of the glass transition process ($\Delta T_{g,T}$) expands slightly. The change in the glass transition temperature is affected by the presence of polar groups, as well as the location thereof.

The glass transition temperature increases with an increase in the number of CN-groups and with an increase in the size of substituents. A decrease in the size of substituents occurs if the polymer chains have sufficient kinetic flexibility. Obviously, the decrease in $T_{g,T}$ is associated with the transformation of nitrile groups into amide and carboxyl groups, which confirms the passage of the Radziszewski reaction. Carboxyl groups are effective catalysts for the cyclization process. Their presence in the polymer leads to a decrease in the temperature level of its manifestation and an increase in the glass transition temperature. This is observed for samples after treatment with a high concentration of hydrogen peroxide (above 100 g/l).

As mentioned earlier, polyacrylonitrile has two glass transition temperatures. It is the second temperature that is determined by the shift of the equilibrium of the dipole interaction of nitrile groups

[20]. The second glass transition temperatures of the original sample based on acrylonitrile and functionalized copolymers can differ by 10 degrees or more.

The TGA method was used to determine the thermal characteristics of the studied samples based on acrylonitrile copolymers. The method is most sensitive to the loss of mass of samples under the influence of temperatures, as well as the loss of water, the content of which varies with surface changes, in particular, with functionalization.

The TGA curves have clear inflection points (Figure 2). The character of polymer decomposition rate curves can be described as bimodal. This indicates the occurrence of the thermolysis process with an increased rate of decomposition in two areas.

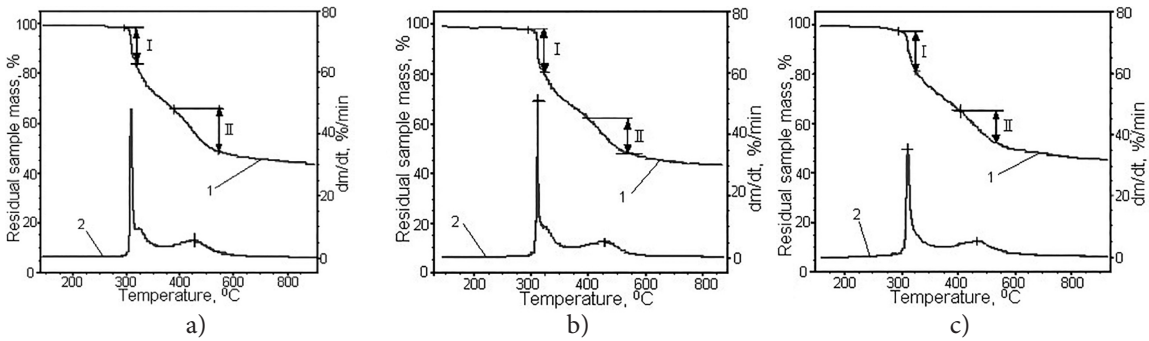


Figure 2: Typical TGA curves (1) in an inert medium and the corresponding differential DTG curves (2) for samples: a) PAN-0, b) PAN-100 and c) PAN-180

As the results of the study show, when using different amounts of hydrogen peroxide, the temperature ranges of degradation and the corresponding

temperature of the rate of degradation are practically unchanged (Table 3).

Table 3: Influence of chemical modification of textile materials based on PAN on their resistance to thermal degradation

Sample	Stage	Temperature ranges of degradation (°C)		Temperature of the max. rate of degradation, T_{dmax} (°C)	The rate of degradation, v_{dmax} (%/min)	Mass loss, Δm (%)
		$T_{ds}^{a)}$	$T_{df}^{b)}$			
PAN-0	I	295	322	312	50.7	14.3
	II	397	470	427	5.0	17.3
PAN-100	I	294	318	309	47.0	14.6
	II	388	471	429	5.3	17.5
PAN-180	I	295	326	312	35.1	13.5
	II	403	468	435	4.9	16.3

a) Starting temperature; b) Final temperature

The temperature of the rate of degradation, which for the first stage (I) lies in the range of 294–326 °C, is 309–312 °C. At the second stage (II), with practically unchanged temperature limits, the increase in the temperature of the rate of degradation is observed. In this case, the degradation rate at the

first stage decreases, while the mass loss remains constant for each stage (Table 3).

Figure 3 depicts TGA and the corresponding DTG curves showing the effect of thermal-oxidative degradation on the properties of textile materials.

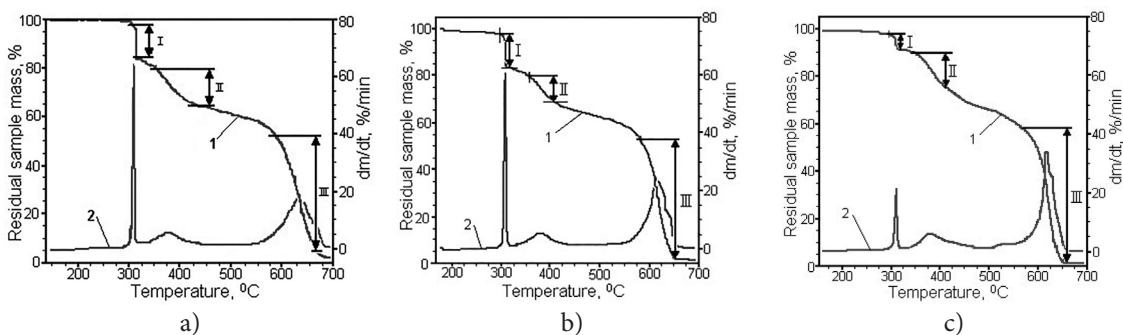


Figure 3: Typical TGA curves (1) in air and corresponding differential DTG curves (2) for samples: a) PAN-0, b) PAN-100 and c) PAN-180

The analysis of the TGA data of thermally-oxidized samples shows that they retain the complex stepwise nature of decomposition in the initial fibrous material based on acrylonitrile copolymers (Table 4).

Table 4: Influence of chemical modification of textile materials based on acrylonitrile copolymers on their resistance to thermal-oxidative degradation

Sample	Stage	Temperature ranges of degradation (°C)		Temperature of the max. rate of degradation, $T_{d(max)}$ (°C)	Temperature of rate of degradation, $v_{d(max)}$ (%/min)	Mass loss, Δm (%)
		$T_{ds}^{a)}$	$T_{dF}^{b)}$			
PAN-0	I	295	317	310	68.4	15.2
	II	334	416	377	5.5	15.6
	III	563	682	634	17.2	55.2
PAN-100	I	297	316	309	64.9	14.2
	II	341	419	379	5.3	14.1
	III	560	657	613	24.0	55.7
PAN-180	I	296	319	310	20.6	6.2
	II	344	485	383	6.0	23.0
	III	570	659	613	24.0	55.7

a) Starting temperature; b) Final temperature

Another well-defined interval appears with the maximum decomposition temperature. Thus, the curves are trimodal with three corresponding stages. A shift in the temperature ranges of decomposition is observed. The rates of mass drop of the samples differ by increasing the temperature: higher in the initial section and smoother, slower in the final section (Table 4). As the amount of amide compounds in the samples increases, the shift of the inflection point to the region of high temperatures may indicate the interaction of macromolecules and the formation of semi-crosslinked systems.

For the PAN-0 sample, a broader peak is observed

at the third stage of degradation. The situation is similar to the PAN-180 sample at the second stage (Table 4). With an increase in the amount of H_2O_2 during processing, the maximum rate of degradation in the first stage also decreases. It is 68.4%/min for PAN-0, 64.9%/min for the PAN-100 sample and 20.6%/min for the PAN-180 sample. In this case, the second stage is accompanied by an increase in mass loss: 15.6% for the PAN-0 sample and 23.0% for PAN-180.

As evident from Table 4, the thermal stability of textile materials increases in the process of functionalization, as seen by higher values of degradation temperatures ($T_{d(5\%)}$, $T_{d(50\%)}$, $T_{d(max)}$).

Table 4: Thermal characteristics (in an inert atmosphere and in air) of initial and functionalized samples of textile materials based on acrylonitrile copolymers

Environment	Sample	$T_{d(5\%)} (°C)$	$T_{d(50\%)} (°C)$	$T_{d(max)} (°C)$
N_2	PAN-0	304.13	456.16	679
	PAN-100	307.6	462.23	685
	PAN-180	309.64	494.44	692
O_2	PAN-0	304.26	590.66	676
	PAN-100	307.92	591.74	680
	PAN-180	314.46	596.94	687

The following changes were also noted during the course of the study: the rate of thermal-oxidative degradation and the index of sample mass loss. This

confirms the change in fibre structure and testifies to changes in the surface under various functionalization conditions.

4 Conclusion

The study of the thermogravimetry and differential thermogravimetry thermooxide of PAN-modified fibres was carried out using additional thermogravimetric analysis. The study of the thermal oxidation and thermal degradation of samples of modified PAN fibres was performed. Thermogravimetric analysis was used for this purpose. It was established that in the process of functionalization there was a change in the rate of thermal-oxidative degradation. There is a change in the loss of the mass of the sample under different conditions of functionalization. When the PAN is heated to 130 °C, the complete glass transition of the polymer occurred. At a temperature of around 100 °C, the self-regulation of the structure of the polymeric material structure intensified with the formation of the mesophase.

When the initial PAN textile material was heated to a temperature of ~70–80 °C, a weakening of intermolecular contacts was observed, which led to an increase in the mobility of macromolecular segments. The temperature of 78 °C can be considered the initial glass transition temperature $T_{g,T1}$, while $T_{g,T2}$ - 121 °C can be considered the final temperature for the initial sample. When heated above a temperature of 130 °C, chemical changes in the polymer occur, in particular, the process of cyclization.

The analysis of the TGA data of thermally oxidized samples shows that they retain the complex stepwise nature of decomposition inherent in the initial fibrous material based on acrylonitrile copolymers. The conducted analysis shows the absence of significant changes in the reasonable conditions of chemical modification and the practical operation of textile materials.

The influence of the surface functionalization of polyacrylonitrile textile material was researched, with results showing insignificant changes in strength characteristics. The absence of the deterioration of consumer properties of fibrous material was observed. Thus, the conducted research highlights the possibility of practical application of

functionalized textile polyacrylonitrile materials in domestic and technical applications (production of knitwear, fabrics for various purposes, carpets, curtain products, filter pads, etc.), where the influence of thermal characteristics on functional properties is an important factor.

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