The effect of accelerated ageing simulation on the properties of polyester fabrics

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Received September 26, 2023

UDC 677.019.394:677.494.674

Original scientific paper

In this work, the influence of artificial ageing on the properties of standard polyester fabrics was investigated. Since the actual test procedures are long-lasting, the natural ageing process is replaced in this work by artificial ageing in accordance with the ISO 105-B02:2013 standard. Standard polyester fabric is artificially aged under direct irradiation for 85 and 170 hours at a relative humidity of 40%. With the aim of gaining an insight into the properties of polyester fabrics during exposure to UV radiation, polyester fabrics were analysed throuhg physico-chemical, morphological-tactile and mechanical properties. The tensile properties decrease with ageing, which depends on the duration and time of exposure to irradiation. The FTIR analysis showed the possible effects of artificial ageing on aged fabrics in the wavelength range around 2900 cm-1, where oxidation of the C–H bond is most likely to occur. The photooxidative degradation of polyester fabrics starts at the surface of the fabric due to the slow diffusion of oxygen into the polymer structure, leading to changes in some physical properties, such as loss of gloss and roughness of the surface. With increasing exposure time, the breaking strength decreases, the mechanical properties become weaker and the stiffness of the fabric also decreases. It was found that the properties of the fabric in terms of its ability to manage moisture also change with different exposure times.

Keywords: *polyester fabric; accelerated ageing; UV radiation; FTIR; breaking force; FTT; MMT*

Izvorni znanstveni rad

U radu je praćen utjecaj umjetnoga starenja na svojstva standardne poliesterske tkanine. Budući da su stvarni procesi ispitivanja dugotrajni, u ovom radu je proces prirodnog starenja zamijenjen umjetnim starenjem prema normi HRN EN ISO 105-B02:2013. Standardna poliesterska tkanina je podvrgnuta izravnom djelovanju umjetnoga svjetla u vremenu od 85 i 170 sati, pri RH 40%. S ciljem dobivanja uvida u svojstva poliesterske tkanine tijekom izlaganja UV zračenju, poliesterske tkanine su analizirane preko fizikalno-kemijskih, morfološko-taktilnih i mehaničkih svojstava. Vlačna svojstva su starenjem smanjena, što ovisi o duljini i vremenu osunčavanja. FTIR analiza je pokazala na mogući učinak umjetnoga starenja na starene tkanine u području valne duljine oko 2900 cm^{-1} gdje se najvjerojatnije dogodila oksidacija na C–H vezi. Fotooksidacijska razgradnja poliesterskih tkanina započinje na površini tkanine zbog spore difuzije kisika u polimernu strukturu, što za posljedicu ima promjene nekih fizičkih svojstava, primjerice gubitak sjaja i hrapavost površine. Produljenjem vremena izlaganja smanjuje se otpornost na lom, oslabljuju mehanička svojstva, a izražena je i krutost tkanine. Utvrđeno je da se različitim vremenom izlaganja mijenjaju i svojstva tkanine sa stajališta sposobnosti upravljanja vlagom.

Ključne riječi: *poliesterska tkanina; ubrzano starenje; UV zračenje; FTIR; prekidna sila; FTT; MMT*

1. Introduction

The natural ageing of textiles is a complex process that takes place over a long period of time under the influence of various environmental factors such as irradiation, humidity, temperature, etc. [1, 2]. Tests under such conditions are often lengthy and require a lot of time, which is unacceptable in terms of obtaining data under controlled conditions. The changes that occur in textile materials are the result of a complex interaction between external influences and the structure of the fibres themselves. Even though some materials are more resistant to ageing than others and vice versa, it is a fact that every textile material degrades and changes over time. The simulation of accelerated ageing of textile materials is a method that predicts the effects of long-term exposure of materials to different conditions in a shorter period of time [2, 3]. This process under controlled conditions enables rapid evaluation and characterisation of materials and is therefore often used to obtain valuable data to ensure the longevity and quality of the product in use.

Comparing the results of accelerated ageing simulations with ageing under natural conditions can provide useful insights into the predictability of the behaviour of textile materials over time. However, it is important to note that simulations always have their limitations and that there may be differences between the results of the simulation and natural ageing. Therefore, it is sometimes necessary to carry out long-term tests in practise to confirm the long-term durability of textile materials.

Polyester fibres are synthetic fibres and can be made from poly(ethylene terephthalate) (PET), poly(butylene terephthalate) (PBT) and poly(1,4-dimethylenecyclohexyl terephthalate) (PCDT), depending on the raw material from which they are made. [4]. From a chemical point of view, they generally consist of long-chain polymers whose backbones are functional ester groups (-COO-). The largest proportion of polyester fibres in textiles in terms of chemical composition is PET [5-9]. It is becoming increasingly popular due to its good performance and structural properties such as strength and durability, crease, shrink and abrasion resistance. Production has doubled in the last 20 years and is estimated to reach 80 million tons by 2030 [10]. Polyester fibres are inherently hydrophobic, with a regain rate of 0.4% [11-13]. The reason for the hydrophobic property is the absence of polar groups (-COOH, -OH) in the polymer chain [14, 15] and the ability to easily attract contaminants such as oil, dust, etc. [6, 16]. It is precisely the properties of low absorbency and non-retention of moisture that make it a quick-drying textile material and easy to care. In blends with other fibres such as cotton and wool, they improve certain properties such as mechanical strength, dimensional and thermal stability [7, 16-18]. Textiles made from polyester fibres are prone to peeling, stiffening and static charge [12,19,20]. The negative impact on the environment is reflected in their production (from non-renewable sources) and low biodegradability. Recently, the negative impact on the environment due to the presence of micro-plastic (MP) fragments in water and air has been strongly emphasised, depending on the stages of the production process and the conditions of use and disposal [21-23]. In order to reduce the release of MP particles, it is therefore necessary to work on prevention in the long term.

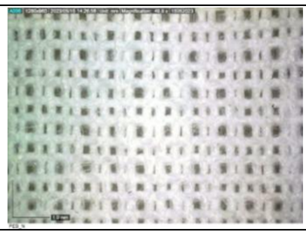
In this work, a process to simulate the artificial ageing of polyester fabrics under controlled laboratory conditions was carried out to quickly obtain data and monitor the effects of artificial ageing on the mechanical, physico-chemical and morphological-tactile properties of standard polyester fabrics.

2. Experimental

2.1. Material

A chemically bleached standard polyester fabric (PES_N) by CFT, Center for Testmaterials BV, Netherlands, was used for the research and its properties are listed in Tab.1.

Tab.1 Properties of standard PES fabric

Label of the fabric		PN-01
Colour		White
Mass per unit area (g/m^2)		156.0
Density (threads /cm)	warp	27.7
	weft	20.0
Thickness (mm)		0.35
Fineness (tex)	warp	30.4
	weft	31.9
Weave		Plain
Microscopic image of the sample, 50x magnification		

2.2. Preparation and processing of polyester fabric

To eliminate the influence of protruding threads from the edges of the material, an ultrasonic cutter, Ultrasonic Cutter TTS400, Sonowave, Italy, was used to prepare test samples of PES fabric. Fabrics of size 30x20 cm have been prepared.

The PES fabrics were exposed to artificial ageing simulation for 85 (85H) and 170 (170H) hours according to EN ISO 105-B02:2013: *Textiles — Tests for colour fastness, Part B02: Colour fastness to artificial light: Xenon arc fading lamp test* in the Xenotest 440 device, SDL Atlas. The conditions recommended for Europe (moderate temperature zone) were used to determine the exposure conditions. The PES fabric was exposed to direct UV radiation (UV-R) according to the standard at a relative humidity of 40% and a radiation of 42±2 W/m^2 in the wavelength range from 300 to 400 nm. The fabric is exposed to direct UV-R without rain (humidity).

The labels of standard PES fabric samples before and after artificial ageing are shown in Tab.2.

Tab.2 Labels of samples of a standard PES fabric

Samples	Description of polyester fabric
PES_N	Standard untreated polyester fabric
PES_85H	Standard polyester fabric artificially aged for 85 hours
PES_170H	Standard polyester fabric artificially aged for 170 hours

2.3. Methods

With the aim of monitoring the properties of standard and aged fabrics before and after the artificial ageing cycle, an analysis of the mechanical, physico-chemical and morphological-tactile properties was carried out.

The microscopic analysis of the standard and artificially aged PES fabrics was carried out using a Dino-Lite AM7013 Premier digital microscope with two magnifications, 50x and 250x.

The mass per unit area of the fabrics before and after irradiation was determined using a precise analytical balance Kern, model ALJ 220-5DNM with a weighing accuracy of ± 0.1 mg according to ISO 3801:1977 *Textile - Woven fabrics - Determination of mass per unit length and mass per unit area*.

The breaking force and elongation at break were performed on the Textechno H. Stein dynamometer, Textechno Statigraph. Three measurements were carried out per sample; with a tube length of 200 mm, a clamping distance of 100 mm, a preload of 5-7 N and a stretching speed of 100 mm/min.

Fourier transform infrared spectroscopy (FTIR) was used to characterise PES fabrics before and after the artificial ageing process.

The FTT (Fabric Touch Tester) SDL Atlas device was used to analyse the tactile properties of the PES samples by calculating the physical properties of the front and back of the fabric.

The measurement of moisture transfer through the sample was determined using the Moisture Management Tester MMT290, SDL Atlas. The classification of moisture transfer after modification by artificial ageing was carried out according to the AATCC 195-2017 *Liquid Moisture Management Properties of Textile Fabrics*.

The abrasion resistance was tested according to ISO 12947-2:2016 *Textiles - Determination of the abrasion resistance of fabrics by the Martindale method - Part 2: Determination of specimen breakdown* and ISO 12947-3:1998 *Textiles - Determination of the abrasion resistance of fabrics by the Martindale method - Part 3: Determination of mass loss* using Mesdan Lab instrument.

3. Results and discussion

The effect of artificial ageing of standard PES fabrics was observed using various methods: determination of changes in mass per unit area and tensile properties, analysis of functional groups by FTIR spectra, determination of moisture absorption capacity, wear resistance and monitoring of fabric characteristics through measuring touch and comfort.

The microscopic analysis of the PES samples is illustrated in Fig.1 by visualising the surfaces of standard and artificially aged PES fabrics at two magnifications. Using a Dino-Lite digital microscope, the pores of polyester fabrics can be clearly recognised, as well as the changes caused by artificial ageing (Fig.1). In the case of PES_N and PES_85H, no changes in the pore size of the fabrics can be recognised. In contrast, the fabric aged for 170 hours (PES_170H) shows clear and visible changes for both magnifications. The pores are more closed and less recognisable, which is a consequence of a certain degradation of the surface of the material.

In order to better understand the effects of artificial ageing on the properties of PES fabrics, an analysis of the changes in surface mass was carried out, Tab.3. The results show that direct exposure to UV-R causes certain changes and mass losses to a greater or lesser extent. The previously discussed results confirm that the artificial ageing process affects the slight degradation of fabrics. By prolonging the artificial ageing, surface changes of the yarn were observed; local thickening of individual fibres [24], which can

Tab.3 Mass per unit area (Q) and weight loss (ΔQ) of the artificially aged PES fabrics

Sample	Q (g/m^2)	ΔQ (%)
PES_N	164.08	-
PES_85H	160.35	2.27
PES_170H	164.06	0.12

Magnification 50x | Magnification 250x

PES_N

PES_85H

PES_170H

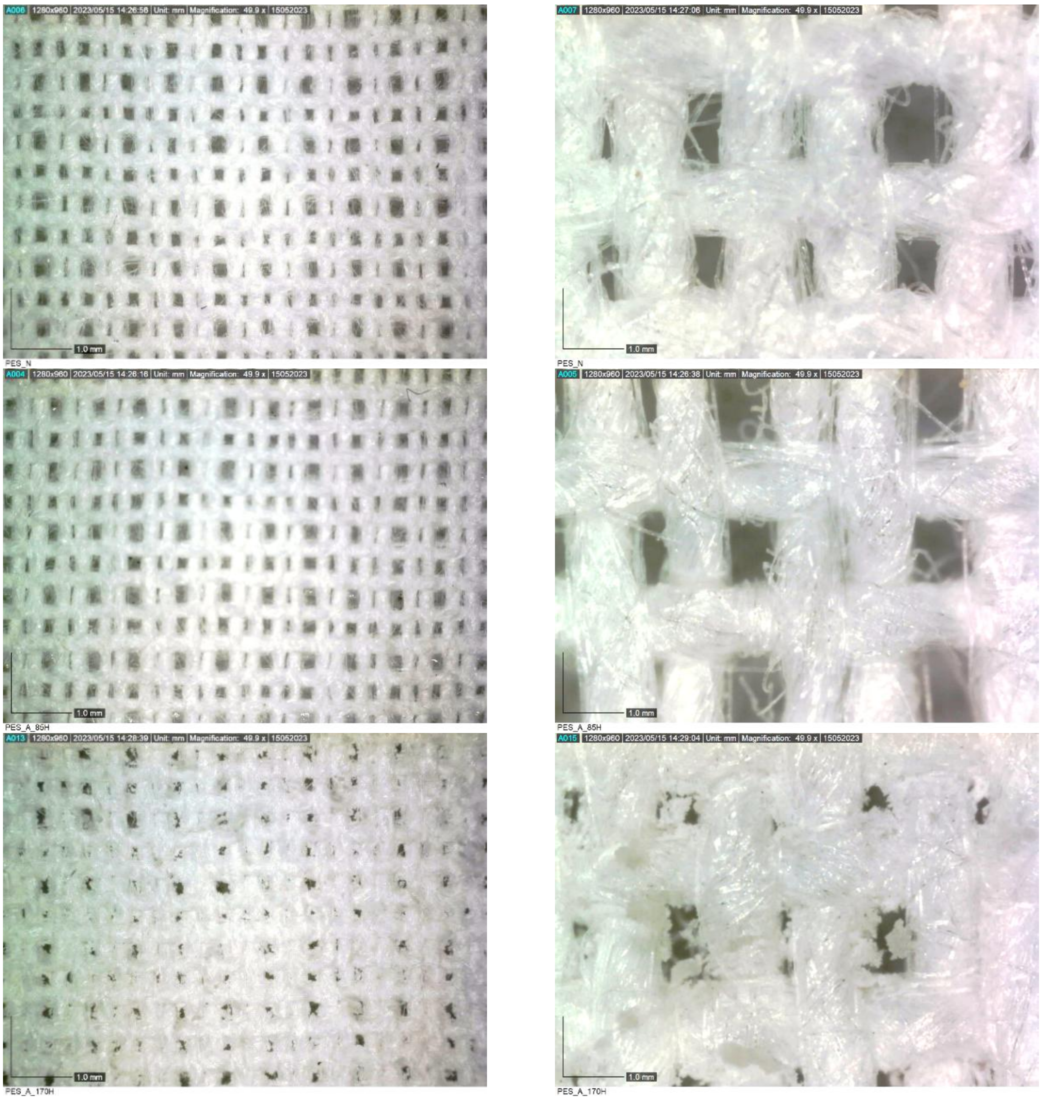


Fig.1 Digital microscope images of standard and artificially aged PES fabrics at 50x and 250x magnification

be attributed to a slight increase in mass compared to PES_85H. The reason for the local thickening of polyester fibres during ageing is the phenomenon known as *annealing*, in which the molecules in the polymer chains are rearranged, which leads to changes in the structure and properties of the fibres. The relaxation of the molecules and the redistribution within the polymer chains led to an increase in the thickness and diameter of polyester fibres, which is in accordance with some previous studies [25, 26].

The tensile properties are important for analysing the structural properties. In this work, they were analysed with two exposure times. Tab 3 shows the values for breaking force and elongation before and after the ageing process.

According to the results in Tab. 4, the standard PES fabric has a breaking force (F_b) of 945.08 N, which shows a good arrangement of the polymer structure.

The process of artificial ageing leads to changes in the mechanical properties, which is confirmed by the breaking force values (650.28 N after 85 hours of irradiation and 456.05 N after 170 hours of irradiation).

Tab.4 Breaking force (F_b) and elongation (ε_b) PES fabrics

Samples		PES_N	PES_85H	PES_170H
F_b (N)	1	976.66	654.70	506.83
	2	936.11	656.13	474.61
	3	922.47	640.02	386.72
	$\bar{x}$	945.08	650.28	456.05
ε_b (%)	1	48.02	47.01	45.79
	2	47.88	49.95	43.91
	3	48.02	47.01	38.32
	$\bar{x}$	47.97	47.99	42.67
ΔF (%)		-	31.19	51.74

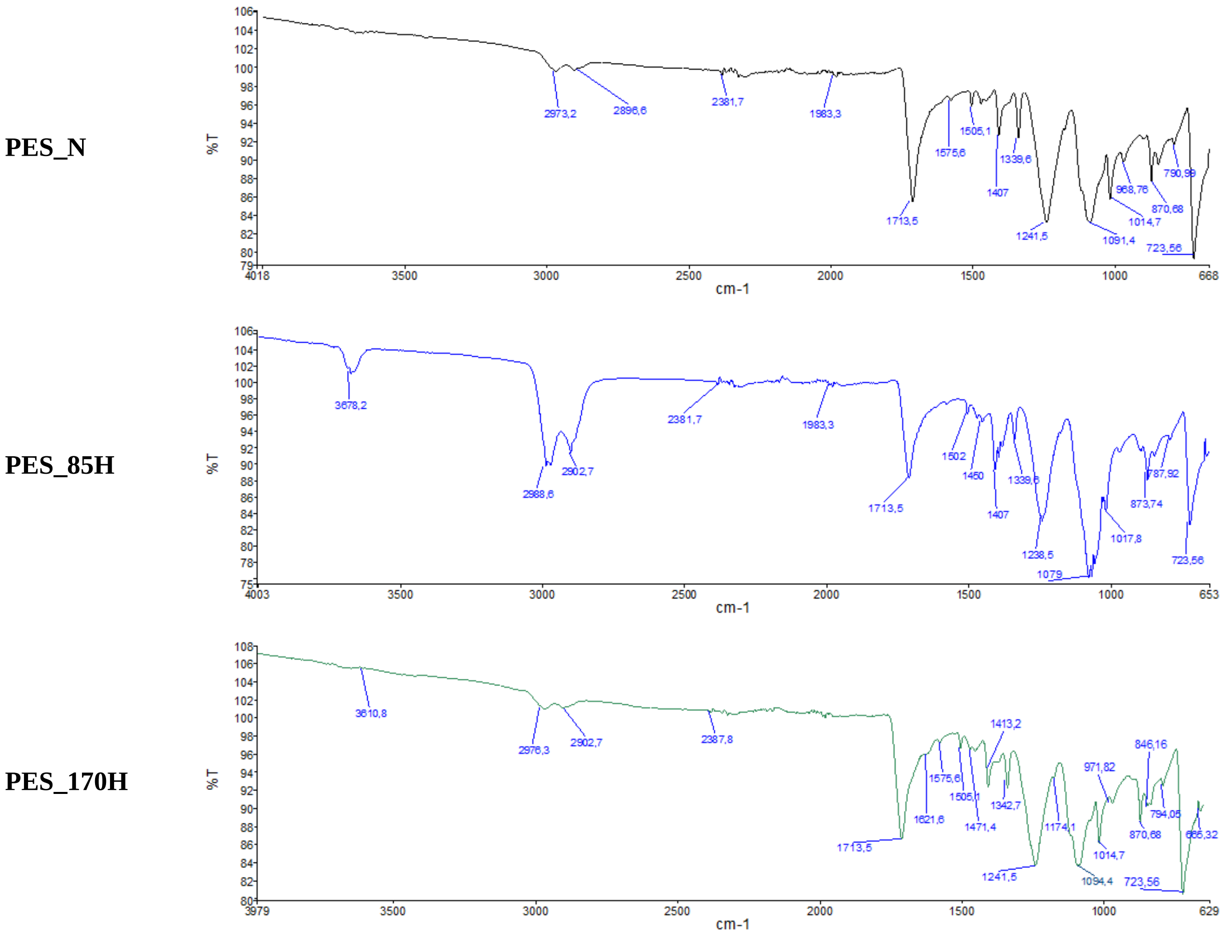


Fig.2 FTIR spectra of the samples

Artificial ageing led to a reduction of the breaking force by 31.19% within 85 hours and doubled after 170 hours (51.74%). Prolonged ageing leads to changes in the tensile properties, as it not only causes a slight degradation of the yarn in the form of slightly protruding fibres and local thickening, but also increases the brittleness of the polyester fabric.

The FTIR spectra confirm the fabric structure and the new groups that formed during and after irradiation, Fig.2. The beginning of the band indicates the functional groups, in this case the stretching of the -OH group of the aged PES fabric with a characteristic peak of 3678.2 cm^{-1}. No -OH group can be detected in the standard PES fabric. The peaks for polyester are visible in the FTIR bands: C=O 1713.5 cm^{-1}; 1407 cm^{-1}; 1241.5 cm^{-1}; 1091.4 cm^{-1}; 870.66 cm^{-1}. The peak at 1505.19 cm^{-1} is characteristic for an aromatic ring. Changes are observed in the region around 2900 cm^{-1} where oxidation occurs at the C–H bond [27-29]. In the PES fabric band aged for 170 hours, slight changes in the peaks in the region around 970 cm^{-1} are visible. Since FTIR provides information about changes in chemical composition and structure, the obtained spectra show that no significant changes in chemical composition occurred after the artificial ageing of 170 hours.

The assessment of the tactile properties was developed throung the analysis of the properties of the test fabrics on the FTT device, the characteristics of which are listed in Tab.5. The tactile properties of PES samples are determined by analysing the physical properties of the top and back of the fabric. In addition feel to the touch, the overall comfort of the fabric was also evaluated, such as softness, warmth and smoothness. The advantages of the FTT device are that it objectively assesses the quality of the samples and measures 18 indices for the specified properties (compression, thermal properties, bending, thickness, stiffness, surface roughness, friction,). Due to the innovative design of the device, it is possible to measure all surface and mechanical properties of the samples with a single test - softness, smoothness, warmth and the total touch feel.

Tab.5 Characteristics of the FTT device

Bending	BAR – Bending Avergae Rigidity
	BW – Bending Work
Compression	CW – Compression Work
	CRR – Compression Recovery Rate
	CAR – Compression Average Rigidity
	RAR – Recovery Average Rigidity
	T – Thickness

Tab.6 Bending and compression of standard and aged polyester fabrics

Samples	Bending				Compression				
	BARa	**BARe**	**BWa**	**BWe**	**T**	**CW**	**CRR**	**CAR**	**RAR**
	gF mm/rad				mm	gf mm		gf/(cm^{2}mm)	
PES_N	0.20	80.67	8.26	633.02	0.39	787.19	0.38	387.09	2289.19
PES_85H	3.29	113.16	13.46	1010.06	0.34	331.65	0.66	999.46	3811.01
PES_170H	2.11	195.61	10.81	1682.34	0.38	539.80	0.53	733.88	2037.83

*a-warp; e-weft direction

Standard PES fabric has a certain bending stiffness, which changes during artificial ageing, Tab.6. In the warp direction, the mean value during bending (BARa) is 0.20 gF mm/rad. In the weft direction it is 633.02 gF mm/rad (BARe). As far as the effects of artificial ageing are concerned, an increase in the mean value can be observed in both directions. The bending increases after artificial ageing, the instrumental results indicate that more work is required for bending, while subjectively the stiffness of the material is also observed, which means that the resilience of the fabric is weaker. The analysis parameter BW, the bending work, is numerically proportional in warp and weft direction - it increases in both warp and weft direction. The thickness of the standard PES fabric (0.39 mm) decreased slightly after artificial ageing, while the value of the compression recovery rate (CRR) increased under the influence of artificial irradiation. Artificial ageing after 85 hours increases the value, while prolonging the irradiation (170H) decreases it, but it is still higher than that of the standard fabric.

Tab.7 FTT results for standard and aged polyester fabrics

Samples	Smoothness	Softness	Warmth	Total Touch Feel
PES_N	5.0	4.0	4.0	4.0
PES_85H	4.0	3.0	3.0	3.0
PES_170H	3.0	3.0	3.0	3.0

The total touch feel is rated 4.0 for the standard PES fabric, while the aged fabric is rated 3.0. The modification by artificial ageing affects the structural properties, and the resulting MMT value indicates the ability to manage moisture.

To determine the moisture management of standard and aged PES fabrics, measurements were carried out according to AATCC TM 195-2017, Tab.8. The results obtained are expressed as mean values with the standard deviation (σ) and coefficient of variation (CV) for each individual property: Absorption Rate (AR), Maximum Wetted Radius (MWR), Wetting Time (WT), Top surface (T) and Bottom surface (B), Spreading Speed (SS), Accumulative One-way Transport Capability (R) and Overall Moisture Management Capability (OMMC), [30, 31].

The wetting time (WT) indicates the time required for the top and bottom surfaces of the test specimen to start wetting. The results show that for the standard PES fabric it is 3.1667 s for the upper side and 3.2917 s for the lower side. With increasing ageing, the wetting time decreases; for PES_85H it is 2.6930 s for the upper side, and 2.7867 s for the lower side. The fabric PES_170H gives values of 2.6873 s for the upper side, and 2.4060 s for the lower side. The reason for the slightly longer wetting time for PES_N lies in the fact, confirmed by previous studies [32, 33], that the standard fabric contains small amounts of silicone-based preparations, the presence of which contributes to the hydrophobic properties of the PES_N sample.

Tab.8 Moisture management properties of PES fabrics

Uzorci		WT		AR		MWR		SS		R (%)	OMMC
		T (s)	**B (s)**	**T (%/s)**	**B (%/s)**	**T (mm)**	**B (mm)**	**T (mm/s)**	**B (mm/s)**		
PES_N	$\bar{x}$	3.1667	3.2917	34.4271	41.1210	20.00	21.66	3.9519	4.0047	202.5640	0.6120
	σ	0.2287	0.1801	12.2941	17.6693	0	2.88	0.1006	0.1739	5.5194	0.0510
	CV	0.0722	0.0547	0.3571	0.4297	0	0.13	0.0255	0.0434	0.0272	0.0830
PES_85H	$\bar{x}$	2.6930	2.7867	42.4327	45.2494	28.33	30.00	4.7087	4.8121	173.4659	0.5982
	σ	0.1894	0.2850	4.5773	7.1913	2.89	0	0.5782	0.5083	58.9471	0.0698
	CV	0.0703	0.1023	0.1079	0.1589	0.10	0	0.1228	0.1056	0.3283	0.1171
PES_170H	$\bar{x}$	2.6873	2.4060	61.2508	67.7321	30.00	30.00	5.4423	5.7808	39.9414	0.5103
	σ	0.1080	0.1430	1.4358	1.1797	0	0	0.1392	0.2278	8.9739	0.0067
	CV	0.0402	0.0595	0.0234	0.0174	0	0	0.0256	0.0394	0.2247	0.0131

Tab.9 The change in mass of PES fabrics depending on different number of rubbing cycles

Samples	m (g)*	m_1 (g)	m (g)*	m (g)*	m (g)*	m (g)*	m (g)*	m (g)*
Cycles			**10 000**	**20 000**	**30 000**	**40 000**	**50 000**	**60 000**
PES_N	164.6252	0.1808	164.6252	164.6243	164.6241	164.5890	164.5847	164.5836
PES_85H	164.7942	0.1750	164.7941	164.7950	164.7943	164.6216	-	-
PES_170H	164.7791	0.1879	164.6602	164.6557	164.6401	-	-	-

*m - mass of the sample + holder in grammes, m_1 – mass of the sample in grammes

The overall moisture management capability of PES_N is 0.6120, while it is 0.5982 for 85 hours and 0.5103 for 170 hours. The accumulative one-way transfer capacity of standard polyester fabric is 202.564% and decreases during artificial ageing. For a fabric that has been aged for 85 hours, it is 173.4659% and with further ageing (170 hours) the value drops to 39.9414%. Artificial ageing has an effect on the moisture management ability of the material.

The results of abrasion resistance, Tab.9, show the cycle in which the first strand of the sample breaks (indicated in red) and the resulting mass loss.

A standard PES fabric breaks after 60 000 cycles with a mass loss of 0.03%. Within PES_85H, the first thread breaks after 40 000 cycles with a mass loss of 0.10%. The fabric aged for 170 hours shows an almost identical mass loss (0.11%), but the breakage occurs at a cycle of 30 000 number of rubs, which also confirms the negative effects of artificial ageing on the structure of the warp and weft threads in the fabric.

4. Conclusion

The effect of accelerated ageing simulation on the properties of PES fabric was investigated under the exposure conditions commonly used in Europe (moderate temperature zone). The exposure time of 85 and 170 hours corresponds approximately to the exposure time of 850 and 1700 hours under natural conditions and had an effect on the changes in the properties of PES fabrics. At the molecular level, ageing leads to oxidation of the polymer chain, the breaking of bonds and the formation of new molecules. In aged fabrics, the stiffness of the material increases, the tensile properties decrease in proportion to the duration of exposure, the threads break faster with a lower number of cycles and with a greater loss of mass, which has a negative effect on the warp and weft threads. A change is visible in the region around 2900 cm^{-1}, where oxidation has most likely taken place at the C-H bond. Slight changes in the peaks in the region around 970 cm^{-1} can be recognised in the polyester fabric band aged for 170 hours.

From the point of view of comfort and moisture management ability, the standard PES fabric is classified as a "moisture management fabric" (rate 3), the aged (85 hours) is rated 4 and defined as a "moisture management fabric" and the aged (170 hours) as a "fast absorbing and fast drying fabric", rate 3. Irradiation causes photooxidative decomposition of polyester, which initially starts at the surface of the material due to the slow diffusion of oxygen into the polymer. As the decomposition is initially limited to the surface layer, it leads to changes in physical properties such as surface roughness and loss of lustre. Over time, the breaking strength decreases and the mechanical properties become weaker.

Acknowledgements

The work of PhD student Ana Šaravanja was partly developed and co-financed by HrZZ DOK-2021-02-6750 "Young researchers' career development project – training of doctoral students" and partly by HrZZ-IP-2020-02-7575, InWaShed-MP of the Croatian Science Foundation.

Part of the experimental results were achieved through a two-month Erasmus+ internship at the University of Maribor, Faculty of Mechanical Engineering, Maribor, Slovenia.

The part of the research was performed on equipment purchased by K.K.01.1.1.02.0024

project "Modernization of Textile Science Research Centre Infrastructure" (MI-TSRC).

Part of the results presented in this article originate from the project task "The effect of accelerated ageing simulation on the properties of polyester fabric", which was prepared as part of the postgraduate doctoral study Textile Science and Technology at the University of Zagreb Faculty of Textile Technology in the academic year 2022/2023, and accepted (25 September 2023) by the Doctoral Study Council of Textile Science and Technology at the Faculty of Textile Technology.

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