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CHEMICAL METHOD OF FIBROUS MATERIALS SURFACE ACTIVATION ON THE BASIS OF POLYETHYLENE TEREPHTHALATE (PET)

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Abstract. The possibility of surface activation of polyethylene terephthalate materials due to their weak alkaline hydrolysis which does not cause weakening of the fibrous material has been shown. The activation has the positive effect on the functional products application on polyester material surface. It has been shown that during weak surface hydrolysis the initially smooth surface of PET becomes rough (at nanoscale level) that increases adhesion of functional products for polyester material.

Keywords: polyester materials, alkaline hydrolysis, chemical activation.

1. Introduction

One of the promising ways to create textiles with special consumer features is the formation of filaments on the surface of ultra-thin layer of functional products. For their strong fixation on the fibrous material it must be installed on the surface of chemically active groups. Fibers based on PET virtually have not such groups (there is only a very small number of terminal hydroxyls and carboxyls). However, it is known that PET in the presence of alkali metal hydroxides, which act as catalysts for ester hydrolysis reactions and the reaction of alkaline hydrolysis, initially occurs in the outer area of the fiber [1-3]. As a result, on the surface of the PET material the hydroxyl and carboxyl groups are formed (Fig. 1).

Alkaline hydrolysis of PET material has been popular in the finishing treatment of the textile industry during 1980–1990 and was used to make synthetic materials with silk-like properties. However, the literature indicates that the positive effect due to hydrolysis of the polymer (superior neck, high hydrophilicity, low electrified, improved colorability) is achieved only when the mass loss of fibrous material is at least 10–30 % [1, 2]. As a result the breaking load of fiber decreases. [2].

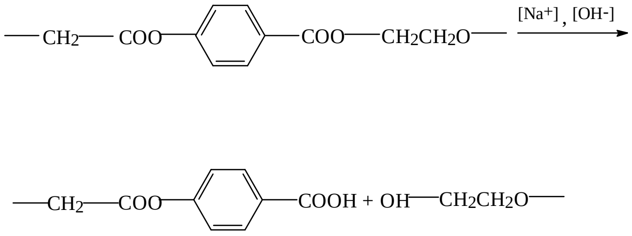


Fig. 1. Alkaline hydrolysis reaction of PET material

Significant weight loss is related to the fact that the improvement in the fiber is largely determined by its morphology. After the exposure of sodium hydroxide micro relief of polyester fibers surface usually gets fossa etching, the size and number of which are determined by the intensity of treatment [1, 4]. We suggest that the formation of active chemical groups on the surface of PET fibers can be achieved under hydrolysis milder conditions.

2. Experimental

The PET fiber was treated with aqueous solutions of sodium hydroxide (concentration in range 0.0125–1.5 mol/l) at the boiling point. The process duration varied from 5 to 20 min. The qualitative observation and identification of new surface groups were recorded using the infrared spectra (ATR-FTIR method). Quantitative determination of localized surface active groups was conducted by two methods.

Titrimetric method of the surface carboxyl groups calculating was described previously [5]. The method is based on the interaction of the carboxyl groups with calcium acetate. The exchange reaction leads to free acetic acid allocation which can be titrated. Titrated against 0.01 M sodium hydroxide was used for solution titration in the presence of a mixed indicator consisting of thymol blue and cresol red. It is necessary to note that due to the low content of carboxyl groups on the fibrous material surface a very small amount of acetic acid was formed. Therewith the acetic acid is generated in the presence of large amounts of water so it is strongly diluted. Due to a low sensitivity of the titrimetric method, it was applied only for the approximate estimation of the quantitative content of carboxyl groups on the surface.

The colorimetric method for determination of the surface hydroxyl groups' concentration was developed for PET films [6]. We adapted this method for the fiber material surface study. The method is based on the ability of active dichlortriazine dyes to form a covalent bond with the hydroxyl group of polyethylene terephthalate. The coloring active dichlortriazine bright blue azoic dye was used. Dyeing of samples was performed by the standard technology for active dyes with an index of "azoic dye" [7]. The amount of dye fixed on the fiber was determined by the difference in the color intensity of the modified and unmodified PET fibers. The color intensity was estimated from the color characteristics of the samples of coated polyester material, which was determined using color measurement complex, equipped with the program "Colorist" (version 4.2.1994, '99, the authors V. Pobedinsky, F. Telegin and I. Danilin).

Strength was estimated by the breaking load of the processed yarn. It was measured by the tensile machine TM-3-1, in accordance with standards.

3. Results and Discussion

3.1. PET Fiber Modification by Treatment in Sodium Hydroxide Solutions without and with Quaternary Ammonium Salts

Fig. 2 shows fragments of the ATR-FTIR spectra of PET films, untreated PET (1) and another one treated with sodium hydroxide (2). The appearance of a band at 3200–3500 cm^{-1} indicates that treated surface layer of polymeric material has a significant additional number of OH groups [8]. Among these groups there can be some hydroxyls ones incoming the carboxyl groups. The change of carboxyl groups which are localized on the surface of PET material was determined by the titrimetric method. As a result of the treatment process, the concentration of carboxyl groups increased in 2.6 times after treatment in sodium hydroxide solution (concentration 0.375 mol/l) at the boiling temperature during 15 min (from $4\cdot10^{-4}$ mol/kg for raw polyester material to $10.4\cdot10^{-4}$ mol/kg).

The new carboxyl groups are localized on the PET surface and are available for interaction. Therefore, the specific surface area was measured independently to characterize the surface carboxyl group density. The calculated values of the surface active groups' concentration were around $1.02\cdot10^{-6}$ mol/m^2 for the initial PET material and $2.66\cdot10^{-6}$ mol/m^2 after modification.

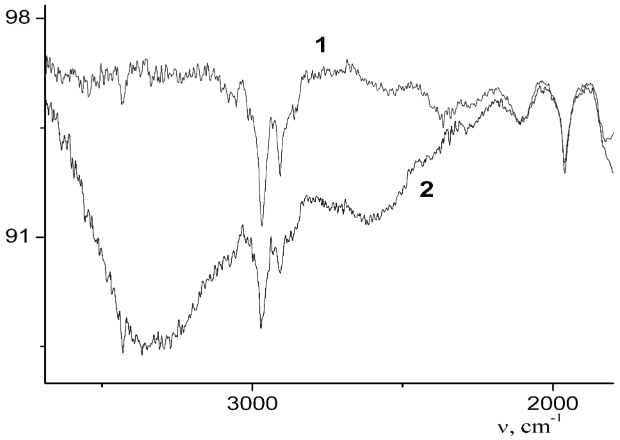


Fig. 2. Vibrational spectra of polyester film (range 2700–3600 cm^{-1}): untreated (initial) film (1) and film after treatment by boiling in 0.0125 NaOH solution for 20 min (2)

The number of hydroxyl groups formed on the surface of the PET material during the treatment process was determined by the colorimetric method (Fig. 3).

Following the data presented in Fig. 3 the processing of PET fiber material with sodium hydroxide solution leads to an increase in concentration of surface hydroxyl groups. The results obtained from the IR-spectra of the PET (Fig. 2) indicate the surface localization of a noticeable amount of hydroxyl groups.

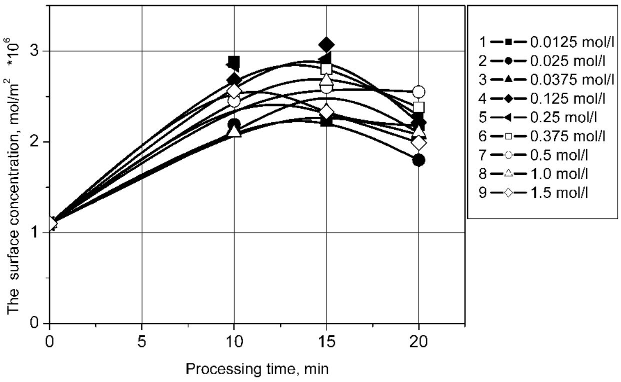


Fig. 3. The concentration of surface OH groups on the PET fibrous material after treatment using sodium hydroxide concentrations of (mol/l): 0.0125 (1); 0.025 (2); 0.0375 (3); 0.125 (4); 0.25 (5); 0.375 (6); 0.5 (7); 1 (8) and 1.5 (9)

While comparing the concentrations of surface hydroxyl and carboxyl groups derived from the treatment process, it could be concluded that they are nearly the same. For example, after treatment of the initial PET material by 0.375 M NaOH solution for 15 min, the surface hydroxyl and carboxyl concentrations are increased to $2.79\cdot10^{-6}$ mol/m^2 and $2.66\cdot10^{-6}$ mol/m^2, respectively. This conformity corroborates the hydrolysis mechanism, resulting in the formation of the equal number of hydroxyl and carboxyl groups (as shown in Fig. 1). Since the colorimetric method allows to analyze several samples at the same time it was used in serial experiments.

It should be noted that in current experiment the NaOH concentration differs by more than hundred times, but the most significant difference between the numbers of active groups formed is only ~ 40 %. The maximum value was recorded during 10–15 min of the treatment process. Processing of PET material with 0.125 M NaOH solution leads to the formation of the greatest number of the surface hydroxyl groups (Table 1).

Considering the available literature data on the mechanism and kinetics of alkaline hydrolysis of PET fiber materials [1-3], we assume that if NaOH concentration is more than 0.1 mol/l, the hydrolysis is enough intensive for the formation of appreciable amounts of hydroxyl and carboxyl groups on the fiber surface. In contrast, when NaOH concentration is above 0.5 mol/l the so-called “etching” of the fibrous material surface appeared. It is rapid and intense degradation of the surface layers of polymer material with the formation of low molecular weight hydrolysis products (oligomers and terephthalic acid, ethylene glycol), passing into the solution. Thus, most of the reactive functional groups are removed from the surface. Perhaps in a higher concentration they are present in the field of micro-cracks and defects of the fiber, where the penetration of the reagent, accompanied by the formation of micro cavities is deeper. The etching of the fibers most inevitably leads to a decrease in the thickness of the fiber and, consequently surface, reducing the breaking load. This is confirmed by the data given in Table 2.

Presented data indicate a decrease in the strength of the PET material with increasing duration time of treatment and the concentration of sodium hydroxide solution. In the most corrosive environment (concentration of sodium hydroxide – 1.5 mol/l) after 20 min the tensile strength of PET material is reduced by 15 %. At the treatment of PET material by 0.125–0.375 M NaOH the breaking load of yarn remains at the almost initial level. Changes in weight of polyethylene fibers in a process of treatment are shown in Table 3. From the data it could be stated that the maximum weight loss of PET fiber material treated for 20 min with a sodium hydroxide solution (concentration of 0.375 mol/l) is only 1.2 %. Therefore, it could be concluded that there is no strong degradation of PET fibers under these conditions.

Table 1

Concentration of surface OH groups after treatment process using different concentrations of NaOH for 15 min at boiling

NaOH concentration, mol/l	0.0125	0.025	0.0375	0.125	0.25	0.375	0.5	1	1.5
Concentration of surface OH groups, mol/m^2	$2.23\cdot10^{-6}$	$2.3\cdot10^{-6}$	$2.34\cdot10^{-6}$	$3.07\cdot10^{-6}$	$2.91\cdot10^{-6}$	$2.8\cdot10^{-6}$	$2.6\cdot10^{-6}$	$2.67\cdot10^{-6}$	$2.33\cdot10^{-6}$

Table 2

The breaking load (cN) of the PET yarn after treatment by boiling in NaOH solutions

Treatment time, min Concentration, mol/l	10	15	20
0.0125	1200±78	1183±63	1194±93
0.0250	1206±86	1210±77	1193±81
0.0375	1161±69	1164±82	1159±90
0.1250	1173±87	1239±111	1135±100
0.2500	1122±75	1142±110	1151±103
0.3750	1134±86	1128±116	1130±74
0.5000	1089±152	1107±83	1094±85
1.0000	1061±87	1074±101	1016±70
1.5000	1060±89	1045±104	1000±78
Breaking load of untreated yarn 1180±115 centi-Newton (cN)			

Table 3

The weight loss (%) of the PET yarn after treatment by boiling in NaOH solutions

Treatment time, min	0.125 mol/l	0.25 mol/l	0.375 mol/l	0.5 mol/l
10	0.18	0.4	0.85	0.91
15	0.44	0.82	1.20	1.25
20	0.28	0.88	1.20	1.68

Summarizing the above data, it should be stated that after treatment of PET fiber material using 0.25–0.375 M sodium hydroxide solutions for 10–15 min the new functional groups appeared on the surface. The tensile strength of fibrous material remains practically unchanged.

The presence of active groups can be used for fixation of functional agent that provides fiber materials imparting special properties. The assumption of increasing the degree of agent fixation on the surface of PET fiber materials after a weak surface hydrolysis was verified in the test experiment. The polyester fabric, treated by sodium hydroxide, was loaded into the solution of specifically synthesized phthalocyanine pigment which has a deodorant action. This sample had bright color. That is why the adhesion is easy to estimate on the amount of the sample, which can be determined by the colorimetric method. The treated sample has much more intense color than the untreated one.

It is known that quaternary ammonium compounds (QAC) may be used as a phase transfer catalyst-carrier [9-11].Their use can significantly reduce the concentration of alkali in the solution. The catalytic properties of QAC are shown not only in the strong alkaline, but also in a slight alkaline medium [10, 11]. The possibility of surface activation of PET material was evaluated by using a dilute solution of sodium hydroxide with the addition of compounds based on QAC using a commercial product of “Ivhimpriom”, (Ivanovo, Russia). For the treatment of PET materials by 0.025 M NaOH with the addition of “alkamon OS-2”, “alkamon OS-3”, “alkamon NP” and “triamon” were used. The “triamon” is a cationic compound based on tris(2-hydroxyethyl)methyl ammonium methyl sulfate, the “alkamon” is a composition of cationic (methyl ammonium methyl sulfate derivatives) and non-ionic agents. The QAC concentration was up to 1 g/l. The process was carried out at the boiling temperature for 15 min.

The results in Fig. 4 show that “alkamon” compounds are more effective than “triamon” ones catalyzing the hydrolysis. Apparently, the nonionic component of alkamons induces dispersing of low molecular weight products (cyclic oligomers of PET [10, 11]) in an aqueous bath. This avoids the blocking of polymer surface active groups. The maximum content of surface hydroxyl groups is achieved at the concentration of compounds based on QAC equal to 0.5 g/l. The breaking load (Table 4) and weight loss (Table 5) of PET material after the treatment are also measured. It could be noticed from the data presented in Table 4 that the breaking loads are inversely proportional to the QAS concentration. These data are in consistent with the published data on the mechanism of QAS action [9, 10]. Namely: hydrolysis of fibrous PET material is greatly enhanced in the areas of QAC sorption due to the formation of defects. This phenomenon reduces the strength of the fibrous material reducing its strength. In this case, there is also the loss of fibrous material mass.

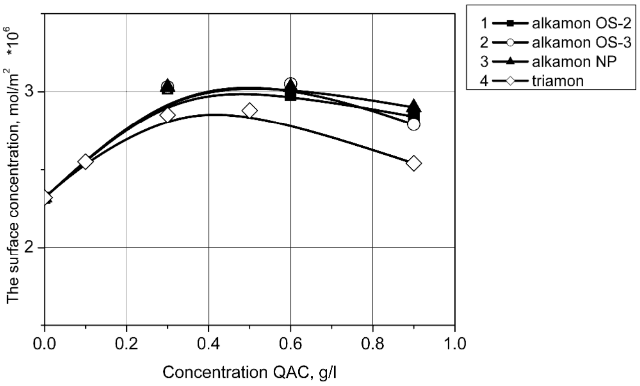


Fig. 4. The concentration of surface OH groups on the PET fibrous material after treatment by boiling in 0.025 M NaOH with the addition of QAC for 15 min

Table 4

The breaking load (cN) of PET yarn after treatment by boiling in 0.025 M NaOH with the addition of QAC for 15 min

QAC concentration, g/l	Alkamon OS-2	Alkamon OS-3	Alkamon NP	Triamon
0.1	-	-	-	1190±101
0.3	1205±100	1150±93	1151±76	1180±83
0.5	-	-	-	1160±82
0.6	1142±93	1141±79	1107±92	-
0.9	1100±77	1130±93	1070±71	1146±72
The breaking load of PET yarn after treatment by boiling in 0.025 M NaOH is 1210±77 cN				

Table 5

The weight loss (%) of PET yarn after treatment by boiling in 0.025 M NaOH with the addition of QAC for 15 min

QAC concentration, g/l	Alkamon OS-2	Alkamon OS-3	Alkamon NP	Triamon
0.3	0.35	0.37	0.2	0
0.6	1.04	0.54	0.69	0
0.9	1.21	0.43	0.88	0

The strength of fibrous material decreases using QAC; that is why it s better to use sodium hydroxide with the concentration of 0.125–0.25 mol/l at the boiling point for 10–15 min.

3.2. Modification of Polyethylene Terephthalate Fibers with Aqueous Solutions of Ammonia and Amides

Strong hydrolysis effect on PET was obtained by the addition of a concentrated aqueous solution of ammonia [12], while the diluted solutions hydrolyzed the surface localized PET oligomers [13, 14]. Consequently, it could be assumed that the presence of an optimum amount of ammonia may provide a weak surface hydrolysis of PET fiber without reducing its strength. The PET fiber material was treated in aqueous solutions of ammonia in the concentration range of 0.01–1.15 mol/l at the boiling temperature for 10–20 min to determine the optimum process conditions under which the maximum number of hydroxyl groups would not lose strength. The surface concentrations of OH groups on the treated PET fiber are shown in Fig. 5.

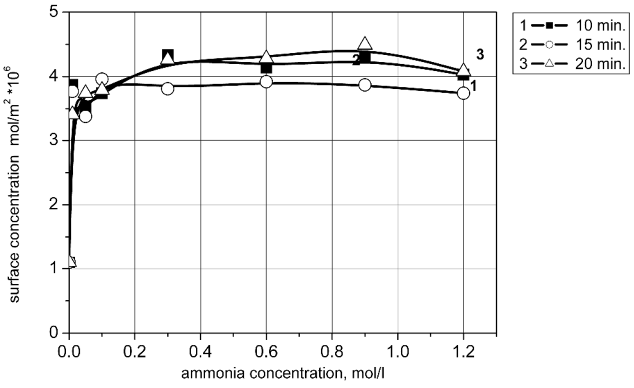


Fig. 5. The surface concentration of OH group on the PET fibrous material after treatment by boiling in aqueous solutions of ammonia for: 10 (1); 15 (2) and 20 (3) min

Comparative analysis of Fig. 3 and Fig. 5 shows that more OH groups are produced on the surface of the PET in the presence of ammonia than those in the presence of sodium hydroxide of the same molar concentration, *i.e.* aqueous ammonia is a more effective catalyst for the hydrolysis of PET material. Fig. 4 also shows that with the increase of ammonia concentration from 0.1 to 0.9 mol/l the number of formed OH groups is almost unchanged. The increase in processing time from 10 to 20 min has also a negligible effect on the OH group surface concentration.

The data in Table 5 show that changes in PET filament breaking load due to the treatment process using ammonia solutions are within experimental error, *i.e.* directed hydrolysis does not adversely affect the strength characteristics of the treated yarn.

Table 5

The breaking load of PET yarn after treatment by boiling aqueous solutions of ammonia

Treatment time, min	0.3 mol/l	0.6 mol/l	0.9 mol/l	1.2 mol/l
10	1123±83	1148±114	1171±124	1222±112
15	1184±130	1234±137	1217±79	1210±139
20	1161±76	1159±106	1156±137	1192±88
Breaking load of untreated yarn is 1180±115 cN				

The comparative analysis of the data in Tables 1 and 5 shows that in the case of processing PET yarns with sodium hydroxide breaking load of the thread remains at the original level by using a solution of the optimal concentration (0.125–0.25 mol/l). In the case of treatment by aqueous solutions of ammonia, the strength is not changed throughout the investigated concentration range (0.01–1.15 mol/l). Apparently, the presence of ammonia, in contrast to sodium hydroxide, eliminates forming the deeper cracks and micro cavities by surface "etching".

Since ammonia is highly fugitive and solutions have a sharp unpleasant odor, the alternative processes were suggested [15]. Therefore, in the present study the effectiveness of carbamide and acetamide as modifying agents was evaluated.

The highest concentration of carbamide (0.33 mol/l) was chosen because two molecules of ammonia are obtained during the thermal hydrolysis of one carbamide molecule. It corresponds to the ammonia concentration of 0.66 mol/l in the solution. The next point was the fact that the concentration of ammonia in the solution of ~ 0.1–0.2 mol/l was sufficient to obtain a high surface concentration of OH group (Fig. 5). Fig. 6 shows that OH group surface concentrations are considerably increased after the PET fiber treatment with aqueous solution of carbamide. The high surface concentration of hydroxyl groups is formed after treatment for 15–20 min by the solution with carbamide concentration of 0.05–0.1 mol/l. Under such conditions the surface concentration of OH approximately in 1.4 times higher than that after treatment with ammonia solutions. Consequently, it could be reported that the mechanism of PET carbamide hydrolysis intensification is not limited to the catalytic effect of ammonia released by carbamide when heated.

The results of PET fibers treatment using acetamide and carbamide in different concentrations are present in Figs. 7a and b. As follows from the data presented in Fig. 7 the surface concentration of OH groups on the PET fiber is significantly higher when using carbamide. It could be stated that the thermal decomposition of carbamide leads not only to the PET material surface hydrolysis by formed ammonia but also to the possible aminolysis of PET surface [12]. The JED-2300 Energy Dispersive X-ray Analyzer was used to study the surface of PET fiber material treated with a carbamide solution. The results are shown in Fig. 8. The surface concentration of the elements C, N, and O are measured by this method and presented in Table 6.

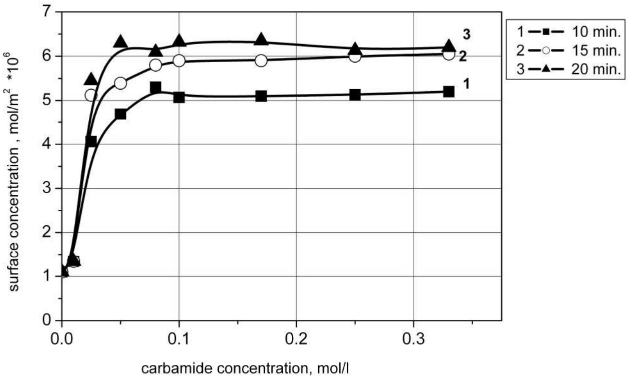
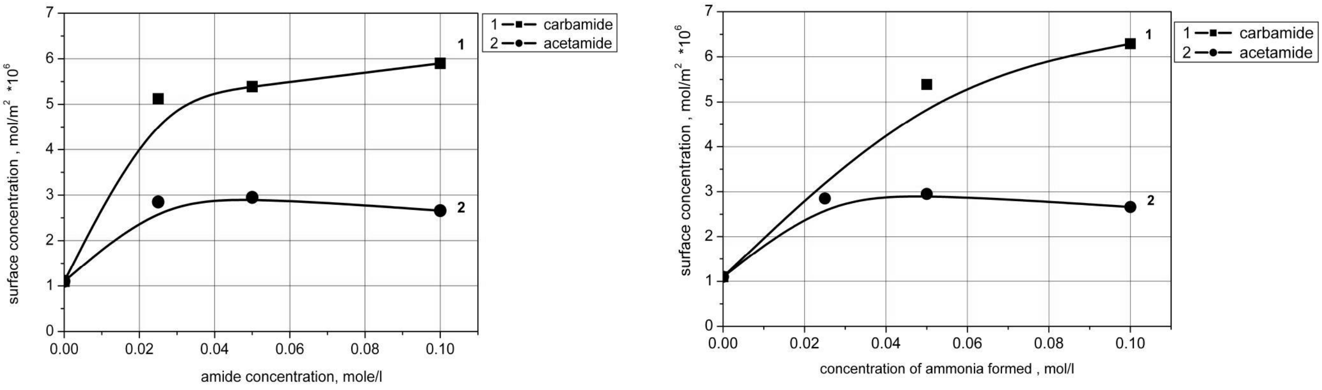


Fig. 6. The concentration of surface OH groups on the PET fibrous material after treatment by boiling in carbamide aqueous solutions for: 10 (1); 15 (2) and 20 (3) min



a)

b)

Fig. 7. The concentration of surface OH groups on the PET fibrous material after treatment by boiling in aqueous solutions of carbamide and acetamide for 15 min: *vs* the initial amides concentration (a) and *vs.* the concentration of ammonia formed due to the thermal decomposition of amides (b)

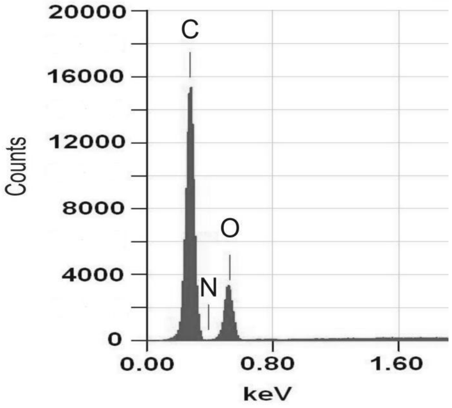
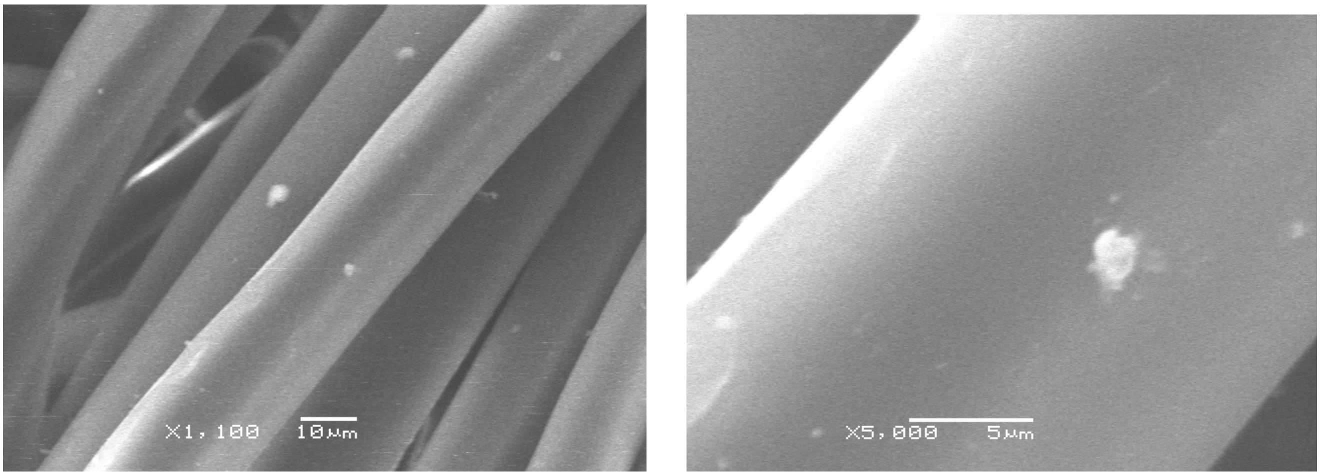


Fig. 8. The results of energy dispersive analysis of PET fabric treated by a carbamide solution of concentration 0.05 mol/l for 15 min



a) b)

Fig. 9. The scanning electron microscopy images of PET fibers treated with 0.05 M carbamide solution for 15 min at boiling point. Magnification of 1100x (a) and 5000x (b)

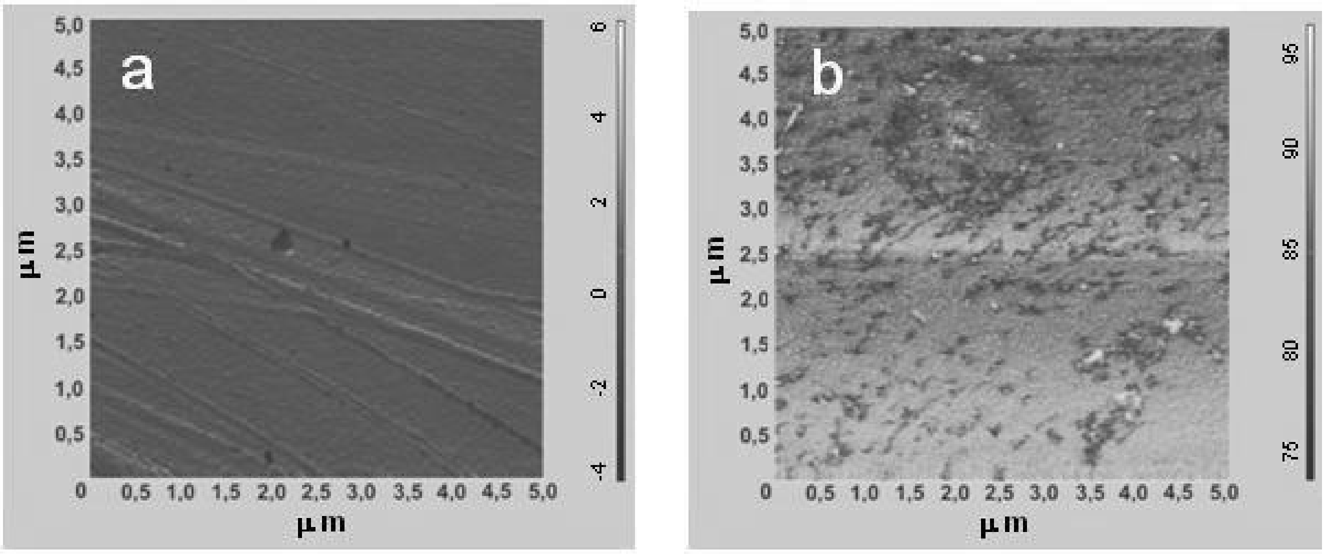


Fig. 10. Phase contrast images of PET film (size 5x5 μm) (AFM): initial (a) and treated for 15 min with a solution of carbamide concentration of 0.05 mol/l (b)

The elemental analysis shows that the great surface concentration of nitrogen for treated PET fabric confirms our assumption. The most likely mechanism for the formation of surface groups, in our opinion is the exchange reaction of carbamide with PET, and further hydrolysis of obtained products.

The measured breaking load of PET fabrics treated with solutions of different concentrations of carbamide is shown in Table 7. The presented breaking load values of PET fibers suggest that the processing with carbamide solutions does not alter its mechanical strength. The comparison of PET fiber surface modifications using sodium hydroxide, ammonia and carbamide shows that the addition of carbamide at concentration of 0.05–0.1 mol/l is most effective.

The presence of reactive groups on the surface of fibrous material and the morphology of surface are very important for technological application. In particular, the adhesion of compounds to a smooth fiber is significantly lower than for a rough one. In this connection it is interesting to assess the impact of carbamide solution on the morphology of PET fibers. It is known that the treatment of PET materials with solutions of sodium hydroxide leads to the improvement of fiber tactile characteristics due to roughness appearing [1, 4, 16]. The treatment was carried out under hard conditions and accompanied by the loss of weight and strength of the material.

Table 6

The surface concentration of the elements C, N, and O of PET fabric treated by a carbamide solution of 0.05 mol/l concentration for 15 min

Element	E, keV	At, %	Error, %
C	0.277	46.17	0.02
N	0.392	29.13	0.34
O	0.525	24.70	0.25

Table 7

The breaking load (cN) of PET yarn after treatment by boiling in aqueous solutions of carbamide

Treatment time, min	0.05 mol/l	0.017 mol/l	0.25 mol/l	0.33 mol/l
10	1191±120	1152±100	1246±90	1208±77
15	1232±104	1177±77	1210±98	1205±119
20	1213±114	1203±104	1184±137	1170±101
Breaking load of untreated yarn is 1180 ± 115 cN				

The images obtained by scanning electron microscopy (Fig. 9) shows that PET fibers treated with a solution of carbamide in the optimal mode (concentration of 0.05 mol/l, duration 15 min) are smooth. At the same time atomic force microscopy (Fig. 10) shows that the surface structures with lateral length ~ 300 nm appear on the treated PET film (b). These structures are apparently associated with a local change in the chemical composition of the surface.

3.3. Effect of Surface Modification of PET Fabrics for their Antimicrobial Finish

The hygienic protection of human is one of important tasks for medicine, transport, military condition *etc*. One way to perform this target is developing of textile materials with biological activity [17]. In spite of the high demand there is a limited range of such products on offer. However it is better to use PET fiber materials for many reasons. They are relatively cheap, have high durability, dimensional stability, aesthetic characteristics. Moreover, they are widely used for clothing and linen sports and medical applications [12].

PET materials have a good resistance to micro-organisms [12], but some types of bacteria can grow on the impurities that may appear on the surface. One of the ways to improve the antimicrobial properties of materials is fixing on the surface of a special compound which imparts the required properties on the surface. However, for PET based textile materials this approach is very difficult, because there is a very small number of active groups on the surface of PET fibers. The increase of active groups' surface concentration is needed for suitable fixation of antimicrobial agents on the PET surface.

We have shown above that in the case of PET fibrous material the formation of chemically active groups in the PET surface layer can be achieved by controlled hydrolysis. In our opinion, we can expand the range of compositions for antimicrobial finishing of PET materials due to oxygen-containing reactive groups formed on the fiber surface. The good fixation will also contribute to improvement of quality and durability of its antimicrobial finish [18].

Preparation of antimicrobial finishing of PET fabric was selected among non-migratory ones, since biocide product must only be applied to the substrate (textile material), and do not migrate to human skin, causing allergic reactions to its action [19]. The most popular products are non-migratory Sanitized T99-19 and T25-25 Sanitized-Silver, produced by Swiss company Sanitized AG (Sanitized AG), which is a world leader in the development and production of biocide agents for textile, leather, paper and plastics.

Sanitized T25-25-Silver (chloride of silver as an active substance) is traditionally used for antimicrobial finishing PET fabrics [19]. It is fixed on fabrics due to intermolecular interaction.

As an alternative the low cost finishing agent Sanitized T99-19 was considered for the permanent finishing of cellulose fabrics. This composition is based on the quaternary ammonium compound of silicon and leads to antimicrobial and antifungal protection. It is not harmful to humans and environment and it has high levels of protection against micro-organisms and bacteria.

For antimicrobial finishing the PET fabric surfaces were pre-treated by 0.05 M carbamide solution during 15 min at the boiling point. Then the sample was washed by distilled water and dried in air. After that the fabric was modified in an aqueous solution of antimicrobial agent (concentration around 0.4 % by weight of fabric) during 5 min at boiling temperature. Then the modified sample was washed by distilled water and dried in air again.

The biocide effect of Sanitized T99-19 modified PET fabric was studied in the Ivanovo State Medical Academy. The Gram-positive and Gram-negative bacterial cultures: *Staphylococcus aureus* (*S. aureus* – Gram(+)) and *Escherichia coli strain* M-17 (*E. coli* - Gram(-)) were tested. The antimicrobial finishing stability after washing and dry friction [7] was also evaluated.

At the first stage the inhibition of microorganisms growth (lysis zone) around the samples after 24 h of incubation at 310 K was measured. In this test we observed the absence of inhibition zone around the samples. It should be noted that it is typical for non-migratory agent, which did not diffuse into the culture medium. At the second stage the simplified version of counting the microbiological test ASTM E 2149 [20] was applied. This test is based on counting the number of colonies produced into saline solution after 24 h contacting with grinded modified PET fabric. Initial number of colonies was inserted as a suspension. The number of colonies formed was evaluated photometrically due to changes in turbidity (light scattering) of the solution.

The results of Sanitized T99-19 application for antimicrobial finishing of PET fabrics are given in Table 8. According to the results presented in Table 8 the initial fabric after antimicrobial finishing by Sanitized T99-19 does not demonstrate antimicrobial effect. The absence of microbial activity of the initial fabric after antimicrobial finishing shows that Sanitized T99-19 is not fixed on inactivated surface. At the same time the fabric pre-treated by 0.05 M carbamide solution during 15 min after antimicrobial finishing demonstrates excellent microbial protection. Thus, it was found that pretreatment process allows using Sanitized T99-19 for antimicrobial finishing of PET fabric. Biocide effect is greatly effective against Gram-positive bacteria (Table 8), *i.e.* it has high resistance to wet and dry friction.

Table 8

ASTM E 2149 test of Sanitized T99-19 application for antimicrobial finishing of PET fabrics

State of PET fabric	Colonies growth, %	
	E. coli	*S. aureus*
Initial fabric after antimicrobial finishing	28.5	21.5
Fabric pre-treated by 0.05 M carbamide solution during 15 min after antimicrobial finishing	0	0
Fabric pre-treated by 0.05 M carbamide solution during 15 min after antimicrobial finishing and dry friction test	6.6	1.3
Fabric pre-treated by 0.05 M carbamide solution during 15 min after antimicrobial finishing and washing test	1	0

4. Conclusions

In this paper the special properties of textile materials were obtained due to the surface fixation of special compounds which impart the required properties. In the case of PET the fixation of compounds is difficult because the chemically active groups are practically absence on the surface. To solve this problem the surface localized hydrolysis was investigated using various chemical reagents as process initiators. It was confirmed that hydrolysis of the surface layer of fibrous PET materials leads to carboxyl and hydroxyl groups formation. By comparative analysis of the concentration of surface OH groups after treatments it was shown that the carbamide solutions at concentration of 0.05–0.1 mol/l were the most effective for modification. These conditions were suitable for the formation of oxygen- and nitrogen containing groups on the surface without noticeable changes in PET filament breaking load.

The pretreatment was applied for antimicrobial finishing of PET fabrics using low cost non-migratory agent Sanitized T99-19. Excellent biocide effect against Gram-positive and Gram-negative bacterial cultures of finished fabric was stated. The textile material properties were resistant to washing and relatively resistant to dry friction.

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ХІМІЧНИЙ МЕТОД ПОВЕРХНЕВОЇ АКТИВАЦІЇ ВОЛОКНИСТИХ МАТЕРІАЛІВ НА ОСНОВІ ПОЛІЕТИЛЕНТЕРЕФТАЛАТУ (ПЕТ)

Анотація. *Показана можливість поверхневої активації поліетилентерефталатних матеріалів з використанням слабкого лужного гідролізу, який не викликає зниження міцності волокнистого матеріалу. Активація позитивно впливає на результати нанесення на поверхню поліетерного волокнистого матеріалу функціональних препаратів. Показано, що в процесі слабкого поверхневого гідролізу початково гладка поверхня ПЕТ набуває шороховатості (на нанорозмірному рівні), що сприяє підвищенню адгезії функціональних препаратів до поліетерного матеріалу.*

Ключові слова: *поліестерний матеріал, лужний гідроліз, хімічна активація.*