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THE INFLUENCE OF PET FIBRES SURFACE ENZYMATIC MODIFICATION ON THE SELECTED PROPERTIES

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Abstract:

The effect of changes in the surface structure of glossy polyester filaments from poly(ethylene terephthalate) in terms of its micro-topography, molecular and supermolecular structure of the fibre surface layers on selected fibre surface and volumetric properties has been assessed. The performed tests and measurements have shown that the change in the general surface characteristics of PET fibres (micro-topography and hydrophilicity) results in very beneficial changes in both their volumetric (dyeability) and surface properties (wettability, pilling, oil–soil removal and electric properties).

Keywords:

polyester fibres, biochemical and chemical modification of surface structure, dyeability, wettability, pilling, electric properties, mechanical properties

Introduction

The recently intensified degradation of the natural environment has brought about a growing interest in the use of biochemical processes in the textile industry. New, environmental-friendly and effective methods of fibre treatment that allow one to obtain expected properties of textiles create the basis of sustainable development of the textile industries. Recent developments of pure technologies are connected with the utilisation of new biocatalysts in the form of enzymatic preparations. In comparison with conventional chemical systems, enzymes are natural and totally biodegradable protein structures, allowing a selective treatment of polymeric materials with low concentrations of chemicals under reasonable thermal conditions.

After many years of the use of enzymes limited exclusively to the treatment of textiles from natural fibres, recent researches create opportunities to use them for the modification of synthetic fibres surface. The most recent studies clearly show that the modification of synthetic fibres with enzymes is an ecological alternative to chemical methods proceeding under drastic conditions. The modification processes with the use of lipases, esterases or proteases allow one to functionalise the fibre surface without deterioration in the volumetric properties of fibres, e.g. strength parameters [15].

The surface properties of fibres are of paramount importance from many points of view. They are important due to the possibilities of fibre processing into final products as well as, because of the aesthetic values and performance properties of textiles. They are also a deciding factor and play a large role in relation to fibre susceptibility to static electricity, resistance to abrasion during use, diffusion of dye molecules in textiles dyeing processes, fibre capabilities to bind finishing agents and end-use finishes, as well as gloss and so-called fabric handle.

From the performed analysis of literature reports, it follows that the modification of PET fibres with lipases, esterases and cutinases results in the formation of hydroxyl and carboxyl groups on fibre surface [1,3,5,12,18-22,26,33,34,39]. The presence of these groups on the PET fibre surface was confirmed by test dyeing with reactive and basic dyes [3,19,20]. The presence of hydrophilic groups (-OH, -COOH) on fibre surface brought about a change in the surface character from hydrophobic to hydrophilic, which creates an opportunity to improve many disadvantageous properties of fibres and fabrics. The change in the chemical character of the surface layer of PET fibres has resulted in the improvement in wettability, comparable to the effect obtained by alkaline modification [1,2,4,7,11,14,19,21,23,32,35,39,42] and increase in hydrophilicity [2,5,6,9,10,15,17,24,25,35]

The consequence of the obtained increased water absorption by the fabric was a durable improvement in antistatic properties resulting from the reduced surface resistance [18-21].

The change in the surface of PET fibres after modification with esterases from hydrophobic to hydrophilic resulted also in the improvement in the resistance to soiling and oil–soil release [19] as well as dyeability with disperse dyes [1,8,32,41].

Another aim of the modification of PET fibres with commercially available enzymatic preparations was to reduce their susceptibility to pilling, a disadvantageous feature of fabrics made from these fibres [13,19,21,35-37].

The hitherto performed fragmentary studies on the modification of PET fibre surface with the use of various enzymes indicate the improvement in selected fibre properties without a distinct loss of fibre strength [1,15,21,23,38,40,42].

In the present study, the effect of changes in the surface structure of PET fibres on their selected volumetric and surface properties was assessed in terms of its micro-topography, molecular and supermolecular structures.

The tests performed by the authors of the present paper concerning the physical and chemical characteristics of PET fibre surface indicate that the most effective method of modification from the point of view of physical and physico-chemical characteristics, both micro-topography and micro-structure of the fibre surface layer, is the modification with *Esterase* preparation. As a result, a uniform and homogeneous relief-type texture of the fibre surface and the highest increase in the crystallinity degree x_{IR} were obtained. It seems to be the result of a selective and effective action of this enzyme to "etch" the non-crystalline portion of the polymer from the fibre surface due to the hydrolytic decomposition of its macromolecular chains.

Analysing the physical and chemical structure of fibre surface, one can observe that the change in the surface character towards hydrophilicity results from the decomposition (shortening) of PET chains in the surface layer of fibres. The increased extent of hydrophilicity of fibre surface due to the modification is confirmed by the fact that the value of polar component of enzyme-modified fibres is increased by more than ten times in comparison with that of unmodified fibres. The most effective method of fibre modification to change the physical and chemical characteristics of fibres seems to be that with the use of enzymatic preparations such as *Lipozyme* and *Esterase*.

Experimental

Materials

Fibres

The test items included bright, continuous polyester fibres with polyethyleneterephtalate:

draw ratio - R=4.0x; thickness of fibre - 27.2 μm; total orientation factor f_o = 0.8584; fibre crystallinity index x_{IR} = 71.1%.

Woven fabric

It use for research woven fabric from cut PET fibre: plain weave, surface weight – 227 g/tex.

Enzymes

The modification of fibre surface by the biochemical method was carried out with the use of four selected enzymatic preparations, active in relation to the fibre-forming polymer and diversified with respect to their origin, biochemical characteristics and application conditions. The characteristics of the enzymes used are given in Table 1.

Treatment procedure

The PET fibre was incubated with enzyme preparation in the sodium phosphate buffer, in autoclave Ahiba–Polymat Oryginal Hanau. Conditions of treatment are shown in Table 2.

Table 1. The characteristics of the enzymatic preparations used in biochemical modification.

Enzyme preparation	Supplier or manufacturer	Source	Optimum temperature (°C)	Optimum pH	Activity
Amano Lipase A	Aldrich	*Aspergillus niger*	45	6.0	≥12,000 U/g
Amano Lipase AK	Aldrich	*Pseudomonas fluorescens*	55	8.0	≥20,000 U/g
Lipozyme®	Fluka	*Mucor miehei*	70	8.0	>100 U/g*
Esterase	Fluka	*Bacillus starothermophilus*	65	7.0	~0.4 u/mg#

*1 U corresponds to the amount of enzyme, which sets free 1 μmol stearic acid per minute at pH 8.0 and 70°C (tristearin, Fluka No. 69498 as substrate).
#1 U corresponds to the amount of enzyme, which releases 1 μmol 4-nitrophenol per minute at pH 7.0 and 65°C (4-nitrophenyl-*n*-caproate as substrate).

Table 2. Parameters of biochemical modification.

Enzyme preparation	Concentration enzyme preparation	Treatment temperature (°C)	pH	Treatment time (min)
Amano Lipase A	2 g/l	45	6.0	30
				120
Amano Lipase AK	2 g/l	55	8.0	30
				120
Lipozyme®	2 g/l	70	8.0	30
				120
Esterase	2%	65	7.0	30
				120

After enzymatic treatment, all samples were washed first with hot water for 10 min, then with sodium carbonate solution for 10 min at 70°C (to remove the remaining protein) and finally rinsed with distilled water at 70°C (6 times). All samples were air-dried at room temperature for 24 h.

Measurement methods

The influence of changes in the fibre surface structure on the volumetric and surface properties of fibres was assessed. To test the volumetric properties of fibres, their dyeability and mechanical properties were determined, while the surface properties were tested by measuring the following parameters:
– wettability with polar and non-polar liquids,
– surface resistivity,
– susceptibility to pilling,
– oil–soil release.

Testing the volumetric properties of fibres

Testing the mechanical properties

The stress–strain curves with the action of axial tensile force constituted the basis for the assessment of changes in the tensile strength of the modified fibres. The fibres to be tested were air conditioned for 24 h under standard atmosphere according to 139:2006/A1:2012P [28]. The linear density of single fibres was determined by the gravimetric method according to PN-ISO 1973:1997/Ap1:1998 [31]. Tests were carried out with the use of INSTRON tensile tester, model 4204, equipped with pneumatic sample holders. Single filaments, previously stuck on paperboard frames, were placed in the tester holder with an initial tension of 1 cN/tex. The travel rate of the traverse was 20 mm/min. The initial filament length was 20 mm. Fifty measurements were performed for each variant. At the moment of break, the following values were recorded: maximal force, breaking force, tenacity and breaking elongation. The tests were carried out according to PN-EN ISO 5079:1999 [29].

Fibre dyeability

PET fibres were dyed with two disperse dyes from BASF in the form of standard products, with considerably different diffusion properties resulting from the differences in the dye molecular structure, i.e. molecule size and shape and its value of dipole moment [16].

The chemical structure and characteristics of dyes are given in Table 3.

The dyeing with disperse dyes was carried out in pressure cups of Ahiba-Polymat dyeing apparatus of Oryginal Hanau with automatic sample agitation. A quantity of 0.2 g samples was dyed at temperatures of 100°C and 115°C for 120 min., using a liquor ratio of 50:1 and 2% of dye in relation to fibre weight. The optimal pH 5.0 of the dyeing bath was fixed with the use of 30% acetic acid.

The dye absorption was assessed by measuring the initial and final dye concentrations in the dyeing bath. Colorimetric measurements of dyeing baths were carried out by means of a Specol spectrophotometer of Carl Zeiss Jena. The dye concentration in fibre is expressed in mg $_{dye}$/100mg $_{fibre}$.

Testing the surface properties of fibres

Contact angle

The contact angle of fibres was measured by the numerical method, using as a base of logarithm Laplace's equation, converted by Yamaki and Katayama [41] into a differential form that takes into account the axial-symmetrical shape of the drop profile and the cylindrical fibre shape. Two liquids were used: α-bromonaphthalene and glycerine. The wetting process of fibres was observed under a Biolar PI microscope equipped with a computer analyser of the image. The values of contact angle were calculated as averages of the measurements for 20 liquid drops formed of fibre.

Electric properties – surface resistivity

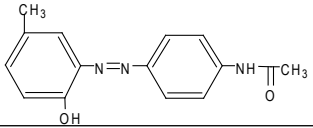
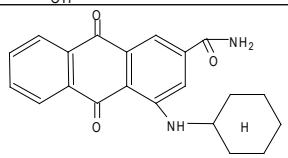
The electric properties of fibres were assessed by measuring their surface resistivity ρ_S, which was calculated from the following equation:

$$\rho_S = R_\Omega N_m \pi n d / l \quad [\Omega m],$$

where R_Ω is the resistance of the fibre sample, Nm the number of multifilaments in a sample (N = 100), n the number of elementary fibres in the multifilament (n =31), d the diameter of elementary fibre, l the intercathode distance (1 = $1x10^{-2}$ m).

The electric resistance was measured according to the standard PN-91/P-04871 [27] by means of a standard

Table 3. Characteristics of disperse dyes.

Name of dye	Chemical building	Number C.I. of dye	Molecular weight	Dipole moment
Palanil Gelb G		3/11855	269	3.6 D
Palanil Blau		6/62050	363	5.4 D

measuring system using rigid strip electrodes, an electrometer, type 610, of Keythle and a stabilised voltage supp.ly, type 4218, of Statron. The electric resistivity was determined on the basis of measuring the intensity of current passing at a constant difference in potentials on electrodes, being U = 600 and 1000 V ($R = U/I_{pass}$). The current intensity was determined on the basis of the difference in the intensities of absorption and depolarisation currents after 30 s. Fibre samples to be tested were preliminarily dried to a relative humidity of 0% and then air conditioned under the testing conditions for 24 h. The tests were performed under isothermal conditions (T = 21°C) at a specified RH: 25%, 65% and 98%, using a Feutron 3001-01 air conditioner.

Susceptibility to pilling

The susceptibility of unmodified and modified fabrics to pilling was assessed by the box method. The tests were carried out according to the standard PN-EN ISO 12945-1:2002 [30]. Fabric sample were subjected to pilling for 2 h. The assessment of the appearance of samples was made in accordance with the above-mentioned standard on the basis of photographic references.

Oil–soil release

Soiled samples were washed at a temperature of 40°C for 24 and 48 h in a bath containing a detergent ECE PHOSPHATE Reference Detergent B. After drying, the samples were rinsed with 1% alcohol solution of methyl orange to visualise the oily spots. The soil-release effects were assessed visually.

Results

Volumetric properties

Results of testing mechanical properties

The values of index characterising change mechanical properties fibre of unmodified fibre and the bio-chemically modified fibre are listed in Table 4.

Results of fibre dyeability

The values of fibre dyeability index C_t of unmodified fibre and modified fibre, at a dyeing with Palanil Gelb G i Palanil Blue in temperature from 100°C to 115°C, are listed in Table 5.

Table 4. The values of brake elongation and tenacity of unmodified and biochemically modified fibres.

Type of fibre modification	Brake elongation (%)	Tenacity (cN/tex)	Loss of tenacity (%)
Untreated	26.19	51.31	–
Treated *Amano Lipase A* 30 min	23.27	47.03	8.34
Treated *Amano Lipase A* 120 min	19.79	42.73	13.22
Treated *Amano Lipase AK* 30 min	20.95	45.85	10.64
Treated *Amano Lipase AK* 120 min	20.76	44.06	14.13
Treated *Lipozyme* 30 min	25.89	48.60	7.63
Treated *Lipozyme* 120 min	23.93	47.80	10.65
Treated *Esterase* 30 min	23.89	43.90	10.44
Treated *Esterase* 120 min	22.76	40.68	15.07

Table 5. The values of sorption coefficient C_t, $mg_{dye}/100mg_{fibre}$ for unmodified fibre and after biochemical modification dyeing temperature of 100°C to 115°C.

Type of fibre modification	**Dyeing temperature**, °C	C_t Palanil Gelb G, $mg_{dye}/100mg_{fibre}$	C_t Palanil Blue, $mg_{dye}/100mg_{fibre}$
Untreated	100	0.055	0.010
	115	0.255	0.085
Treated *Amano Lipase A* 120 min	100	1.015	0.050
	115	2.195	0.220
Treated *Amano Lipase AK* 120 min	100	1.075	0.060
	115	2.445	0.205
Treated *Lipozyme* 120 min	100	0.945	0.285
	115	1.740	0.340
Treated *Esterase* 120 min	100	1.605	0.315
	115	2.585	0.370

Results of fibre surface properties

Results of testing contact angle

The values of contact angle Θ of unmodified fibre and biochemically modified fibre are listed in Table 6.

Results of testing the fibre surface resistivity

The results of testing the fibre surface resistivity ρ_S of unmodified fibre and biochemically modified fibre are listed in Table 7.

Results of testing the fibre susceptibility to pilling

The assessment of the woven fabric susceptibility to pilling appearance of samples was made on the basis of photographic references (standard no. 2 – medium pilling).

The results of the woven fabric susceptibility to pilling before and after modification are listed in Table 8.

Table 6. The values of contact angle Θ, deg of unmodified fibre and biochemically modified fibre.

Type of fibre modification	Contact angle Θ, deg	
	Polar liquid	Non-polar liquid
Untreated	70.4	28.0
Treated *Amano Lipase A* 30 min	56.3	28.0
Treated *Amano Lipase A* 120 min	44.9	27.5
Treated *Amano Lipase AK* 30 min	576	27.5
Treated *Amano Lipase AK* 120 min	43.7	27.0
Treated *Lipozyme* 30 min.	50.1	27.3
Treated *Lipozyme* 120 min.	42.9	26.8
Treated *Esterase* 30 min	49.6	26.0
Treated *Esterase* 120 min	38.4	25.3

Table 7. The results of the fibre surface resistivity ρ_S, Ωm of unmodified fibre and biochemically modified fibre with different relative humidity of air %.

Type of fibre modification	Relative humidity of air φ, %		
	25	**65**	**98**
Untreated	$1.32 \cdot 10^{12}$	$1.29 \cdot 10^{11}$	$7.51 \cdot 10^{8}$
Treated *Amano Lipase A* 30 min	$8.67 \cdot 10^{13}$	$3.48 \cdot 10^{10}$	$1.22 \cdot 10^{8}$
Treated *Amano Lipase A* 120 min	$7.14 \cdot 1^{01}4$	$1.48 \cdot 10^{10}$	$3.15 \cdot 10^{8}$
Treated *Amano Lipase AK* 30 min	$2.35 \cdot 10^{16}$	$3.84 \cdot 10^{10}$	$4.78 \cdot 10^{8}$
Treated *Amano Lipase AK* 120 min	$3.62 \cdot 10^{15}$	$2.12 \cdot 10^{10}$	$6.50 \cdot 10^{8}$
Treated *Lipozyme* 30 min	$6.82 \cdot 10^{14}$	$1.41 \cdot 10^{11}$	$4.50 \cdot 10^{8}$
Treated *Lipozyme* 120 min	$5.14 \cdot 10^{15}$	$3.53 \cdot 10^{10}$	$5.55 \cdot 10^{8}$
Treated *Esterase* 30 min	$9.79 \cdot 10^{14}$	$2.66 \cdot 10^{10}$	$4.73 \cdot 10^{8}$
Treated *Esterase* 120 min	$9.17 \cdot 10^{14}$	$1.01 \cdot 10^{10}$	$4.48 \cdot 10^{8}$

Table 8. The fibre susceptibility to pilling

Type of fibre modification	Susceptibility to pilling
Untreated	3
Treated *Amano Lipase A* 120 min	4.5
Treated *Amano Lipase AK* 120 min	4.5
Treated *Lipozyme* 120 min	5
Treated *Esterase* 120 min	5

Results of testing the oil–soil-release

The results of testing the oil–soil-release shown in Figure 1 indicate a practically complete oil–soil-release of PET fibres modified with enzymatic preparations.

Discussion and interpretation of results

Volumetric properties

Results of testing mechanical properties

The enzymatic modification of fibres results in about 10% decrease in fibre strength in the case of all the enzymes used, maintaining a beneficial elongation at break.

Results of fibre dyeability

The results of testing the fibre dyeability after enzymatic modification, given in Table 8, in the form of sorption coefficient C_t, are very beneficial, especially in the case of the fibres modified with *Lipozyme* and *Esterase* preparations. The dyeings obtained already at a dyeing temperature of 100°C are characterised by high intensities. Hence, it may be concluded that after the enzymatic modification, the dyeing process can be performed by conventional methods without the need to use pressure apparatus, which could provide a considerable energy saving.

Surface properties

Results of testing contact angle

The values of contact angle of the fibres investigation clearly depend of the type of liquid used. The obtained results indicate a considerable reduction in the contact angle with a polar liquid, while its value is practically unchanged when non-polar liquid is used. The most effective reduction in the contact angle with a polar liquid (45.5%) is observed in the case of using *Esterase* preparation.

Results of testing the fibre surface resistivity

The results of testing the fibre surface resistivity are clearly dependent on the relative humidity of air:

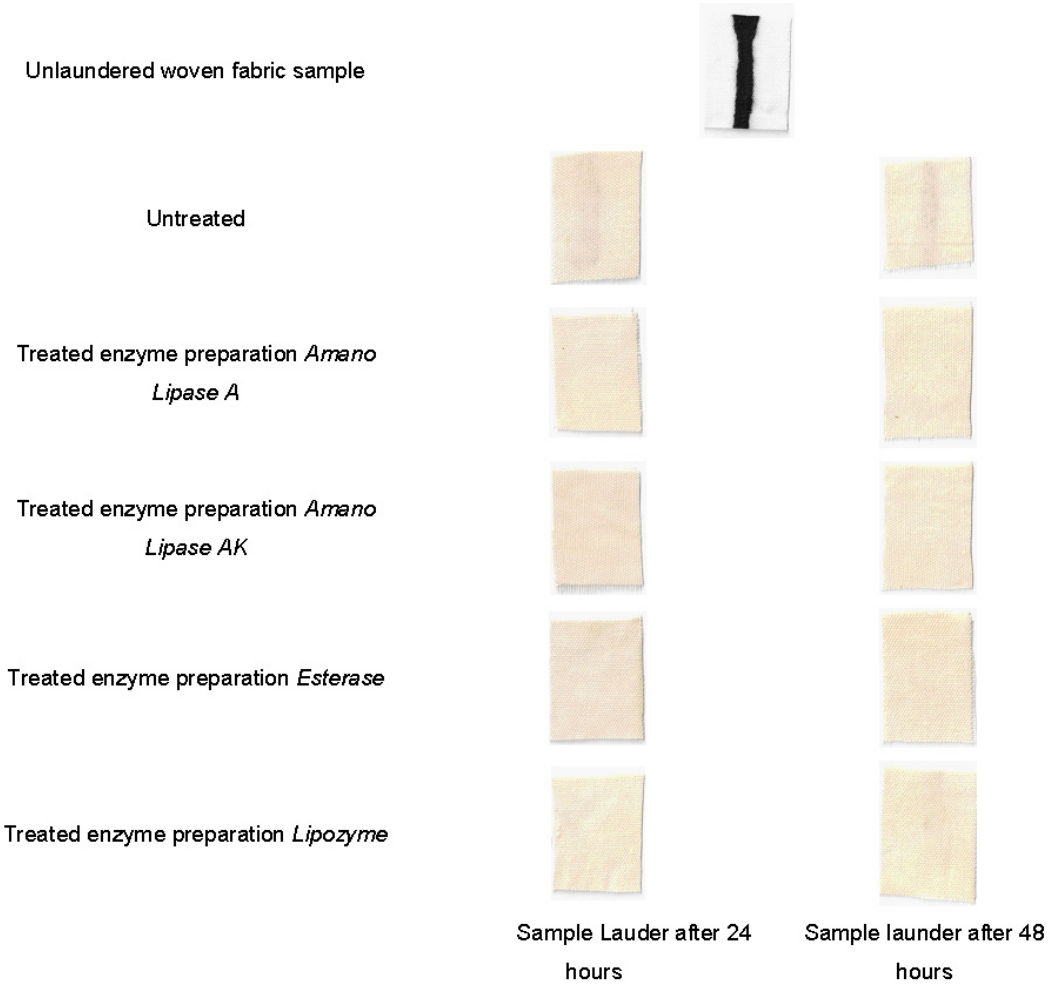


Figure 1. The results of testing the oil–soil-release woven fabric modified 120 min.

- under dry conditions (φ=25%), the effect of fibre modification leads to unbeneficial increase in the ρ_S value even by four orders of magnitude,
- under normal conditions (φ=65%), the effect of fibre modification results in the reduction in the ρ_S value by one order of magnitude,
- under wet conditions (φ=98%), the values of ρ_S are practically the same for both the unmodified and modified fibres.

Results of testing the fibre susceptibility to pilling

The results given in Table 8 indicate an effective action of the enzymatic preparations on fibres, which results in a practically complete elimination of the fibre susceptibility to pilling (degree 5).

Results of testing the oil–soil release

The results shown in Figure 1 indicate a practically complete oil–soil release of PET fibres modified with enzymatic preparations.

Conclusions

Based on the results obtained, one can draw the following conclusions:

- The change in the general surface characteristics of the fibres under investigation (micro-topography and hydrophilicity) results in very beneficial changes in both volumetric and surface properties.
- The change in the fibre surface structure due to the enzymatic modification fails to make significant changes in the fibre volumetric structure.
- The enzyme-modified fibres show high dyeing capabilities already at a temperature of 100°C.
- The enzyme-modified fibres are characterised by a totally reduced susceptibility to fabric pilling.
- The enzyme-modified fibres show a restricted ability to combine oily impurities and a high oil–soil-release capability.
- The enzyme-modified fibres show a surface resistivity decreased by one order of magnitude under normal conditions (RH = 65%).

In the light of the obtained test results, the method of PET fibre modification with the use of enzymatic preparations should be considered to be an effective, pro-ecological and energy-saving process for changing the fibre surface structure, leading to the elimination or a significant limitation of several unbeneficial properties of PET fibres.

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