

# tekstilec

1/2021 • vol. 64 • 1–80

ISSN 0351-3386 (tiskano/printed)

ISSN 2350-3696 (elektronsko/online)

UDK 677 + 687 (05)





<http://www.tekstilec.si>

Časopisni svet/*Publishing Council*

**Barbara Simončič**, predsednica/*President*

**Katja Burger**, Univerza v Ljubljani

**Silvo Hribernik**, Univerza v Mariboru

**Tatjana Kreže**, Univerza v Mariboru

**Gašper Lesjak**, Predilnica Litija, d. o. o.

**Nataša Peršuh**, Univerza v Ljubljani

**Petra Prebil Bašin**, Gospodarska zbornica

Slovenije

**Melita Rebič**, Odeja, d. o. o.

**Tatjana Rijavec**, Univerza v Ljubljani

**Daniela Zavec**, ZITTS

**Helena Zidarič Kožar**, Inplet pletiva d. o. o.

**Vera Žlabravec**, Predilnica Litija, d. o. o.

Glavna in odgovorna urednica/  
*Editor-in-Chief*

**Tatjana Rijavec**

Namestnica glavne in odgovorne  
urednice/*Assistant Editor*

**Tatjana Kreže**

Področni uredniki/*Associate Editors*

**Matejka Bizjak, Katja Burger, Andrej**

**Demšar, Alenka Pavko Čuden, Andreja**

**Rudolf, Barbara Simončič, Sonja Šterman,**

**Brigita Tomšič, Zoran Stjepanović**

Izvršna urednica za podatkovne baze/  
*Executive Editor for Databases*

**Irena Sajovic**

Mednarodni uredniški odbor/  
*International Editorial Board*

Arun Aneja, Greenville, US

Andrea Ehrmann, Bielefeld, DE

Aleš Hladnik, Ljubljana, SI

Petra Forte Tavčer, Ljubljana, SI

Darinka Fakin, Maribor, SI

Jelka Geršak, Maribor, SI

Karl Gotlih, Maribor, SI

Memon Hafeezullah, Shanghai, CN

Abu Naser Md. Ahsanul Haque, Daka, BD

Geelong, AU

Ilda Kazani, Tirana, AL

Svetlana Janjić, Banja Luka, BA

Igor Jordanov, Skopje, MK

Petra Komarkova, Liberec, CZ

Mirjana Kostić, Beograd, RS

Manja Kurečič, Maribor, SI

Rimvydas Milasius, Kaunas, LT

Olga Paraska, Khmelnytskyi, UA

Irena Petrinić, Maribor, SI

Željko Penava, Zagreb, HR

Tanja Pušić, Zagreb, HR

Zenun Skenderi, Zagreb, HR

Snežana Stanković, Beograd, RS

Jovan Stepanović, Leskovac, RS

Zoran Stjepanović, Maribor, SI

Simona Strnad, Maribor, SI

Jani Toroš, Ljubljana, SI

Mariana Ursache, Iai, RO

Antoneta Tomljenović, Zagreb, HR

Dušan Trajković, Leskovac, RS

Hidekazu Yasunaga, Kyoto, JP

**tekstilec** (ISSN: 0351-3386 tiskano, 2350-3696 elektronsko) je znanstvena revija, ki podaja temeljne in aplikativne znanstvene informacije v fizikalni, kemijski in tehnološki znanosti, vezani na tekstilno in oblačilno tehnologijo, oblikovanje in trženje tekstilij in oblačil. V prilogah so v slovenskem jeziku objavljeni strokovni članki in prispevki o novostih v tekstilni tehnologiji iz Slovenije in sveta, prispevki s področja oblikovanja tekstilij in oblačil, informacije o raziskovalnih projektih ipd.

**tekstilec** (ISSN: 0351-3386 printed, 2350-3696 online) the scientific journal gives fundamental and applied scientific information in the physical, chemical and engineering sciences related to the textile and clothing industry, design and marketing. In the appendices written in Slovene language, are published technical and short articles about the textile-technology novelties from Slovenia and the world, articles on textile and clothing design, information about research projects etc.

Dosegljivo na svetovnem spletu/*Available Online at*  
[www.tekstilec.si](http://www.tekstilec.si)



Tekstilec je indeksiran v naslednjih bazah/*Tekstilec is indexed in*  
Emerging Sources Citation Index – ESCI (by Clarivate Analytics)  
Leiden University's Center for Science & Technology Studies (2019: SNIP 0.496)  
SCOPUS/Elsevier (2019: Q3, SJR 0.19, Cite Score 0.45, H Index 10)  
Ei Compendex  
DOAJ  
WTI Frankfurt/TEMA® Technology and Management/TOGA® Textile Database  
World Textiles/EBSCO Information Services  
Textile Technology Complete/EBSCO Information Services  
Textile Technology Index/EBSCO Information Services  
Chemical Abstracts/ACS  
ULRICHWEB – global serials directory  
LIBRARY OF THE TECHNICAL UNIVERSITY OF LODZ  
dLIB  
SICRIS: 1A3 (Z, A', A1/2); Scopus (d)

# tekstilec

## Ustanovitelj / *Founded by*

- Zveza inženirjev in tehnikov tekstilcev Slovenije /  
*Association of Slovene Textile Engineers and Technicians*
- Gospodarska zbornica Slovenije – Združenje za tekstilno,  
oblačilno in usnjarsko predelovalno industrijo /  
*Chamber of Commerce and Industry of Slovenia – Textiles,  
Clothing and Leather Processing Association*

## Revijo sofinancirajo / *Journal is Financially Supported*

- Univerza v Ljubljani, Naravoslovnotehniška fakulteta /  
*University of Ljubljana, Faculty of Natural Sciences and Engineering*
- Univerza v Mariboru, Fakulteta za strojništvo /  
*University of Maribor, Faculty for Mechanical Engineering*
- Javna agencija za raziskovalno dejavnost Republike Slovenije /  
*Slovenian Research Agency*

## Izdajatelj / *Publisher*

Univerza v Ljubljani, Naravoslovnotehniška fakulteta /  
*University of Ljubljana, Faculty of Natural Sciences and Engineering*

## Sponzor / *Sponsor*

Predilnica Litija, d. o. o.

Revija Tekstilec izhaja štirikrat letno /  
*Journal Tekstilec appears quarterly*

Revija je pri Ministrstvu za kulturo vpisana v  
razvid medijev pod številko 583. Letna naroč-  
nina za člane Društev inženirjev in tehnikov  
tekstilcev je vključena v članarino.

Letna naročnina za posameznike 38 €  
za študente 22 €  
za mala podjetja 90 € za velika podjetja 180 €  
za tujino 110 €

Cena posamezne številke 10 €

Na podlagi Zakona o davku na dodano  
vrednost sodi revija Tekstilec med proizvode,  
od katerih se obračunava DDV po stopnji 5 %.

Transakcijski račun 01100–6030708186  
Bank Account No. SI56 01100–6030708186  
Nova Ljubljanska banka d. d.,  
Trg Republike 2, SI–1000 Ljubljana,  
Slovenija, SWIFT Code: LJBA SI 2X.

## Naslov uredništva / *Editorial Office Address*

Uredništvo Tekstilec, Snežniška 5, SI–1000 Ljubljana  
Tel./Tel.: + 386 1 200 32 00, +386 1 200 32 24  
Faks/Fax: + 386 1 200 32 70  
E–pošta/E–mail: [tekstilec@ntf.uni-lj.si](mailto:tekstilec@ntf.uni-lj.si)  
Spletni naslov / *Internet page*: <http://www.tekstilec.si>

Lektor za slovenščino / *Slovenian Language Editor* Milojka Mansoor

Lektor za angleščino / *English Language Editor* Tina Kočevar Donkov,  
Glen David Champaigne, Barbara Luštek Preskar

Oblikovanje platnice / *Design of the Cover* Tanja Nuša Kočevar

Oblikovanje / *Design* Miha Golob

Oblikovanje spletnih strani / *Website Design* Jure Ahtik

Tisk / *Printed by* PRIMITUS, d. o. o.

Copyright © 2021 by Univerza v Ljubljani, Naravoslovnotehniška fakulteta,  
Oddelek za tekstilstvo, grafiko in oblikovanje

Noben del revije se ne sme reproducirati brez predhodnega pisnega dovoljenja  
izdajatelja/*No part of this publication may be reproduced without the prior  
written permission of the publisher.*

SCIENTIFIC  
ARTICLES/  
*Znanstveni članki*

- 4** *Veronika Štampfl, Klemen Možina, Jure Ahtik*  
Different Textile Materials as Light Shaping Attachments in Studio Photography and Their Influence on Colour Reproduction  
*Različni tekstilni materiali kot nastavki za oblikovanje svetlobe v studijski fotografiji in njihov vpliv na reprodukcijo barv*
- 16** *Selestina Gorgieva, Darinka Fakin, Alenka Ojstršek*  
Confocal Fluorescence Microscopy as a Tool for Assessment of Photoluminescent Pigments Print on Polyester Fabric  
*Konfokalna fluorescenčna mikroskopija kot orodje za ovrednotenje tiska fotoluminiscenčnih pigmentov na poliestrni tkanini*
- 25** *Bosiljka Šaravanja, Tanja Pušić, Krešimir Malarić, Anica Hursa Šajatović*  
Durability of Shield Effectiveness of Copper-Coated Interlining Fabrics  
*Obstojnost elektromagnetne zaščite tkanin, prevlečenih z bakreno oblogo*
- 32** *Thomas Grethe*  
Biodegradable Synthetic Polymers in Textiles – What Lies Beyond PLA and Medical Applications? A Review.  
*Biorazgradljivi sintetični polimeri v tekstilstvu – kaj sledi PLA in medicinskim načinom uporabe? Pregled.*
- 47** *Madan Lal Regar, Akhtarul Islam Amjad, Shubham Joshi*  
Effect of Solvent Treatment on Siro and Ring Spun TFO Polyester Yarn  
*Vpliv obdelave s topilom na poliestrni preji, izdelani po postopku SiroSpun® in prstanskem postopku*
- 55** *Shubham Joshi, Vinay Midha, Subbiyan Rajendran*  
Investigation of Durable Bio-polymeric Antimicrobial Finishes to Chemically Modified Textile Fabrics Using Solvent Induction System  
*Raziskava trajnih biopolimernih protimikrobnih apretur za kemijsko plemenitenje tekstilij z uporabo indukcijskega sistema s topilom*
- 70** *Md. Reazuddin Repon, Nure Alam Siddiquee, Mohammad Abdul Jalil, Daiva Mikučionienė, Md. Rezaul Karim, Tarikul Islam*  
Flame Retardancy Enhancement of Jute Fabric Using Chemical Treatment  
*Izboljšanje ognjevarnosti jutne tkanine s kemično obdelavo*

Veronika Štampfl, Klemen Možina, Jure Ahtik

University of Ljubljana, Faculty of Natural Sciences and Engineering, Aškerčeva 12, 1000 Ljubljana, Slovenia

---

# Different Textile Materials as Light Shaping Attachments in Studio Photography and Their Influence on Colour Reproduction

*Različni tekstilni materiali kot nastavki za oblikovanje svetlobe v studijski fotografiji in njihov vpliv na reprodukcijo barv*

Original scientific article/Izvorni znanstveni članek

Received/Prispelo 9-2020 • Accepted/Sprejeto 11-2020

Corresponding author/Korespondenčni avtor:

Assist Prof dr. Jure Ahtik

E-mail: jure.ahtik@ntf.uni-lj.si

ORCID: 0000-0002-0212-4897

---

## Abstract

The research focuses on the quality of colour reproduction when using different light sources, often used to illuminate scenes in a photo studio, and different types of fabrics as lighting shapers. With the latter, the light can be converted into softer and more diffuse light, but the properties of the fabrics used affect the colour impression and thus the quality of the reproduced colours. This was evaluated by analysing the colour differences which were calculated from the colorimetric values of the colour patches of the X-Rite ColorChecker Passport test chart. Test chart was photographed in a controlled environment and illuminated with different combinations of light sources and tested fabrics. The results confirmed that not all combinations of variables are suitable for use if the goal of the photograph is to achieve high quality colour reproduction.

Keywords: photography, light, fabrics, colour reproduction, light shaping attachments

## Izvleček

Raziskava se osredinja na kakovost barvne reprodukcije pri uporabi različnih svetlobnih virov, ki se pogosto uporabljajo za osvetljevanje scen v fotografskem studiu, in različnih vrst tkanin kot nastavkov za oblikovanje svetlobe. S temi lahko svetlobo preoblikujemo v mehkejšo in bolj razpršeno, vendar lastnosti uporabljenih tkanin vplivajo na barvni vtis in posledično na kakovost reproduciranih barv. Le-to smo vrednotili z analizo barvnih razlik, ki smo jih izračunali iz odčitanih kolorimetričnih vrednosti barvnih polj testne tablice X-Rite ColorChecker Passport. Ta je bila fotografirana v nadzorovanem okolju in izpostavljena različnim kombinacijam svetlobnih virov ter testiranih tkanin. Rezultati potrjujejo, da niso vse kombinacije spremenljivk primerne za uporabo, če je cilj fotografiranja kakovostna barvna reprodukcija. Ključne besede: fotografija, svetloba, tkanine, reprodukcija barv, nastavki za oblikovanje svetlobe

---

## 1 Introduction

For photography, light is of crucial importance as it is needed for the ability to represent a subject/object. In a photo studio, the photographer himself controls the

various properties of the light emitted by the lights on stage. These are usually the intensity and colour temperature, but we can also control other properties, such as softness and dispersion of the light beam, which is achieved by using light shaping attachments.

These attachments can be of various shapes, which affect the angle of the dispersed light. The consequence of the attachment's material is the quality of light, it being soft or hard [1]. Often used attachments are softboxes, which soften the original light by transmitting it through a white fabric.

Studio lighting continues to evolve with technological progress. Today, xenon studio flashes (stroboscopic lights) and continuous LED lights are increasingly used in studio photography. Unlike halogen lamps, they do not generate high temperatures between 400 °C and 1000 °C [2]. By reducing the operating temperatures of the lights, it is no longer necessary to use special fabrics with a high melting point to soften the light. Instead, fabrics of organic origin can be used, whose structure begins to change at temperatures around 150 °C [3]. We can also use polymer fabrics with lower melting points, since the working temperatures of available lights are now lower and will not melt the fabrics.

With a wider range of light shaping attachments, the amount of final light shapes also increases. By using light shaping attachments we transform also the colour of the original light emitted by the light source. Therefore, the light illuminating the scene cannot be described precisely.

This research focuses on the quality of colour rendering and correlation between five different lighting conditions and the use of five different types of fabrics as light shaping attachments. The findings of this research will benefit the understanding and describing light used in researches, since no prior research has been found addressing the problem of the effects of fabrics as light shaping objects on the quality of colour reproduction.

The study included three types of lights that are commonly used to illuminate the photographic scene, but are very different from each other, since they emit light of different intensity and colour temperature while producing different amounts of heat.

A photographic studio flash that illuminates the scene with a xenon lamp was used. Its light flashes are extremely short therefore this type of lighting does not involve high operating temperatures. The second type of light source that does not produce high temperatures are LED lights (light-emitting diodes), with which colour temperature and light intensity can be adjusted. The third type of lighting is the illumination with a tungsten halogen lamp (hereinafter referred to as halogen lamp). This type of light source generates higher temperatures during illumination, so when staging

the scene, we must pay attention to the distance of the flammable materials from the light head [2].

Textile fibres rarely melt, as their change at high temperatures does not necessarily mean a change in physical state from solid to liquid. Exposure to a suitable temperature ensures a change in the crystalline structure, which is also referred to as melting in the literature [4]. Synthetic fibres such as polyamide and polyester therefore have a melting point at temperatures between 223 °C and 270 °C, depending on the properties of the fibre polymers. Natural fibres, such as cotton, linen and wool, do not melt but carbonize at a certain temperature, i.e. such changes begin to show at temperatures around 150 °C [4, 3].

In order to compare the influence of natural and synthetic fabrics on the quality of colour reproductions when used as light shaping attachments, five types of fabric were tested, made entirely from one of the five fibre types, cotton, linen, wool, polyamide or polyester. A series of natural and synthetic fibres has been chosen, since it has been studied that light reflects differently depending on the fibre structure [5]. A colour test chart has been illuminated using five different lighting conditions with alternating use of five fabrics as light shapers. Photographs of the scene were measured and the quality of colour reproduction assessed.

## 2 Experimental

For research purposes, a special cube that allows control over the tested light and limits the influence of its reflections has been created. With five different lights, the colour chart has been photographed and then the process repeated five more times, changing the fabric that served as the light shaping attachment. The combinations of variables and their labels are shown in Table 1.

Each combination of variables was photographed three times, making a total of 90 photos. By adjusting the whiteness and brightness of the photos, the variables that would affect the colour differences caused by using different light shaping attachments, in this case fabrics, were eliminated. After these adjustments, the RGB values of all the colour patches were measured. The average values measured from three photos of the same combination of variables were converted to CIELAB values, and then the colour differences between the values without and with fabric as the light shaping attachment were calculated.

Table 1: Combination of fabrics and lightning conditions used in the research and their labels

Fabric	Xenon	Halogen	LED 3230 K	LED 5000 K	LED 6260 K
Cotton (CO)	X/CO	H/CO	L3/CO	L5/CO	L6/CO
Linen (LI)	X/LI	H/LI	L3/LI	L5/LI	L6/LI
Wool (WO)	X/WO	H/WO	L3/WO	L5/WO	L6/WO
Polyamide (PA)	X/PA	H/PA	L3/PA	L5/PA	L6/PA
Polyester (PES)	X/PES	H/PES	L3/PES	L5/PES	L6/PES

## 2.1 Scene

### The test cube

The photographs were taken in a photo studio where there is no influence of outside light. A 60 cm cube was made, lined on the inside with black felt (Figures 1 and 2), in order to prevent the reflectance of the light, which could affect the reproduction of the colour of the test chart.

There are two round holes 20 cm in diameter in the centres of the two opposite sides of the cube, which serve as openings for illuminating the test chart (Figure 1a). This ensures that the angle of the incident light on the test plate is always constant. Test chart was mounted on the holder in such a way that it was always evenly illuminated from the left and right at an angle of 45° (Figure 1b).

The tested light source was 30 cm from the sides of the cube and positioned so that the centres of the light source were aligned with the centres of the round cut-outs on opposite sides.

If a light shaping attachment was used, it was placed on the side of the outer part of the cube so that it completely and evenly covered the opening in the side (Figure 1c).

At the level of the illumination holes, there is an additional hole with a diameter of 10 cm which allows the camera to be mounted so that the lens reaches into the cube (Figure 1d). This limits the possibility of additional reflections in the lens and prevents the capture of light outside the test area.

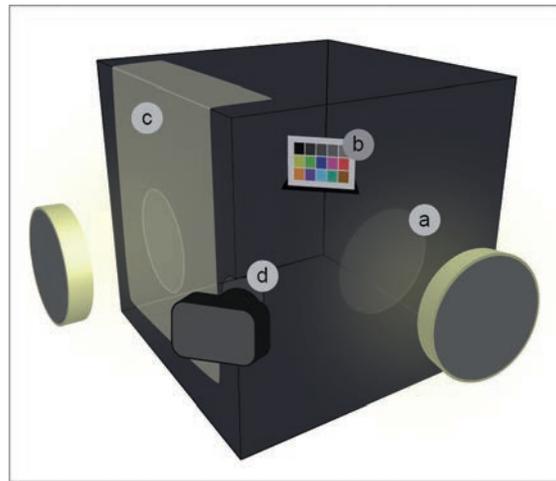


Figure 1: Test cube: a) illumination opening, b) test chart, c) fabric sample, d) camera opening



Figure 2: A photograph of the set

### Colour test chart

To evaluate the quality of colour reproduction with different light shaping attachments, an X-Rite ColorChecker Passport Photo 2 test chart was photographed, which consists of twenty-four colour patches from the ColorChecker Classic colour plate [6, 7]. The RGB values of the colour patches on the test chart were measured with an X-Rite i1 Pro spectrophotometer and Argyll software, using the *spotread* command to perform the measurement. A two-degree observer and a standard D50 illumination were used. The CIELAB values obtained were converted to RGB

values of the sRGB colour space, whose white point is D65. This is the same conversion method that is stated in the X-Rite manufacturer's specification for the values in the test chart used [8].

The measured RGB values of the colour patches deviate from the values given in the specification of the test plate [8], which has no influence on the final results of the study, since the values under different lighting conditions were compared, and not the colour differences between reproduction and original. The measured and reference RGB values are shown in Table 2.

Table 2: Comparison of the measured RGB values of colour fields and the values according to specification [6]

Patch number	Colour patch	According to specification			Measured		
		R	G	B	R	G	B
1	dark skin	115	82	68	119	84	68
2	light skin	194	150	130	201	145	129
3	blue sky	98	122	157	98	138	150
4	foliage	87	108	67	91	107	64
5	blue flower	133	128	177	130	127	172
6	bluish green	103	189	170	90	187	169
7	orange	214	126	44	222	125	44
8	purplish blue	80	91	166	71	90	168
9	moderate red	193	90	99	201	91	94
10	purple	94	60	108	96	60	102
11	yellow green	157	188	64	157	198	63
12	orange yellow	224	163	46	229	161	35
13	blue	56	61	150	43	65	149
14	green	70	148	73	66	150	73
15	red	175	54	60	183	52	56
16	yellow	231	199	31	241	203	7
17	magenta	187	86	149	198	85	148
18	cyan	8	133	161	52	133	166
19	white (.05*)	243	243	242	251	246	238
20	neutral 8 (.23*)	200	200	200	204	202	200
21	neutral 6.5 (.44*)	160	160	160	163	161	161
22	neutral 5 (.70*)	122	122	121	120	120	119
23	neutral 3.5 (1.05*)	85	85	85	83	81	81
24	black (1.50*)	52	52	52	51	50	50

### Light sources

A photographic studio flash Elinchrom ELC500 PRO HD was used. The intensity of the light flash was regulated depending on the textile material tested, as these transmit different amounts of light and require

uniformly illuminated images for each measurement. The light flashes at four intensities used, namely 8, 38, 53 and 75 J, lasted 1/2940, 1/4650, 1/5000 or 1/2740 s. Rotolight Anova PRO ECO FLOOD lights have been used as LED source, with which colour temperature

and light intensity can be adjusted. 100% intensity and three colour temperatures (3230 K, 5000 K and 6260 K) were used for testing, as the latter is due to the switching on and off of individual light emitting diodes that emit light in the cold or warm part of the visible part of the electromagnetic wave spectrum. A Kaiser studio lamp H with maximum intensity and the Osram No. 64575 lamp with a power of 1000 W and an electrical voltage of 230 V were used to light the scene with halogen light.

An X-Rite i1 Pro spectrophotometer in light emission measurement mode and Argyll software were used to measure the light emission spectrum of applied lighting conditions. Each scene was measured three times in steps of 10 nm representing the brightness of the emitted light at wavelengths from 380 nm to 730 nm. The values of the measured emission spectra have been normalized for easier comparison and analysis.

### Light shaping attachments (fabrics)

Images of fabrics were taken with a digital stereo microscope Leica S9i at 40-times magnification. Warp, weft and weave of the materials were determined. Thicknesses of materials was measured with micrometer Metrimpex 6-12-1/B and with measuring unit Mitutoyo ID-C125XB, while colour properties with a Spectraflash 600 Plus CT spectrophotometer, measuring reflected light under measuring conditions of ten-degree observer and a D65 white point. Measured CIE xyY values were used to calculate whiteness of the materials and were converted to CIELAB for further comparison.

### 2.2 Photographic equipment and settings

Nikon D850 DSLR camera and a Nikkor AF-S 50 mm 1:1.4G lens with a HOYA Pro1Digital 58 mm MC UV(0) filter were used to photograph the ColorChecker Passport Photo 2 test chart. The filter serves only as a protection for the lens and does not affect the quality of the colour reproduction.

Photographs were captured in the RAW output format (file type NEF) in order to obtain as much data as possible. The ISO value or sensor sensitivity setting was set at 100 for all the photographs in order to cause

as little noise as possible. The white balance setting was changed according to exposure using camera presets and manual white balance settings as shown in Table 3 [9]. An aperture value of f/4.5 was used, due to the sharpness of the lens is at its best at this aperture and the degree of colour distortion (chromatic aberration) is among the lowest [10]. The shutter speed was adjusted according to other settings so that the photo exposition was ideal. Shutter speed was monitored using a camera light meter and histogram analysis of each photo. The exposure time required was influenced by the type of lighting, as not all lights provide the same intensity of light emitted, and by the tested fabric, as each allows a different amount of light to pass through, thus affecting the brightness of the scene.

### 2.3 Method of analysis

All photos were imported into Adobe Lightroom Classic CC 2019 where we used the Crop tool to crop the edges of the photographed test charts (Figure 3). This reduced the size of the photos per area, resulting in a smaller file size. However, the data important for analysis is retained. The dimensions of the final photos are approximately 1500 px × 1000 px (pixels), with minor size variations, and a resolution of 300 ppi (pixels per inch).

For all cropped photos the whiteness was set to colour field no. 22, in order to colour match the photographs. The white balance adjustment was made in the largest possible range of 15 px × 15 px.

The NEF photo format is no longer suitable for further processing of photos and the information they contain. Therefore, they were exported to a 16-bit TIFF format, which is the least lossy export format available. The exported photos were opened in the ImageJ program. Since the photos are 16-bit TIFF, the program displays them as grayscale values of each colour channel (red, green and blue). Therefore, they were converted to a 32-bit RGB colour image using the *Image - Type - RGB Colour command* [11, 12].

It was also necessary to unify the brightness of the photos, as it was not possible to ensure exactly the same light intensities due to the lighting conditions.

Table 3: Setting the white balance for all used light sources

	Xenon	Halogen	LED 3230 K	LED 5000 K	LED 6260 K
White balance setting	preset Flash (5400 K)	preset Incandescent (3000 K)	Choose colour temp. – 3230 K	Choose colour temp. – 5000 K	Choose colour temp. – 6260 K



Figure 3: Photo of the test chart after cropping with marked colour patches regarding to Table 2

For the control area the colour field no. 22 was chosen, which has RGB values of 120/120/119. With the *RGB Measure plug-in* [13] average RGB values in the area of 100 px × 100 px in the colour field were read. Since the colour adjustment was already provided for when the white balance was set, the ratios of the read RGB values match, but in some photos, it was necessary to lighten or darken by adjusting the brightness. The measurement of the RGB value was repeated and the procedure was repeated until a sufficiently good adjustment was made to reach the reference RGB values of 120/120/119. The corresponding brightness was checked on all 90 photos and adjusted it if necessary. All 24 colour patches in 90 photos were then converted to RGB values, measuring areas of 100 px × 100 px. The same combination of variables was photographed three times, so in the next step we calculated the average RGB values of the individual colour fields in three photographs of the same combination of variables. CIELAB values were needed to calculate the colour differences and thus determine the impact of using fabrics as light shaping attachments.

The conversion was first done between RGB and CIEXYZ then between CIEXYZ and CIELAB [14, 15]. The average R, G and B values of the colour patches were normalized and the inverse sRGB compression function (Equation 1) was used to obtain the linear values of  $r$ ,  $g$  and  $b$ . These were multiplied by the matrix  $M$  (Equation 2) with respect to equation (Equation 3). The matrix  $M$  considers the sRGB colour space and the white point D65 [16]. The result are normalized values of  $X$ ,  $Y$

and  $Z$  which are divided by the corresponding  $X_r$ ,  $Y_r$  and  $Z_r$  values of white point D65 (Equation 4) [17] according to equations (Equation 5), (Equation 6) and (Equation 7) to obtain the values of  $x_r$ ,  $y_r$  and  $z_r$ . From this  $f_x$ ,  $f_y$  and  $f_z$  with respect to the values of Equations 8, 9 and Equations 10, 11 and 12 were calculated. The spectral distribution functions  $f_x$ ,  $f_y$  and  $f_z$  are used to calculate the values of  $L^*$ ,  $a^*$  and  $b^*$  with respect to Equations 13, 14 and 15. To calculate the colour differences, the equation CIE 1976 (Equation 16) was chosen [18]. According to Equation 17 [19], the colour purity  $C$  was calculated for each colour field under different test conditions and then the differences between the illumination without and with fabrics as light shaping attachments were determined.

$$v = \begin{cases} V/2.92 & \text{if } V \leq 0.04045 \\ ((V + 0.055)/1.055)^{2.4} & \text{if } V > 0.04045 \end{cases} \quad (1)$$

$$[M] = \begin{bmatrix} 0.4124564 & 0.3575761 & 0.1804375 \\ 0.2126729 & 0.7151522 & 0.0721750 \\ 0.0193339 & 0.1191920 & 0.9503041 \end{bmatrix} \quad (2)$$

$$\begin{bmatrix} X \\ Y \\ Z \end{bmatrix} = [M] \begin{bmatrix} r \\ g \\ b \end{bmatrix} \quad (3)$$

$$\begin{bmatrix} X_r \\ Y_r \\ Z_r \end{bmatrix} = \begin{bmatrix} 0.95047 \\ 1.00000 \\ 1.08883 \end{bmatrix} \quad (4)$$

$$x_r = \frac{X}{X_r} \quad (5)$$

$$y_r = \frac{Y}{Y_r}$$

$$z_r = \frac{Z}{Z_r}$$

$$\kappa = 903.3$$

$$\epsilon = 0.008856$$

$$f_x = \begin{cases} \sqrt[3]{x_r} & \text{if } x_r > \epsilon \\ \frac{\kappa x_r + 16}{116} & \text{if } x_r \leq \epsilon \end{cases}$$

$$f_y = \begin{cases} \sqrt[3]{y_r} & \text{if } y_r > \epsilon \\ \frac{\kappa y_r + 16}{116} & \text{if } y_r \leq \epsilon \end{cases}$$

$$f_z = \begin{cases} \sqrt[3]{z_r} & \text{if } z_r > \epsilon \\ \frac{\kappa z_r + 16}{116} & \text{if } z_r \leq \epsilon \end{cases}$$

$$L = 116f_y - 16$$

$$a = 500(f_x - f_y)$$

$$b = 200(f_y - f_z)$$

$$\Delta E = \sqrt{(L_1 - L_2)^2 + (a_1 - a_2)^2 + (b_1 - b_2)^2}$$

where  $(L_1, a_1, b_1)$  are first and  $(L_2, a_2, b_2)$  second colour

$$C = \sqrt{a^2 + b^2}$$

(6) The colour differences for each colour field of the test chart were calculated, describing the influence of the use of different fabrics and lighting types on the colour reproduction. The colour difference value is described by the perception of a standard observer,

(7) where:

(8)  $0 < \Delta E_{ab}^* < 1$  – the observer does not notice the difference between the colours,

(9)  $1 < \Delta E_{ab}^* < 2$  – only an experienced observer notices the difference between the two colours,

(10)  $2 < \Delta E_{ab}^* < 3,5$  – even an inexperienced observer notices the difference between the colours,

$3,5 < \Delta E_{ab}^* < 5$  – an obvious difference between the colours is observed,

(11)  $5 < \Delta E_{ab}^*$  – the observer perceives the colour as two different [20].

To make the results easier to interpret, the mean values of the colour differences for each test combination from 24 colour differences of individual colour patches were calculated.

### 3 Results and discussion

#### 3.1 Light sources properties

Figure 4 shows the emission spectrum of the light flashes at the four intensities used, namely 8 J, 38 J, 53 J and 75 J, which lasted 1/2940 s, 1/4650 s, 1/5000 s or 1/2740 s. The spectral distributions are similar. The variability of the curve of the emission spectrum increases with the intensity of the flash, which is due to the greater amount of light and thus the possibility of a more accurate reading of the measuring

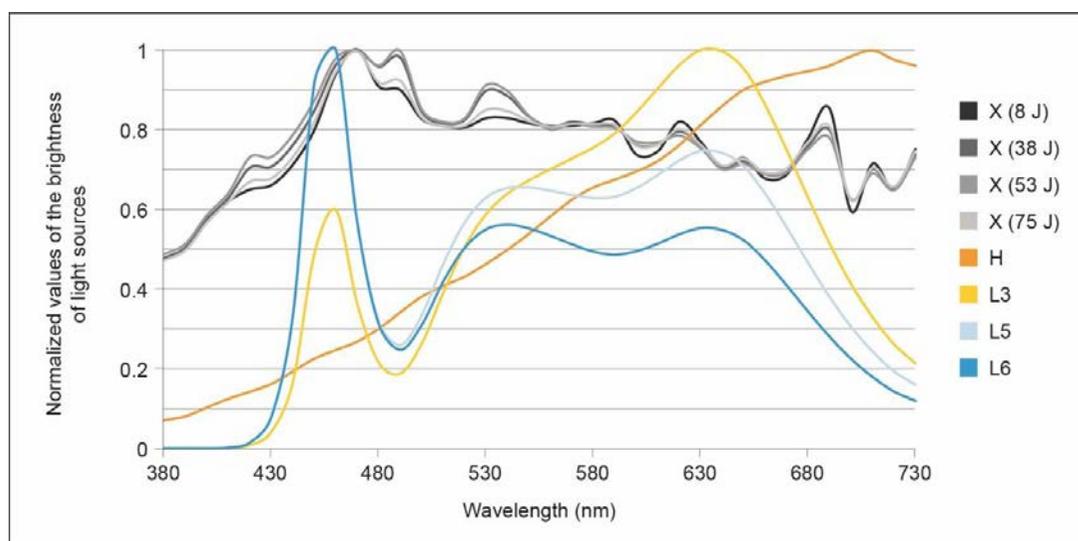


Figure 4: Normalized emission spectra of all used light sources

device. The normalized emission spectra of the emitted lights from halogen source (H) and three LED sources with colour temperatures 3230 K, 5000 K and 6260 K (L3, L5 and L6, respectively) are shown in Figure 4 as well.

Spectral distributions differ depending on the used light source (Figure 4). A light source X with a xenon bulb emits the most uniform spectrum, because its maximum is indistinct in the blue part of the spectrum but shows less emitted light in the purple part. A light source H with a tungsten halogen bulb emits the greatest amount of light in the orange-red part of the spectrum, while LED light sources vary according to colour temperature. L3 shows a smaller fall in the blue part of the spectrum, while the curve rises sharply in the yellow-orange spectrum. This indicates the warm light from the light source. Standard light at 5000 K represents the light source L5, which has a maximum in the blue part of the spectrum and corresponds to the maximum of the light source L6. They differ from each other by the emitted light between the wavelengths 530 nm and 780 nm, with white light having higher values.

### 3.2 Fabrics properties

The values of fabrics' variables are shown in Table 4. In Figure 5 the images of fabrics are represented, showing the differences in fabric density and weave. The calculated colour properties in CIELAB are pictured in Figure 6, while in Figure 7 the differences can be observed visually.

The differences in the measured colorimetric values are mainly due to the yarns used, which are derived from the raw material composition. Twisted yarns are used in natural fiber fabrics, i.e. CO, LI and WO, while synthetic fabrics, i.e. PA and PES, are made from multifilament yarn. The uniformity of the yarn structure has a significant influence on the perception of light passing through it. In the case of wool and polyester fabrics, where the yarns used are the most voluminous (Figure 5), the pores are the smallest and consequently the differences in light colour are the greatest (Figure 6). The passage of photons through the pores has no effect on their transformation, whereas the passage of photons through the yarn significantly transforms them, resulting in a greater change in colour (Figure 6). The degree of whiteness varies significantly, where wool has the lowest level of whiteness and polyester the highest, the latter being a consequence of high amount of optical brightening agents.

### 3.3 Colour differences

Colour differences  $\Delta E^*_{ab}$  of individual colour patches are presented in Figure 8. A red line indicates the value 2, which illustrates the boundary between the colour differences that can be recognized by any observer. Figure 9 shows the average values of all measurements according to the tested light sources and fabrics.

The average colour differences (Figure 8) show that the greatest differences occur with the halogen

Table 4: Specification of tested fabrics. Temperature of change is taken after [3, 4].

Fabric	Label	Weave density (cm <sup>-1</sup> )		Weave	Thickness (mm)	CIE x	CIE y	CIE Y	W	Temperature of change (°C)
		Warp	Weft							
Cotton	CO	40	40	plain	0.228	0.318	0.336	91.57	79.71	150
Linen	LI	20	20	plain	0.435	0.327	0.345	80.80	46.44	150
Wool	WO	30	20	plain	0.359	0.338	0.358	69.84	(4.58)	150
Polyamide	PA	90	40	plain	0.140	0.317	0.334	90.29	82.63	223–265
Polyester	PES	20	20	plain	0.445	0.294	0.306	86.95	145.29	270

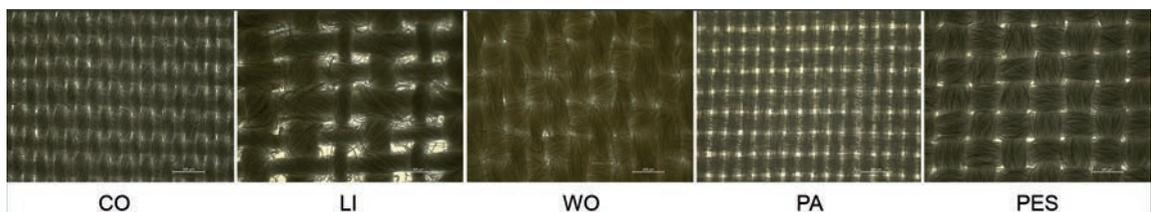


Figure 5: Images of tested fabrics under 40× magnification

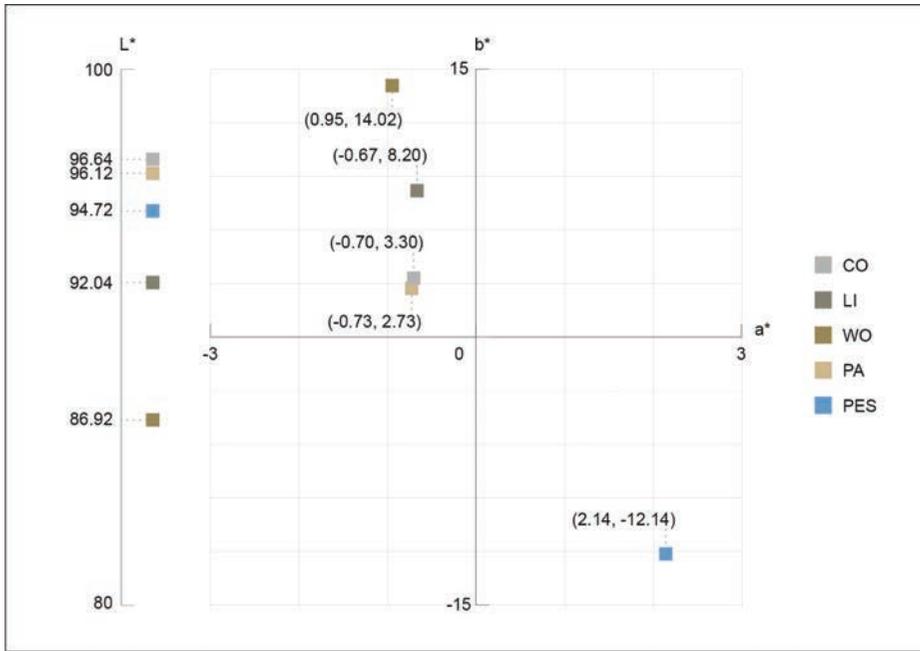


Figure 6: CIELAB values of tested fabrics

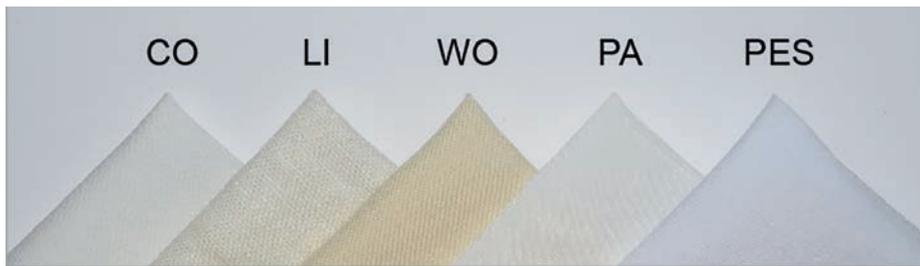


Figure 7: Colour comparison of tested fabrics

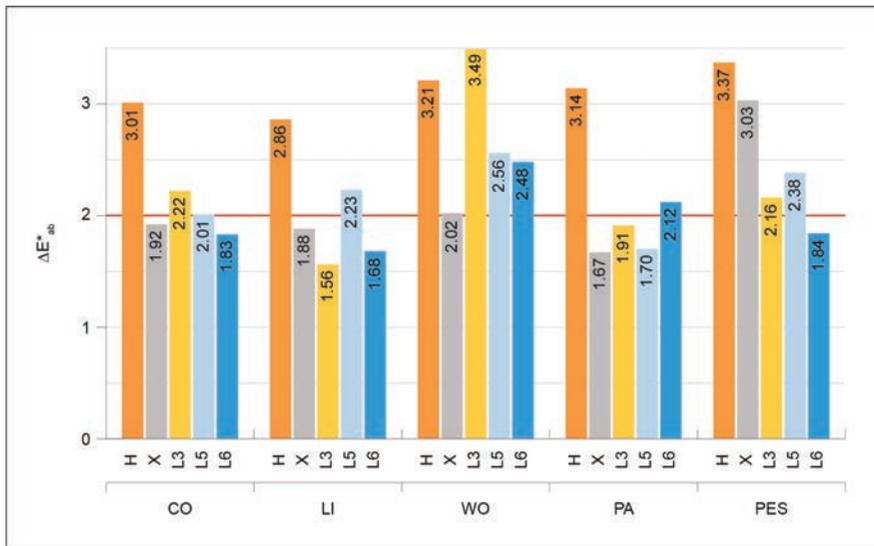


Figure 8: Average values of the colour differences  $\Delta E^*_{ab}$  of twenty-four colour patches for each tested light source and fabric combination

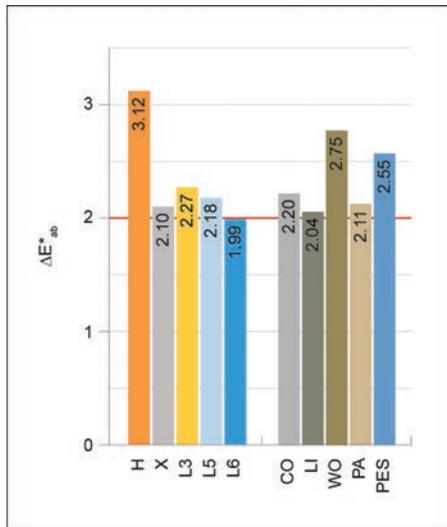


Figure 9: Average values of colour differences for each light source and fabric

light source (marked H) and independently of the fabric used. The values are between 2.86 and 3.37, indicating a recognizable difference between the colours. The other four tested light sources give better average results, as the average colour differences according to Figure 8 are between 1.99 and 2.27. Xenon light (marked with an X) causes the smallest colour differences when using different fabrics, however, we observe a large difference in the illumination through polyester (PES). The LED light sources L3, L5 and L6 give similar results, but individual differences are noticeable, i.e. in the

combinations L5/LI and L3/WO. The smallest colour differences were measured when using linen (LI) and polyamide (PA), followed by cotton (CO). Wool (WO) is the least suitable, due to the large colour difference reflected by all tested light sources. The same applies to PES, which however shows completely different deviation tendencies than WO under different light conditions.

When comparing the colour differences in xenon flash illumination, it was noticed that PES causes a large colour difference, which is also perceived by an inexperienced observer. The use of wool also results in a colour difference greater than 2, but an excess of 0.02 is neglected. From Figure 10 we can see that the PES fabric in combination with xenon light X is different from the other four combinations of this light with other fabrics. It shows a greater colour shift towards green and blue and a lower brightness. The difference in colour hue can be related to the whiteness of the fabric (Figures 6 and 7), as the only fabric tested did not show any yellowing. The PES fabric is also sparsely weaved, which means that a lot of transmitted light is undeformed, so that the colour reproduction is affected by the cold light from light source X.  $\Delta C^*$  also shows the deviation of X/PES from other combinations with light X, as it gives the lowest value, which means that the colours reproduced are less pure. We conclude that the fabrics CO, LI, WO and PA in the tested composition and weave density are suitable for use as light shaping attachments when using xenon light.

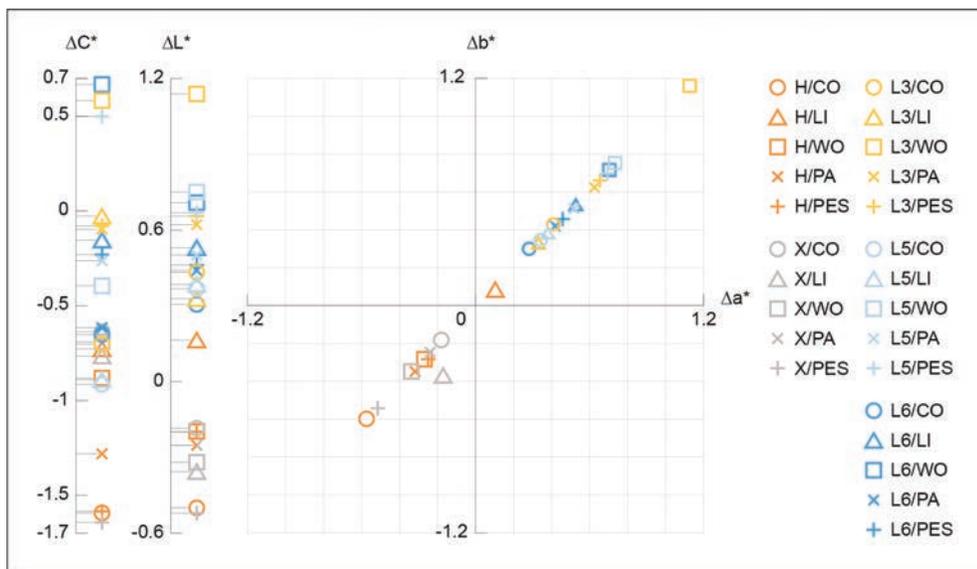


Figure 10: Display of colour deviations  $\Delta C^*$ ,  $\Delta L^*$ ,  $\Delta a^*$  and  $\Delta b^*$ , which occur when using different types of lighting and fabrics

The same trend as X/PES is shown by H/CO, i.e. a combination of halogen light with cotton fabric. The similarity of the mean values of the colour differences from Figure 8 is then also observed in Figure 10, where the same colour deviation is shown along the axes  $L^*$ ,  $a^*$ ,  $b^*$  and  $C^*$ . CO is a slightly yellowed fabric with a higher weave density, which means that it reflects most of the yellow spectrum of electromagnetic waves. The rest is absorbed by the fabric, and some of it travels on, as the textile fibres are not completely opaque and allow light to pass through. The tested cotton fabric has a high weave density, which means that it transmits less unshaped warm light. This may justify the colour shift towards a green-blue hue, despite the use of a warm light source. For comparison, we can take H/LI, whose weaving density is half that of CO, and the yellowing is even higher. As more unformed light is transmitted, the colour hue differences shift towards yellow-red. Even though some shifts in hue are similar for both X and H lighting, the colour differences are large because the  $\Delta E^*_{ab}$  equation takes all three parameters  $L^*$ ,  $a^*$  and  $b^*$  into account. From the results, it can be therefore concluded that the tested fabrics are not suitable for use in combination with halogen light sources, as the influence on the quality of colour reproduction is too great.

The test combinations in which LED light sources were used show similar changes in colour hue and brightness, the only significant deviation occurs in the L3/WO combinations, which represent warm light at 3230 K and the use of woollen fabric. The sparse weave density of the wool fabric causes a higher light transmission and, in combination with its yellowing, the degree of which is the highest of all the fabrics tested, causes the greatest shift in colour hue towards yellow-red. Figure 10 shows that the same trend of colour shift when using WO also occurs with the other two LED light sources (L5 and L6). Despite the colour shift and therefore poor colour reproduction, WO in combination with warm and cold LED light (L3 and L6) produces cleaner colours. A third such example is the L5/PES combination, which is in complete agreement with the findings made in this paragraph for wool and LED light combinations. Other LED lighting combinations and the use of the other four fabrics show similar results without particular variations with respect to the values  $L^*$ ,  $a^*$ ,  $b^*$  and  $C^*$ . It can be concluded that the fabrics CO, LI, PA and PES are suitable for shaping the light from

LED light sources, apart from the combination L5/PES, which represents LED light at 5000 K and polyester fabrics.

## 4 Conclusion

Colour differences are influenced by the interaction of light and fabric. Since the latter differ in colour and weave density, they transmit different amounts of light and absorb different parts of the visible spectrum of electromagnetic waves. Research has shown that the quality of colour reproduction is influenced by light sources and different properties of fabrics used as light shaping attachments, as the final colour impressions depend on their whiteness and weave density. It is assumed that the opacity of textile fibres also plays an important role, which opens new possibilities for further research.

Considering the variables of the light source and the density and whiteness of the tested fabrics, it can be concluded that not all combinations are suitable for use if we want to achieve good colour reproductions. If xenon light is used as the light source on the photographic scene, they are suitable for the use of CO, LI, WO and PA with tested properties, whereas there are large colour differences when PES is used. Not only are most fabrics not suitable for use near halogen light sources due to the high operating temperatures of the lamps, it also turns out that the colour differences are the largest of all tested combinations. This leads to the conclusion that halogen lamp types, regardless of the fabric used, are not suitable for high-quality colour rendering. LED light sources allow the widest range of fabrics, as the colour rendering is satisfactory when using all but WO. An exception is the combination of a light LED source with a colour temperature of 5000 K and the use of PES, so the use of such combination is not recommended.

The research led to the conclusion that the most significant impact on the quality of colour reproduction is a consequence of the whiteness of the fabric used as light shaping attachment on the light source. The type of the fabric has not shown itself as significant, while its weave density plays an important role. The denser the fabric, less opening are in it and less light is transmitted undeformed, therefore the change is lower. While fabrics with higher weave densities transform the light in a higher manor, resulting in more obvious changes, in our case wool and polyester.

## Acknowledgements

The research was cofounded by the Slovenian Research Agency (Infrastructural Centre RIC UL-NTF).

## References

1. GALER, Mark. *Digital photography in available light : essential skills. 3rd edition.* Oxford : Focal Press, 2006.
2. DAVIDSON, Michael W. Tungsten-halogen incandescent lamps [accessible from a distance]. ZEISS Microscopy [accessed 30. 10. 2019]. Available on World Wide Web: <<http://zeiss-campus.magnet.fsu.edu/articles/lightsources/tungstenhalogen.html>>.
3. MCKENNA, Henry A., HEARLE, W.S. John, O'HEAR, Nick. *Handbook of fibre rope technology.* Cambridge : Woodhead Publishing, 2004, p. 53.
4. BURKINSHAW, Stephen M. *Physico-chemical aspects of textile coloration.* Chichester : John Wiley & Sons in association with the Society of Dyers and Colorists, 2016, p. 37–40.
5. GABRIJELČIČ, Helena. Colour and optical phenomena on fabric. *Tekstilec*, 2007, **50**(4–6), 93–132.
6. ColorChecker Passport Photo 2 [accessible from a distance]. X-Rite [accessed 31. 03. 2020]. Available on World Wide Web: <[https://xritephoto.com/ph\\_product\\_overview.aspx?id=2572&catid=158](https://xritephoto.com/ph_product_overview.aspx?id=2572&catid=158)>.
7. AHTIK, Jure. Comparison of ICC and DNG colour profile workflows based on colorimetric accuracy. *Journal of Print and Media Technology Research*, 2017, **6**(3), 115–121.
8. ColorChecker Charts [accessible from a distance]. X-Rite [accessed 31. 03. 2020]. Available on World Wide Web: <[https://xritephoto.com/documents/literature/en/ColorData-1p\\_EN.pdf](https://xritephoto.com/documents/literature/en/ColorData-1p_EN.pdf)>.
9. Digital camera D850 : user's manual. Tokyo : Nikon Corporation, 2017.
10. Lens test data comparison : Nikon AF-S Nikkor 50mm F/1.4G on Nikon D800 [accessible from a distance]. Digital Photography Review [accessed 30. 10. 2019]. Available on World Wide Web: <[https://www.dpreview.com/reviews/lens-compare?lensId=nikon\\_50\\_1p4\\_g&cameraId=nikon\\_d800&version=0&fl=50&av=5.6&view=mtf-ca](https://www.dpreview.com/reviews/lens-compare?lensId=nikon_50_1p4_g&cameraId=nikon_d800&version=0&fl=50&av=5.6&view=mtf-ca)>.
11. ImageJ – Image Menu – Type Submenu [accessible from a distance]. ImageJ [accessed 01. 04. 2020]. Available on World Wide Web: <<https://imagej.nih.gov/ij/docs/menus/image.html>>.
12. AHTIK, Jure, MUCK, Deja, STAREŠINIČ, Marica. Detail diversity analysis of novel visual database for digital image evaluation. *Acta Polytechnica Hungarica*, 2017, **14**(6), 115–132.
13. RUSBAND, Wayne. RGB measure [accessible from a distance]. ImageJ [accessed 21. 11. 2019]. Available on World Wide Web: <<https://imagej.nih.gov/ij/plugins/rgb-measure.html>>.
14. LINDBLOOM, Bruce J. RGB to XYZ [accessible from a distance]. Bruce Justin Lindbloom [accessed 23.11.2019]. Available on World Wide Web: <[http://www.brucelindbloom.com/index.html?Eqn\\_RGB\\_to\\_XYZ.html](http://www.brucelindbloom.com/index.html?Eqn_RGB_to_XYZ.html)>.
15. LINDBLOOM, Bruce J. XYZ to Lab [accessible from a distance]. Bruce Justin Lindbloom [accessed 23. 11. 2019]. Available on World Wide Web: <[http://www.brucelindbloom.com/index.html?Eqn\\_XYZ\\_to\\_Lab.html](http://www.brucelindbloom.com/index.html?Eqn_XYZ_to_Lab.html)>.
16. LINDBLOOM, Bruce J. RGB/XYZ matrices [accessible from a distance]. Bruce Justin Lindbloom [accessed 27. 11. 2019]. Available on World Wide Web: <[http://www.brucelindbloom.com/index.html?Eqn\\_RGB\\_XYZ\\_Matrix.html](http://www.brucelindbloom.com/index.html?Eqn_RGB_XYZ_Matrix.html)>.
17. LINDBLOOM, Bruce J. Chromatic adaptation [accessible from a distance]. Bruce Justin Lindbloom [accessed 27. 11. 2019]. Available on World Wide Web: <[http://brucelindbloom.com/index.html?Eqn\\_ChromAdapt.html](http://brucelindbloom.com/index.html?Eqn_ChromAdapt.html)>.
18. LINDBLOOM, Bruce J. Delta E (CIE 1976) [accessible from a distance]. Bruce Justin Lindbloom [accessed 27. 11. 2019]. Available on World Wide Web: <[http://brucelindbloom.com/index.html?Eqn\\_DeltaE\\_CIE76.html](http://brucelindbloom.com/index.html?Eqn_DeltaE_CIE76.html)>.
19. JELER, Slava, KUMAR, Marko, TIŠLER, Vesna, BOŽIČ, Dušan. Interdisciplinarnost barve. Maribor: Društvo koloristov Slovenije, 2001, p. 211.
20. MOKRZYCKI, Wojciech S., TATOL, Maciej. Colour difference  $\Delta E$  : a survey. *Machine Graphics and Vision*, 2011, **20**(4), 383–411 <https://wisotop.de/assets/2017/DeltaE-%20Survey-2.pdf>.

Selestina Gorgjeva<sup>1,2</sup>, Darinka Fakin<sup>1</sup>, Alenka Ojstršek<sup>1,2</sup>

<sup>1</sup> University of Maribor, Faculty of Mechanical Engineering, Institute of Engineering Materials and Design, Maribor, Smetanova ulica 17, 2000 Maribor, Slovenia

<sup>2</sup> University of Maribor, Faculty of Electrical Engineering and Computer Science, Institute of Automation, Koroška cesta 46, 2000 Maribor, Slovenia

---

# Confocal Fluorescence Microscopy as a Tool for Assessment of Photoluminescent Pigments Print on Polyester Fabric

*Konfokalna fluorescenčna mikroskopija kot orodje za ovrednotenje tiska fotoluminiscenčnih pigmentov na poliestrni tkanini*

Original scientific article/Izvirni znanstveni članek

Received/Prispelo 10-2020 • Accepted/Sprejeto 11-2020

Corresponding author/Korespondenčna avtorica:

Assist Prof dr. Selestina Gorgjeva

E-mail: selestina.gorgjeva@um.si

ORCID: 0000-0002-2180-1603

Tel.: +386 2 220 7935

---

## Abstract

The size and distribution of the photoluminescent pigment particles within the selected binder may affect the quality and appearance of the final print significantly. Yet, the techniques for precise evaluation of size distribution of the pigment particles within a 3D fabric space are rather limited, based on their intrinsic fluorescent properties. The presented work demonstrates a simple screen-printing process for the sustainable application of three different types of commercial fluorescent pigments on polyester (PES) fabric, using polydimethylsiloxane (PDMS) as a binder. A comprehensive toolbox was used to compare and study different commercial photoluminescent pigments and their corresponding prints, by means of size distribution and concentration effect of emission intensity, including Confocal Fluorescence Microscopy (CFM) and Scanning Electron Microscopy (SEM) in combination with complementary spectroscopic techniques, i.e. Energy Dispersive X-ray Spectroscopy (EDX) and Ultraviolet-visible (UV-vis) spectroscopy. The focus is on CFM utilised as a non-destructive tool, used for the evaluation of photoluminescent pigments' spatial distribution within printing pastes, as well as on/within the PES fabrics.

Keywords: Photoluminescent pigments, polyester fabric, confocal fluorescent microscopy, spectroscopy

## Izvleček

Velikost in porazdelitev fotoluminiscenčnih delcev pigmenta v izbranem vezivu lahko bistveno vpliva na kakovost in videz končnega tiska. Vendar pa so tehnike za natančno oceno porazdelitve velikosti delcev pigmenta v 3-D prostoru tkanine na podlagi njihovih notranjih fluorescenčnih lastnosti precej omejene. Predstavljeno delo opisuje preprost postopek sitotiska za trajnostno nanašanje treh različnih vrst komercialnih fluorescenčnih pigmentov na poliestrno (PES) tkanino z uporabo polidimetilsiloksana (PDMS) kot veziva. Uporabili smo kombinacijo orodij, s katerimi smo primerjali in proučevali različne komercialne fotoluminiscenčne pigmente in njihove ustrezne tiske s porazdelitvijo velikosti in učinka koncentracije na intenzivnost emisij, vključno s konfokalno fluorescenčno mikroskopijo (CFM) in vrstično elek-

tronsko mikroskopijo (SEM) v kombinaciji z disperzno rentgensko spektroskopijo (EDX) in UV-vis spektroskopijo. Glavni poudarek je bil na CFM, ki se uporablja kot nedestruktivno orodje za oceno prostorske porazdelitve fotoluminescenčnih pigmentov v tiskarskih vezivih in na PES-tkanini ali v njej.

Ključne besede: fotoluminescenčni pigmenti, poliestrna tkanina, konfokalna fluorescenčna mikroskopija, spektroskopija

## 1 Introduction

Textile materials with minimum utility are far off today's people's needs and expectations; to this end, functional textiles, among which the photoluminescent textiles belong, have attracted significant attention in the textiles consumers' end-user segment [1]. Photoluminescent textiles are obtained either by fixing pigments in the fibre or onto the polymer fibrous surface using chemical binders, by coating or printing diverse patterns onto textile fabrics [2]. The photoluminescent pigments as "glow-in-the-dark" powder particles are used as textile pigments for creating high-resolution patterns [3], as an alternative to LEDs, electro-luminescent wires and optical fibres for designing light-emitting fabrics, decoration, military facilities, communication and transportation, fire emergency systems, etc. One of the interesting, and cost-acceptable applications of luminescent pigments is the protection of the original products' originality [4]. In general, photoluminescent pigments are immobilised onto textile fabrics by means of printing as the most affordable and simple procedure, fostering its use by 80% of printed merchandise [5]. Recently, the aqueous pigment-binder spray-coating technique was used as a facile method for production of coated garments hosting luminescent pigments [6].

A photoluminescent pigment consists mainly of crystals of aggregated elements and photonic traps, where, upon light source exposure, the crystals stay excited and keep discharging light, being supported by photonic traps (rare elements), which extend their phosphorescence time until the entire exhaustion of the stored light photons. Besides their chemical composition, the dimensions of photoluminescent pigments and their distribution within binders, influence the print quality and final appearance of printed material. Techniques used for precise assessment of pigments' size and pattern distribution when printed on fabric (s.c. *in situ* evaluation) are rather limited. Instead, the segmental and micro destructive sampling is mainly utilised, followed by extensive laboratory analysis [7]. In art works and cultural heritage examination of photoluminescent pigments' distribution, several *in situ* identification techniques are

in use, limited mostly to single point analysis such as vibrational spectroscopy, laser-induced breakdown spectroscopy and x-ray spectroscopy for atomic-level analysis, as well as diffuse reflectance imaging, fluorescence and fluorescence lifetime imaging [7], etc. The latter are not yet reported as relevant for evaluation of printed textiles. When only particles are considered (excluding the printed fabric), Dynamic Light Scattering (DLS) methodology is applicable for nano pigments, preferably for monodisperse and, ideally, circular particles, and, as such, it is not completely applicable for the fairly heterogeneous (photoluminescent) pigment used in textile printing. The precise microscopic tools (such as SEM and TEM microscopy) give distribution of patterns easily; however, the obtained information is limited to relatively small surfaces being examined, as well as costs of equipment and the examination itself. When commercial pigments with unknown composition and properties (particles' sizes and their distribution, excitation wavelength, emission time, etc.) are utilised, together with additives (binders, brighteners), the complexity of pigments' evaluation may increase exponentially. In the presented work, a facile procedure for screen-printing of luminescent pigments on polyester (PES) fabrics is presented, using three types of commercial luminescent pigments and polydimethylsiloxane (PDMS) as a binder. Commercial pigments and their respective prints on PES fabrics were examined by complementary microscopic (SEM and CFM) and spectroscopic (EDX and UV-vis) techniques. Special focus in this research work is the utilisation of CFM as a non-destructive tool for simultaneous examination of the morphological and optical properties of photoluminescent pigments, and respective fabric prints in a 3D spatial manner. CFM is already used for micro particles [8–10] and auto fluorescent pigments' assessment [11], and recently our group introduced it for assessment of pigment prints on cotton fabric [12], wherein this work presents the translation of a similar procedure to PES fabric. Nonetheless, examination of the spatial distribution of photoluminescent pigments is of high relevance, since the same was found to affect the propagation and external emission of light highly, through mechanisms

such as charge diffusion, re-absorption, or changes in the mean free path of the emitted photons [13].

## 2 Materials and methods

### 2.1 Materials

PES fabric in plain weave was used, with a mass of 165 g/m<sup>2</sup>, thickness of 0.505 mm, warp density of 19 threads/cm and weft density of 21 threads/cm. Polydimethylsiloxane (PDMS), a two-part liquid elastomer kit, Sylgard 184 (Dow Corning, USA), that consists of a pre-polymer base and crosslinking curing agent in the ratio 10:1, was used as a binder. Three (violet, blue and yellow-green) commercially available photoluminescent pigments Sirius (Samson, Slovenia) were utilised in the printing procedure.

### 2.2 Fabrication of luminescent samples by screen-printing

Before printing, the PES fabric was washed at 40 °C for 30 min, using a solution of 2 g/L of standard neutral non-ionic washing agent, without optical bleacher, water rinsed, and dried at ambient temperature. Individual luminescent pigment in concentrations: 1%, 5%, 10% and 30 wt.% (per weight of PDMS) was admixed in a PDMS binder for 5 min, using a high-speed paddle-stirring apparatus, to acquire homogeneous pigments' distribution. The prepared dispersions were applied onto PES fabric according to the flat screen-printing procedure using a semiautomatic printing table (Johannes Zimmer, Austria). In order to achieve an even coverage over the entire sample, a PES 125 mesh was used, as well as the following optimal parameters: A roll-rod diameter of 15 mm, speed of 1 m/min, max magnet pressure, and 2 application layers. To cross-link the PDMS, printed PES fabrics were dried for 48 hours at ambient temperature.

### 2.3 Scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) spectroscopy

Morphological analysis and elemental composition of three commercial photoluminescent pigments were accomplished by the SEM-EDX system, using a Zeiss Gemini Supra 35 VP Microscope (Carl Zeiss NTS GmbH, Germany), equipped with an X-ray energy dispersion spectrometer (EDX, Oxford Instruments, model Inca 400). Pigments in powder form were positioned onto the holder by double carbon tapes, and sputter coated with Palladium (Pd). 500 X magnification and 1.00 kV setting were applied.

### 2.4 Confocal fluorescent microscopy (CFM)

Square pieces of (non)printed PES fabric were positioned on a transparent glass holder above the 20 x (dry) objective of an inverted CFM Leica TCS SP5 II, equipped with an LAS AF software program. The photoluminescent pigments were excited with an argon laser ( $\lambda_{\text{ex}} = 458 \text{ nm}$ ), while the obtained signal was detected by two hybrid detectors, with a pre-set emission range from 500 nm to 550 nm. High-resolution images (1024 pixels x 1024 pixels) were obtained by image adjusted light gain and 8 x line averaging. Each sample was depicted at several positions, in order to check the printing uniformity in the x-y direction, while in the z-direction (sample thickness), the confocal mode with 10  $\mu\text{m}$  stack thickness was applied, and assessed additionally by the depth (colour) coding function. By moving the focal plane in the z direction, up to 30 optical slices were obtained, and combined further in a 3D image stack for digital processing. The bright field images were captured in parallel, to depict the pigment-lean areas of printed PES fabrics, using a Dodt detector. The images from both the fluorescence and bright field channels were used in split or overlaid mode for further assessment by the ImageJ program, an image processing program, designed for scientific multidimensional images, in particular, the z-project function (for tracking of pigments' distribution within the PES fabric). In parallel, the pigments'-PDMS binder dispersion was also coated onto the thin glass. The particle analysis plug-in (Image J) was employed for the assessment of the size and distribution of pigments within the PDMS binder on the glass. Pure PES fabric and PDMS were analysed as controls.

### 2.5 UV-Vis spectroscopy: luminescence decay assessment

A qualitative assessment of the luminescence decay of the PES samples, printed with a combination of PDMS and individual photoluminescence pigment in different concentrations, was performed by initial, 5 min exposure of samples under UV light with a wavelength of 366 nm, within a UV chamber. Afterward, samples were exposed immediately to deep dark within the chamber, and after 20 seconds and 1 min, the photos were taken. For relative quantification of time-dependent luminescence decay, the same samples were also evaluated on a Tecan Infinite M200 Pro microplate reader in luminescence mode, without using emission filters. Printed PES fabrics were again excited under the same conditions as for the

qualitative assessment, and loaded immediately into the instrument, to commence measurement. Each decay curve was normalised by its initial intensity. Before testing, all printed PES fabrics were exposed to darkness for a 2-day period, to attenuate any afterglow illumination completely.

### 3 Results and discussion

The SEM micrographs on Figure 1 (left) depict luminescent pigment particles with irregular shape and large heterogeneity in terms of size in all three types, which we anticipate to be a consequence of their conventional solid-state bulk synthesis, sintering and powdering procedures [14]. The morphologically distinct regions, as well as large size scattering (i.e. the presence of fine powder particles up to merged aggregates) are present in all pigments. This is expected to affect the luminescent properties, influencing the amount of light absorbed, and, consequently, the emission intensity. In contrast to SEM, the CFM

resolved a larger number of particles due to the lower magnification used. Moreover, CFM has the advantage of generating high contrast images through optical sectioning, resulting from the superposition of sharp features from the focal plane and blurry features coming out of focus [15]. The CFM images (Figure 1, middle) depict particles embedded into the binder, thus, excluding the necessity of examination of pigment powders, the latter having a shortage of presenting real state spatial distribution within the applied printing binder. The CFM data (Figure 1, right) demonstrates wide size distribution within all pigments in a range of 3–80  $\mu\text{m}$ , which is reported for commonly used pigments containing Eu-doped strontium aluminates [16]. In the yellow-green pigment, the smallest fraction ( $d \sim 3 \mu\text{m}$ ) is missing, the same being dominant in the other two (blue and violet) pigments, which can be one of the factors contributing to the highest glowing intensity over the other two types at the same concentration. The size distribution of the violet pigment differs significantly when compared to the other two pigments in terms

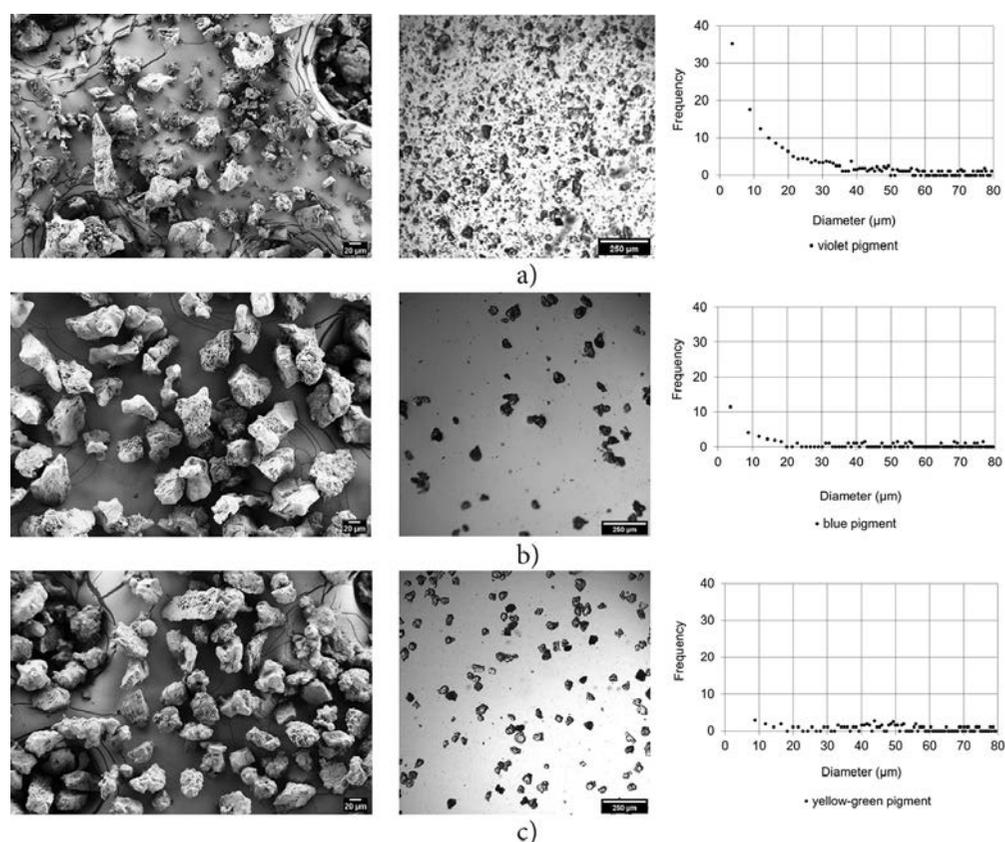


Figure 1: SEM micrographs (left), CFM micrographs (middle) and size distribution extracted (right) from CFM images of a) violet, b) blue and c) yellow-green luminescent pigments. The scale bar in the CFM images corresponds to 250  $\mu\text{m}$ .

Table 1: EDX data of violet, blue and yellow-green photoluminescent pigments

Violet pigment			Blue pigment			Yellow-green pigment		
Element	Weight percentage	Atomic percentage	Element	Weight percentage	Atomic percentage	Element	Weight percentage	Atomic percentage
<b>O K</b>	34.82	65.54	<b>O K</b>	31.68	73.21	<b>O K</b>	34.82	65.54
<b>Al K</b>	17.72	19.78	<b>Mg K</b>	3.69	5.62	<b>Al K</b>	17.72	19.78
<b>Sr L</b>	34.28	11.78	<b>Cl K</b>	1.84	1.92	<b>Sr L</b>	34.28	11.78
<b>Pd L</b>	5.10	1.44	<b>Sr L</b>	29.76	12.56	<b>Pd L</b>	5.10	1.44
<b>Eu M</b>	8.08	1.45	<b>Ta M</b>	17.67	3.61	<b>Eu M</b>	8.08	1.45
			<b>W M</b>	15.36	3.09			

of count, which is approximately three times higher for particles in the 3–30  $\mu\text{m}$  range, yet the presence of larger, irregular particles in this pigment is expected to decrease the luminescence intensity [17].

The EDX data, gathered in Table 1, identify the presence of the rare earth element europium (Eu), in the yellow-green and violet pigments. Eu is known as a photoluminescent pigments' dopant [18]. Besides Eu, the yellow-green pigment contains strontium (Sr), aluminium (Al) and oxygen (O), which suggest the presence of strontium aluminates, also having phosphorescent properties, long afterglow at room temperature, and broad spectrum distribution of luminescence from the blue to green region. In the case of the violet pigment, the calcium aluminate elements were identified, presumably as mixed compounds rather than a particular phase. As relatively diverse compositional profiles were identified for each pigment, we can speculate on the combined effect of composition, and size/distribution profile of pigments, the latter seeking further investigation involving fractionation and comparison of effects of by size-resolved components.

As promising for identification of pigments' size and distribution within the PDMS binder, the CFM was used further for the investigation of the 3D spatial distribution of pigments when printed on PES fabric. Indeed, CFM permits depiction and localisation of the fluorescing compounds unambiguously at light microscopy resolution [8], offering a non-destructive method for in-depth imaging, without demanding preparation [19], sometimes even without the need of additional fluorescent labelling [20, 21] which is also the case for PES fabric. This is due to the point by point illumination of samples, and rejection of out of focus light, which enables acquisition of z- stack (thickness) images and construction of 3D micrographs [22, 23]. The same imaging parameters were

applied for both pigments and PES fabrics. Under a specified excitation wavelength ( $\lambda_{\text{ex}} = 458 \text{ nm}$ ), the particles were visualised clearly and distinguished from the PES fabric (Figure 2), which itself exhibited a fluorescent signal tracked by a parallel detector. Image collects the maximum intensity signal from the consecutive images. Interestingly, the increase in pigment concentration from 1 up to 10 wt.% does not supplement the fluorescence signal significantly; the same can also be observed in the fluorescence emission photographs (Figure 4). Relatively homogeneous distribution of pigments was observed from the maximum intensity image, like within single pigment analysis, which points-out the suitability of the selected pigment-binder-printing procedure, allowing homogeneous distribution. At the same concentration (5%), the yellow-green pigment provides the largest filler pores' blocking effect, due to the domination of large particle fractions over the other pigments.

Printed pigments were observed within an inspected thickness ( $< 300 \mu\text{m}$ ), and not through the whole fabric thickness, as the thickness value is in the function of an objective aperture. To visualise the distribution of pigments in the z axis better (thickness), a colour-coded projection is presented in Figure 3, demonstrating the inclusion of printed particles in deeper regions of the PES fabric. Bonding of pigments on individual PES yarns is pronounced in the case of the blue pigment (Figure 3a), which is not the case with the yellow-green pigment (Figure 3b), where the presence of fibrous, fibre-like glowing impurities and significantly different pigment particles by means of structure and size are observed.

For the macroscopic investigation of printed PES fabrics, the same were illuminated with UV at 254 nm and 366 nm, and further depicted (Figure 4) and evaluated by means of afterglow decay kinetic (Figure 5).

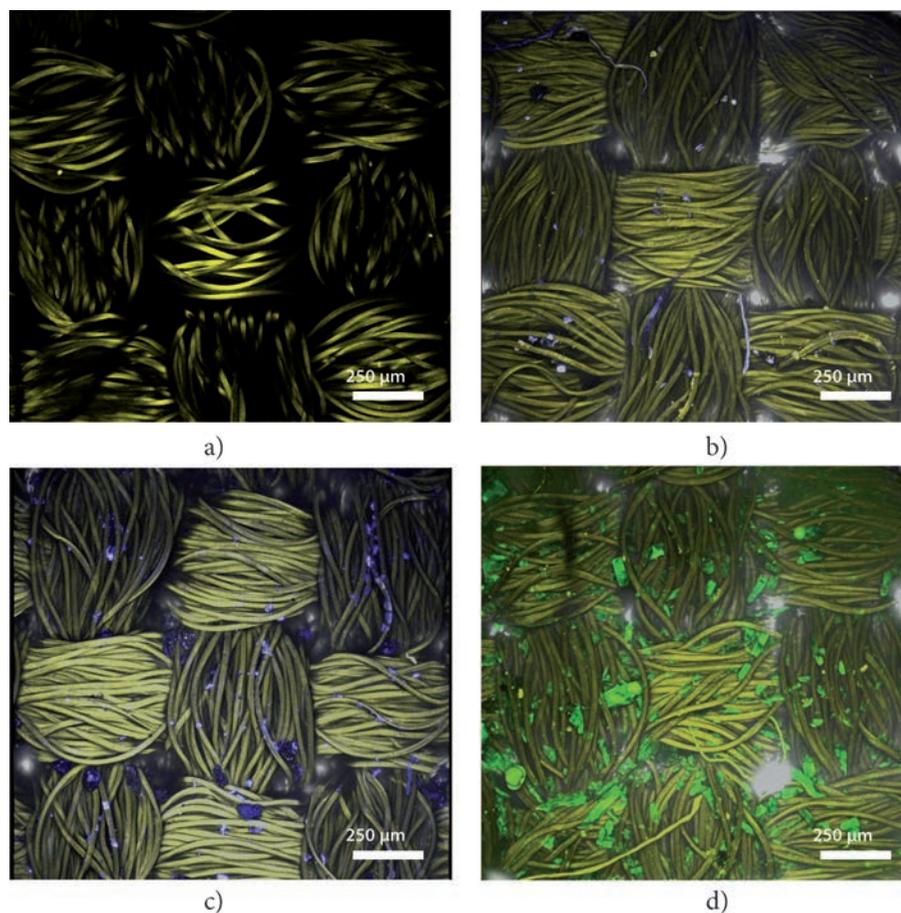


Figure 2: CFM micrographs of PES fabric a) without (reference) and with 5% of b) violet, c) blue and d) yellow-green pigment; obtained by fabric excitation by argon laser at  $\lambda_{ex} = 458 \text{ nm}$  and signal accumulation by two hybrid detectors, with a pre-set emission range from 500 nm to 550 nm range. The white scale bar corresponds to 250  $\mu\text{m}$  in all CFM micrographs.

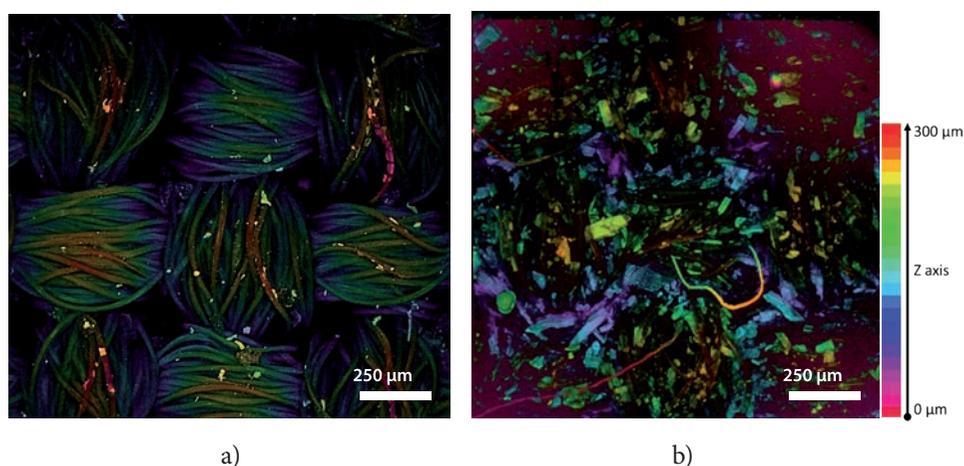


Figure 3: CFM micrographs of PES fabric with 5% of a) blue and b) yellow-green pigment; obtained by fabric excitation by argon laser at  $\lambda_{ex} = 458 \text{ nm}$  and signal accumulation by two hybrid detectors, with a pre-set emission range from 500 nm to 550 nm. The white scale bar corresponds to 250  $\mu\text{m}$  in all CFM micrographs. The colour scale bar refers to the image colours presenting the relative z depth, where glowing particles are positioned.

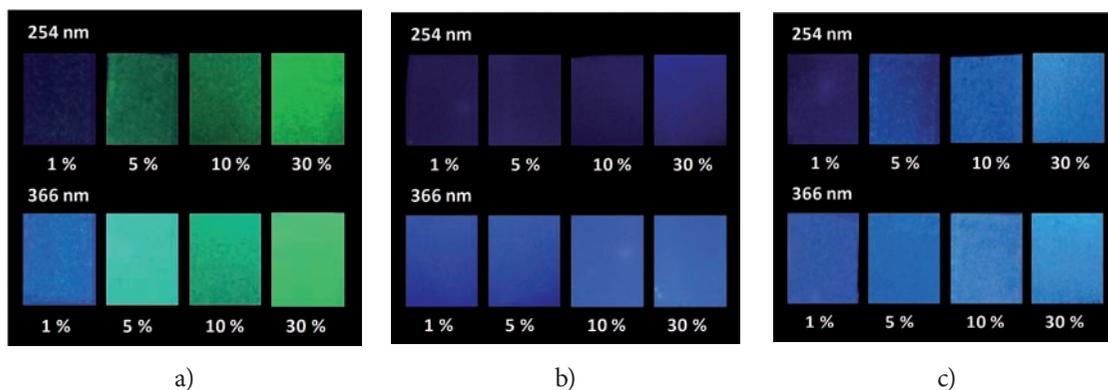


Figure 4: Photographs of screen-printed PES with 1%, 5%, 10% and 30 % of a) yellow-green; b) violet and c) blue pigments in PDMS; excited within a UV chamber at 254 nm and 366 nm for 5 min

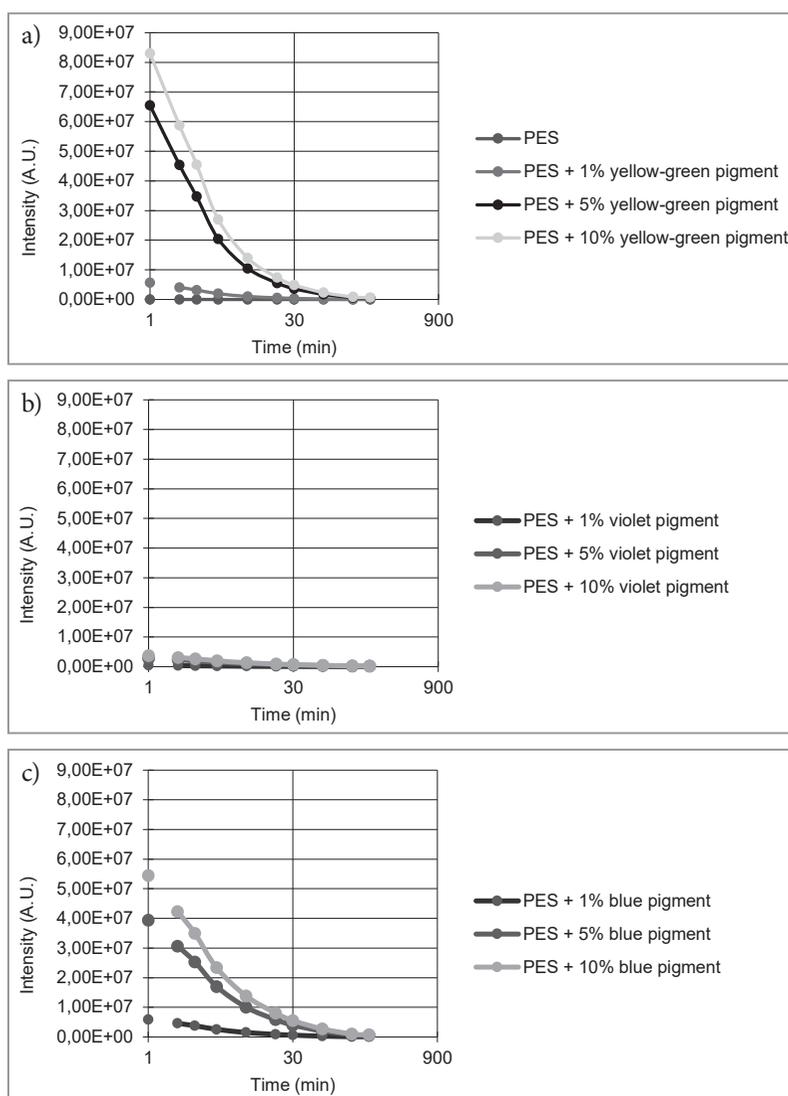


Figure 5: Luminescence decay curves of screen-printed PES with 1%, 5% and 10 % of a) yellow-green; b) violet and c) blue pigments in a 180 min period

From the photographs in Figure 4, an expected increase in luminescence intensity could be observed, with the increase in pigment's concentration (irrespective of pigment type), while, among different pigments, the intensity decreased in the order yellow-green > blue > violet at the same concentrations. Luminescence decay curves, presented in Figure 5 were obtained from the relatively wide time span measurement, i.e. from 1 up to 180 min. The presence of rare earth aluminates activated by Eu brought the highest emission intensity in the yellow-green pigment, as well as the longest afterglow, compared to the other two pigments.

## 4 Conclusion

Three commercial photoluminescent pigments were evaluated by complementary microscopic and spectroscopic techniques, before and after being screen-printed onto PES fabric by use of a PDMS binder. Herein, we identify CFM as an advanced, non-destructive visualisation tool for pigments' assessment by means of their size distribution, as well as 3D (spatial) distribution, within a binder and PES textile fabric. The proposed assessment procedure can be applied further in analysis related to the conservation of artefacts. In this preliminary work we speculate on the potential size and distribution effect of particles onto their glowing intensity, and it's complementing with the effect of their composition. Yet, further investigation and analysis are needed (e.g., including pre-fractionation of pigments) before providing a solid statement for our findings.

## Acknowledgements

*The MATUROLIFE project leading to this research has received funding from the European Union's Horizon 2020 Research and Innovation Program under Grant Agreement No 760789. The authors acknowledge the Textile Chemistry Program (P2-0118(B)) under financial support from the Slovenian Research Agency (ARRS), as well as Dr. Silvo Hribernik for the SEM-EDX measurements.*

## References

1. YAN, Y., ZHU, Y., GUO, X., GE, M. The effects of inorganic pigments on the luminescent properties of colored luminous fiber. *Textile Research Journal*, 2014, **84**(8), 785–792, doi: 10.1177/0040517513507361.
2. IYER, S.N., BEHARY, N., NIERSTRASZ, V., GUAN, J., CHEN, G. Study of photoluminescence property on cellulosic fabric using multifunctional biomaterials riboflavin and its derivative Flavin mononucleotide. *Scientific Reports*, 2019, **9**, 1–16, doi: 10.1038/s41598-019-45021-5.
3. TAVČER, Petra Forte, AHTIK, Jure, GODEC, Modec. Lastnosti fosforescenčnih pigmentov, tiskanih na tkanino. *Tekstilec*, 2016, **59**(3), 226–236, doi: 10.14502/Tekstilec2016.59.226-236.
4. TOMČÍKOVÁ, I.Z., TOMČÍKOVÁ, Z., UJHELYIOVÁ, A., MICHLÍK, P., KRIVOŠ, Š., HRICOVÁ, M. Structure and properties of polypropylene fibres modified with photoluminescent pigment as a tool for the protection of original products. *Tekstilec*, 2017, **60**(4), 283–289, doi: 10.14502/Tekstilec2017.60.283-289.
5. KHATTAB, T.A., REHAN, M., HAMOUDA, T. Smart textile framework : photochromic and fluorescent cellulosic fabric printed by strontium aluminate pigment. *Carbohydrate Polymers*, 2018, **195**, 143–152, doi: 10.1016/J.CARBPOL.2018.04.084.
6. KHATTAB, T.A., REHAN, M., HAMDY, Y., SHAHEEN, T.I. Facile development of photoluminescent textile fabric via spray coating of Eu(II)-doped strontium aluminate. *Industrial and Engineering Chemistry Research*, 2018, **57**(34), 11483–11492, doi: 10.1021/acs.iecr.8b01594.
7. COMELLI, D., D'ANDREA, C., VALENTINI, G., CUBEDDU, R., COLOMBO, C., TONIOLO, L. Fluorescence lifetime imaging and spectroscopy as tools for nondestructive analysis of works of art. *Applied Optics*, 2004, **43**(10), 2175–2183, doi: 10.1364/AO.43.002175.
8. LAMPRECHT, A., SCHÄFER, U., LEHR, C.M. Structural analysis of microparticles by confocal laser scanning microscopy. *AAPS PharmSciTech*, 2000, **1**(3), 10–19, doi: 10.1208/pt010317.
9. SKINNER, J.P., SWIFT, K.M., RUAN, Q., PERFETTO, S., GRATTON, E., TETIN, S.Y. Simplified confocal microscope for counting particles at low concentrations. *Review of*

- Scientific Instruments*, 2013, **84**(7), 074301, doi: 10.1063/1.4812782.
10. GORGIEVA, S., JANČIČ, U., HRIBERNIK, S., FAKIN, D., STANA KLEINSCHEK, K., MEDVED, S., FAKIN, T., BOŽIČ, M. Processing and functional assessment of anisotropic cellulose nanofibril/Alolt/sodium silicate: based aerogels as flame retardant thermal insulators. *Cellulose*, 2020, **27**(3), 1661–1683, doi: 10.1007/s10570-019-02901-3.
  11. BORDOWITZ, J.R., MONTGOMERY, B.L. Exploiting the autofluorescent properties of photosynthetic pigments for analysis of pigmentation and morphology in live *Fremyella diplosiphon* cells. *Sensors*, 2010, **10**(7), 6969–6979, doi: 10.3390/s100706969.
  12. GORGIEVA, S., VIRANT, N., OJSTRŠEK, A. Complementary assessment of commercial photoluminescent pigments printed on cotton fabric. *Polymers*, 2019, **11**(7), 1–18, doi: 10.3390/polym11071216.
  13. PEREZ, M.J., GONZALEZ, I., SAIZ, J.M., MORENO, F., GONZALEZ, F. Saturation processes of photoluminescent pigments embedded in sinterized glass. *Journal of Modern Optics*, 2009, **56**(13), 1466–1474, doi: 10.1080/09500340902990015.
  14. YESILAY Kaya, S., KARACAOGLU, E., KARASU, B. Particle size influence of starting batches on phosphorescence behaviour of  $\text{Sr}_4\text{Al}_{14}\text{O}_{25}$  based bluish green phosphors. *Advances in Applied Ceramics*, 2012, **111**(7), 393–397, doi: 10.1179/1743676112Y.0000000005.
  15. JONKMAN, J., BROWN, C.M., WRIGHT, G.D., ANDERSON, K.I., NORTH, A.J. Tutorial : guidance for quantitative confocal microscopy. *Nature Protocols*, 2020, **15**, 1585–1611, doi: 10.1038/s41596-020-0307-7.
  16. PENG, T., YANG, H., PU, X., HU, B., JIANG, Z., YAN, C. Combustion synthesis and photoluminescence of  $\text{SrAl}_2\text{O}_4:\text{Eu,Dy}$  phosphor nanoparticles. *Materials Letters*, 2004, **58**(3-4), 352–356, doi: 10.1016/S0167-577X(03)00499-3.
  17. ANESH, M.P., GULREZ, S.K.H., ANIS, A., SHAIKH, H., ALI MOHSIN, M.E., AL-ZAHRANI, S.M. Developments in  $\text{Eu}^{+2}$ -doped strontium aluminate and polymer/strontium aluminate composite. *Advances in Polymer Technology*, 2014, **33**(S1), 221436–21445, doi: 10.1002/adv.21436.
  18. RAO, J., WANG, Y., WANG, W., KE, H., LI, Y., ZHAO, Y., DIAO, Z., JIA, D., ZHOU, Y. Mechanism of superior luminescent and high-efficiency photocatalytic properties of  $\text{Eu}$ -doped calcium aluminate by low-cost self-propagating combustion synthesis technique. *Scientific Reports*, 2017, **7**, 1–9, doi: 10.1038/s41598-017-03099-9.
  19. GORGIEVA, S., HRIBERNIK, S. Microstructured and degradable bacterial cellulose–gelatin composite membranes : mineralization aspects and biomedical relevance. *Nanomaterials*, 2019, **9**(2), 1–20, doi: 10.3390/nano9020303.
  20. GORGIEVA, S., VUHERER, T., KOKOL, V. Autofluorescence-aided assessment of integration and  $\mu$ -structuring in chitosan/gelatin bilayer membranes with rapidly mineralized interface in relevance to guided tissue regeneration. *Materials Science and Engineering : C*, 2018, **93**, 226–241, doi: 10.1016/J.MSEC.2018.07.077.
  21. MALEŠ, L., FAKIN, D., BRAČIČ, M., GORGIEVA, S. Efficiency of differently processed membranes based on cellulose as cationic dye adsorbents. *Nanomaterials*, 2020, **10**(4), 1–18, doi: 10.3390/nano10040642.
  22. PRASAD, V., SEMWOGERERE, D., WEEKS, E.R. Confocal microscopy of colloids. *Journal of Physics : Condensed Matter*, 2007, **19**(11), 25, doi: 10.1088/0953-8984/19/11/113102.
  23. KIRKBRIDE, K.P., TRIDICO, S.R. The application of laser scanning confocal microscopy to the examination of hairs and textile fibers: An initial investigation. *Forensic Science International*, 2010, **195**(1-3), 28–35, doi: 10.1016/J.FORSCIINT.2009.10.030.

Bosiljka Šaravanja<sup>1</sup>, Tanja Pušić<sup>2</sup>, Krešimir Malarić<sup>3</sup>, Anica Hursa Šajatović<sup>1</sup>

<sup>1</sup> University of Zagreb Faculty of Textile Technology, Department of Clothing Technology,  
Prilaz baruna Filipovića 28a, HR-10000 Zagreb, Croatia,

<sup>2</sup> University of Zagreb Faculty of Textile Technology, Department for Textile Chemistry & Ecology,  
Prilaz baruna Filipovića 28a, HR-10000 Zagreb, Croatia

<sup>3</sup> University of Zagreb Faculty of Electrical Engineering and Computing, Department of Wireless Communications,  
Unska 3, HR-10000 Zagreb, Croatia

---

## Durability of Shield Effectiveness of Copper-Coated Interlining Fabrics

*Obstojnost elektromagnetne zaščite tkanin,  
prevlečenih z bakreno oblogo*

Original scientific article/Izvirni znanstveni članek

Received/Prispelo 9-2020 • Accepted/Sprejeto 10-2020

Corresponding author/Korespondenčna avtorica:

**Prof. Tanja Pušić, Ph. D.**

E-mail: tanja.pusic@ttf.unizg.hr

Phone: +385 1 48 77 353

---

### Abstract

The protective shield effectiveness (SE) of copper-coated polyamide and polyester interlining fabrics at frequencies of 1.8 GHz, 2.1 GHz and 2.4 GHz are presented in this study. These fabrics were treated through 10 cycles of dry and wet cleaning. After the characterization of shield effectiveness, fabrics were stored in a dark and dry place. On the basis of the SE values at all measured frequencies, it was confirmed that the copper-coated protective polyester fabric had better SE durability under storage conditions than the copper-coated polyamide fabric. Accordingly, the copper-coated polyester interlining fabric is more suitable for integration into functional clothing due to its use cycle.

Keywords: interlining copper-coated fabrics, electromagnetic shield, storage, dry cleaning, wet cleaning.

### Izvleček

V raziskavi je predstavljena učinkovitost z bakrom prevlečenih poliamidnih in poliestrskih tkanin pred elektromagnetnim sevanjem (EMS) pri frekvencah 1,8 GHz, 2,1 GHz in 2,4 GHz. Tkanine so bile 10-krat kemično čiščene in prane. Po karakterizaciji učinkovitosti zaščite pred sevanjem EMS so bile tkanine shranjene v temnem in suhem prostoru. Na podlagi izmerjenih vrednosti zaščite pred EMS pri vseh proučevanih frekvencah je bilo potrjeno, da je z bakrom prevlečena poliestrna tkanina pri skladiščenju obstojnejša kot z bakrom prevlečena poliamidna tkanina. Iz teh ugotovitev izhaja, da je zaradi načina uporabe z bakrom prevlečena poliestrna tkanina primernejša za vgradnjo v funkcionalna oblačila kot medvlaga.

Ključne besede: medvlaga, prevlečena z bakrenim premazom, elektromagnetna zaščita, skladiščenje, kemično čiščenje, pranje

---

## 1 Introduction

Mobile technology has made significant progress in recent decades and it is difficult to imagine everyday activities and tasks without mobile phones, tablets and laptops. Wi-Fi has become a “must have”, resulting in an increase in exposure to radio frequency electromagnetic (EM) fields. Base stations offer good signal coverage that cannot avoid the impact of electromagnetic radiation, even in the open [1, 2]. It is assumed that the pathogenic electromagnetic radiation of various modern technologies surrounding mankind is 100 to 200 million times stronger than 100 years ago [4].

The intensive technological development of new electric and electronic devices and gadgets resulted in demand for increased control of technology’s negative impact and a way to prevent that impact. This is why limits on EM emissions for all electric and electronic devices were set to minimise the possibility of radio and wireless communications interfering with human and other organisms. Among other numerous solutions, textile materials and products have attracted considerable attention, primarily due to their diversified and comprehensive application [5]. The intensive development of textile products with EM shield effect properties has attracted considerable interest of researchers, and the textile and garment

industries, which have been trying to manufacture woven and knitted fabrics, as well as garments with EM shield effect properties [6–8].

The research in this paper focused on monitoring the durability of shield effectiveness (SE) of the S1 and S2 copper-coated interlining fabrics, the purpose of which is protection (shielding) against electromagnetic microwave radiation. The impact of 10 cycles of professional care processes, dry and wet cleaning, and storage time over a period of seven years on SE properties was evaluated at frequencies 1.8 GHz, 2.1 GHz and 2.4 GHz.

## 2 Materials and methods

### 2.1 Materials

The durability of protective effects was tested on copper-coated polyamide (S1) and polyester (S2) interlining fabrics woven in plain weave (P1/1). They are intended to be sewn into the pockets of garments (jackets, coats, trousers and other garments) in which mobile devices are most commonly carried. The S2 copper-coated polyester interlining has a mass per unit area that is approximately 28 g/m<sup>2</sup> higher than that of the S1 polyamide interlining. The basic properties of the S1 and S2 copper-coated protective interlining fabrics are shown in Table 1.

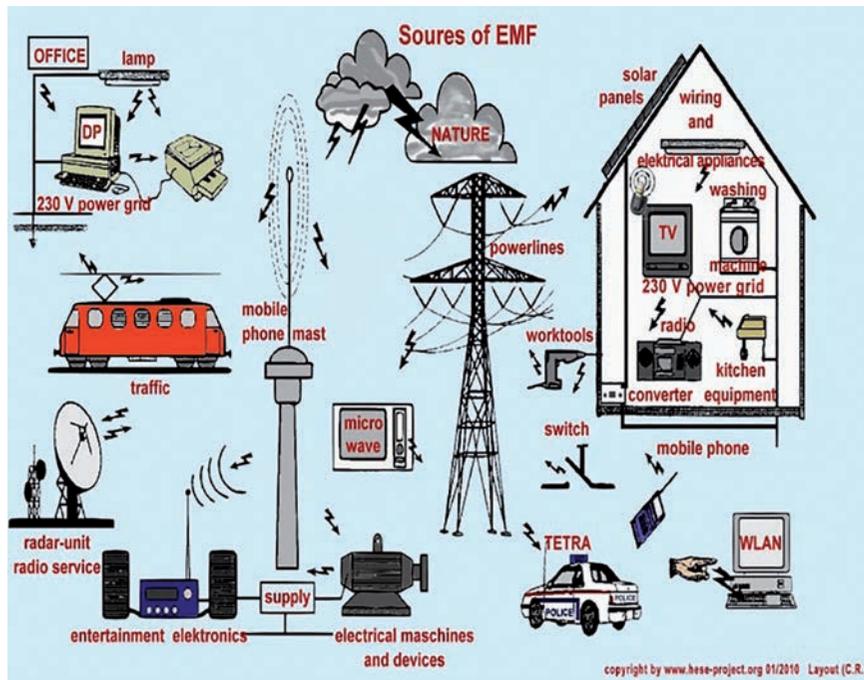


Figure 1: Exposure to radio frequency electromagnetic radiation outdoors and indoors [3]

Table 1: Basic characteristics of the S1 and S2 protective copper-coated interlining fabrics

Characteristics	S1	S2
Sample image		
Raw material composition	100% polyamide filament	100% polyester fibre
Mass per unit area (g/m <sup>2</sup> )	57.0	84.8
Warp yarn density (ends/cm)	50	58
Weft yarn density (picks/cm)	25	40
Warp yarn count (tex)	6.35	7.36
Weft yarn count (tex)	6.1	9.71
Fabric thickness – untreated (mm)	0.11	0.08
Fabric thickness – 10 dry-cleaning processes (mm)	0.07	0.07
Fabric thickness – 10 wet-cleaning processes (mm)	0.07	0.08
Fabric weave structure	Plain weave (1/1)	Plain weave (1/1)

## 2.2 Methods

### 2.2.1 Dry cleaning

The dry-cleaning process (DC) was performed in a two-bath procedure. A special detergent formulated with new, biodegradable cationic surfactants

intended for antistatic effects and a good cleaning effect ( $w = 2\%$ ) was added in the first PERC bath. The process specified in Table 2 was performed in a dry-cleaning machine with a load of 16 kg. Protective interlining fabrics measuring 1 m x 1 m were dry-cleaned in 10 cycles and interphase ironed at 110 °C.

Table 2: Dry-cleaning process

Parameters	1 <sup>st</sup> bath	2 <sup>nd</sup> bath	Drying
	Pre-cleaning	Cleaning	
Temperature (°C)	20	20	60
Time (min)	4	6	30 (5/5)
Number of revolutions (min <sup>-1</sup> )	300	360	-
PERC <sup>a)</sup> (l)	20	40	-
Detergent	2%	-	-
Bath ratio (BR)	1:2	1:4	-

<sup>a)</sup> perchloroethylene

Dry cleaning as a professional care process was carried out according to the HRN EN ISO 3175-2 standard.

### 2.2.2 Wet cleaning

The wet-cleaning procedure (WC) was carried out in a Renzacci wet-cleaning machine according to the cleaning program for highly sensitive fabrics in four stages:

- pre-treatment, 20 °C
- processing – wet cleaning, special, formulated detergent, 20 °C, 10 min
- after-treatment, QAC – quarternary ammonium compound in combination with ethoxylated fatty alcohol, 20 °C, 3 min
- drying in tumbler (60 °C, 2 min) combined with ambient relaxation.

Wet cleaning of the S1 and S2 interlining fabrics measuring 1 m x 1 m was carried out according to the HRN EN ISO 3175-3 standard.

### 2.3 Measurement of EM shielding effectiveness (SE)

The impact of professional care cycles and storage in the dark under ambient conditions on shielding properties of the tested fabrics was tested using the method developed at the University of Zagreb Faculty of Electrical Engineering and Computing in the Microwave Laboratory of the Department of Wireless Communications. The measurement of the effectiveness of the EM shielding was carried out using the measurement equipment (NARDA SRM 3000 measuring instrument, HP 8350 B signal generator, IEV horn antenna – A12 type and the frame for the shielding fabric (1 m × 1 m) made according to the recommendations of the international IEE-STD 299-97, MIL STD 285 and ASTM D-4935-89 standards [9–11].

The EM protection factor was determined as the ratio between the EM field intensity ( $E_0$ ) measured without the test material and the EM field intensity ( $E_1$ ) with the material placed between the radiation source and the measuring device. Shield effectiveness (SE), expressed in dB, is calculated according to equation 1 [13, 14]:

$$SE = 20 \log \frac{E_0}{E_1} \quad (1)$$

Measurements of the SE properties of functional protective fabrics before and after 10 cycles of dry and wet cleaning were taken at frequencies of 1.8 GHz, 2.1 GHz and 2.4 GHz in March 2013. The same fabric

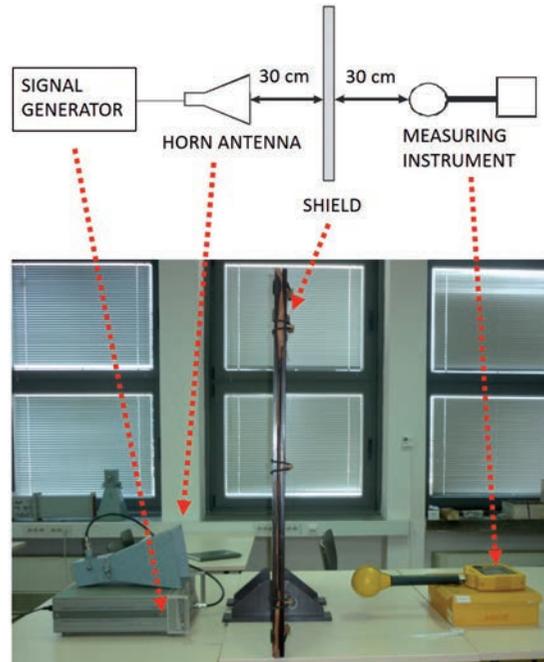


Figure 2: Measurement set up [12]

samples with known SE properties were stored in a dark room for a period of seven years. Repeated measurements at the same frequencies were carried out in March 2020.

The change in shield effectiveness of the S1 and S2 interlining fabrics after 10 cycles of professional treatments and dwell time were expressed using equations 2–4:

$$dSE = SE_0 - SE_{DC} \quad (2)$$

$$dSE = SE_0 - SE_{WC} \quad (3)$$

$$dSE = SE_0 - SE_s \quad (4)$$

where  $SE_0$  represents the initial shield effectiveness of interlining fabrics,  $SE_{DC}$  represents the shield effectiveness of interlining fabrics after 10 dry-cleaning cycles,  $SE_{WC}$  represents the shield effectiveness of interlining fabrics after 10 wet-cleaning cycles, and  $SE_s$  the shield effectiveness of stored interlining fabrics.

## 3 Results and discussion

The results of the effectiveness of the SE protection of the face and back side of the tested protective interlining fabrics before and after 10 cycles of professional care, and after storage in a dark room are presented in Tables 3 and 4, and Figure 3.

Table 3: SE of the face side of the S1 and S2 interlining fabrics before and after 10 cycles of professional care (DC and WC) in the periods 2013 and 2020

Frequency (GHz)	Period	SE (dB)					
		Prior DC and WC		After 10 <sup>th</sup> DC		After 10 <sup>th</sup> WC	
		S1 – face	S2 – face	S1 – face	S2 – face	S1 – face	S2 – face
1.8	2013	21.07	14.32	1.85	5.81	0.07	4.42
	2020	14.46	14.00	0.70	5.50	0	4.09
2.1	2013	25.68	13.58	3.11	5.26	0.06	4.49
	2020	18.02	12.84	0.24	5.06	0	4.18
2.4	2013	21.36	17.78	2.85	5.74	0.02	4.67
	2020	16.28	17.05	0.41	5.28	0	4.21

Table 3 shows the results of the effectiveness of SE protection on the face of the S1 and S2 interlining fabrics, and Table 4 shows the results of the effectiveness of the SE protection on the back side of the S1 and S2 interlining fabrics.

The presented results show that the SE values of the S1 and S2 protective fabrics decrease under the influence of 10 cycles of dry and wet cleaning in relation to the initial values at all tested frequencies. The results confirm that the dry and wet-cleaning processes have affected the decline in SE properties. The SE resistance of the S2 fabric to dry and wet cleaning is better than of the S1 fabric, which has almost completely lost its protective properties after 10 cycles of dry and wet cleaning.

A similar trend can be seen in the decrease in the protective properties of these fabrics during the dwell period of over seven years. The reduction index under the influence of storage is significantly higher for the S1 fabric than the S2 fabric. The influence of the

measurement frequency of the SE properties is also observed, whereby it decreased by 6.61 dB at 1.8 GHz, by 7.66 dB at 2.1 GHz and by 5.08 dB at a frequency of 2.4 GHz for the S1 fabric. Based on previous studies of the initial SE properties using SEM images [11, 12], the recognised differences in fabrics can be attributed to compactness and better state of preservation of copper coating on the S2 fabric compared to the S1 fabric.

The results of the SE properties of the back side of the S1 and S2 protective fabrics show similar changes like on the face of these fabrics after 10 cycles of professional care and dwell time. The decrease in the SE value for the S1 protective fabric depends on the frequencies, whereby at 1.8 GHz the decrease of SE by 6.81 dB was measured, at 2.1 GHz by 6.71 dB and at a frequency of 2.4 GHz by 4.67 dB. A much smaller drop in SE was recorded for the S2 protective interlining fabric, i.e. at all tested frequencies it was less than 1 dB.

Table 4: SE of the back side of the S1 and S2 interlining fabrics before and after 10 cycles of professional care (DC and WC) in periods 2013 and 2020

Frequency (GHz)	Period	SE (dB)					
		Prior DC and WC		After 10 <sup>th</sup> DC		After 10 <sup>th</sup> WC	
		S1 – back	S2 – back	S1 – back	S2 – back	S1 – back	S2 – back
1.8	2013	21.82	14.96	2.46	5.95	0.10	4.89
	2020	15.01	14.44	0.90	5.72	0	4.63
2.1	2013	26.13	14.37	4.49	5.56	0.06	4.91
	2020	19.42	13.76	0.76	5.34	0	4.48
2.4	2013	22.08	18.43	3.82	6.12	0.09	5.13
	2020	17.41	18.12	1.02	6.04	0	4.72

The results in Tables 3 and 4 show that the face and back of the S2 protective interlining fabric have a very good resistance to storage. On the basis of these performances and its better resistance to professional care conditions, it can be concluded that the S2 fabric is more acceptable as a built-in interlining fabric than the S1 fabric. These characteristics are significant taking into account the expected life cycle of garments with integrated interlinings of 5 to 7 years [15]. Figure 3 shows the change in shield effectiveness (dSE) of the S1 and S2 protective interlining fabrics after 10 cycles of dry and wet cleaning.

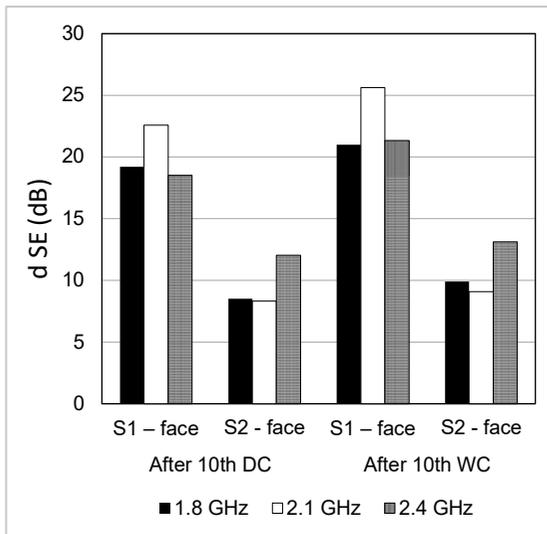


Figure 3: Change in shield effectiveness (dSE) of the S1 and S2 protective interlining fabrics after 10 cycles of dry and wet cleaning

The change in SE of the S1 and S2 interlining fabric after 10 cycles of dry and wet cleaning is different, i.e. Figure 3. The impact of the dry-cleaning process on dSE of fabric S1 depends on the frequency and is manifested through the somewhat better preservation of shield effectiveness on higher frequencies. The dry-cleaning process is a better option for preservation of the SE properties of S1 than the wet-cleaning process. The change in SE of protective interlining fabric S2 after 10 cycles of dry and wet cleaning is smaller when compared to S1. The impact of both processes on dSE of S2 is similar. The difference in SE properties of fabric S2 after the process of dry and wet cleaning at frequencies 1.8 GHz and 2.1 GHz is less than at frequency 2.4 GHz.

Figure 4 shows the change in shield effectiveness (dSE) of the S1 and S2 protective interlining fabrics after dwell time.

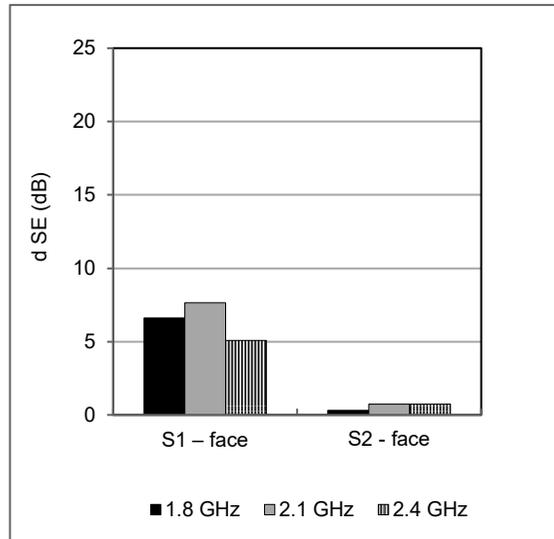


Figure 4: Change in shield effectiveness (dSE) of the S1 and S2 protective interlining fabrics after dwell time

The impact of dwell time on the dSE of the S1 fabric presented in Figure 4 depends on the frequency at which the degree of protection was measured. The change in SE is prominent at the middle frequency of 2.1 GHz and less prominent at the frequency of 2.4 GHz. The initial SE of the S2 fabric is lower compared to the S1 fabric, but the impact of ageing on the change in protective properties of the S2 fabric is insignificant compared with the S1 fabric. Despite the fact that the initial SE properties are weaker, this fabric possesses better stability of protective properties on ageing and better acceptability for incorporation into cloth.

## 4 Conclusion

Measurements of SE properties of the face and back of the polyamide (S1) and polyester (S2) protective lining fabrics showed differences between these fabrics before and after professional care cycles and storage, despite the fact that both are copper-coated. Professional care cycles of the S1 fabric resulted in a decline in SE properties at all frequencies, while the durability of protection properties of the S2 fabric was significantly better. The analysis of the obtained results of stability to storage of untreated fabrics showed a decrease in the SE value of the face and back of the S1 protective lining fabric, depending on the frequencies. In a comparative analysis of the SE results of the face and back of the S2 protective

fabric, the SE drop was up to 1 dB at all measured frequencies. The same trend was achieved after 10 cycles of professional care.

#### Funding – Acknowledgment

*This work was supported in part by the Croatian Science Foundation under project no. HRZZ-IP-2018-01-7028.*

#### References

1. *Biological effects of electromagnetic fields : mechanisms, modeling, biological effects, therapeutic effects, international standards, exposure criteria.* Edited by Peter Stavroulakis. Berlin : Springer, 2003.
2. MARATHE, NV. DESAI, UB., MERCHANT, SN. Base station selection strategy in multihop cellular networks : a new approach. In *2008 International Conference on Signal Processing, Communications and Networking*. Chennai : The Institute of Electrical and Electronics Engineers, 2008, pp. 401–404, doi: 10.1109/ICSCN.2008.4447227.
3. Best EMF meters to measure home & wireless radiation [accessible from a distance]. TheCityEdition [accessed 24. 4. 2020]. Available on World Wide Web: <[https://www.emfradiation.net/best\\_EMF\\_meters\\_reviews\\_buyers\\_guide.html](https://www.emfradiation.net/best_EMF_meters_reviews_buyers_guide.html)>.
4. CEKEN, Fatma, KAYACAN, Ozlem, OZKUR, Ahmet, UGURL, Sebnem Seckin. The electromagnetic shielding properties of copper and stainless steel knitted fabrics. *Tekstil*, 2011, **60**(7), 321–328.
5. OZEN, Mustafa, USTA, Ismail, BEYIT, Ahmet, UZUN, Mehmet, SANCAK, Erhan, ISGOREN, Ersane. An investigation of electromagnetic wave absorption potential of woven fabrics with stainless steel wire. In *RMUTP International Conference Textiles & Fashion*. Bangkok : Rajamangala University of Technology Phra Nakhon, 2012, pp. 4–17.
6. DURAN, Deniz, KADOGLU, Huseyin. A research on electromagnetic shielding with copper core yarns. *Tekstil ve Konfeksiyon*, 2012, **22**(4), 354–359.
7. ROH, Jung-Sim, CHI, Yong-Seung, KANG, Tae Jin, NAM, Sang-Wook. Electromagnetic shielding effectiveness of multifunctional metal composite fabrics. *Textile Research Journal*, 2008, **78**(9), 825–835, doi: 10.1177/0040517507089748.
8. PERUMALRAJ, R., DASARADAN, BS., ANBARASU, R., AROKIARAJ, P., LEO HRISH, S. Electromagnetic shielding effectiveness of copper core-woven fabrics. *The Journal of the Textile Institute*, 2009, **100**(6), 512–524, doi: 10.1080/00405000801997587.
9. ASTM D4935-18 *Standard test method for measuring the electromagnetic shielding effectiveness of planar materials*. West Conshohocken : ASTM International, 2018, doi: 10.1520/D4935-18.
10. IEEE STD 299-2006 *Standard method for measuring the effectiveness of electromagnetic shielding enclosures*. IEEE, 2006, doi: 10.1109/IEEESTD.2007.323387.
11. MIL-STD-285 *Military standard: Attenuation measurements for enclosures, electromagnetic shielding, for electronic test purposes*. IEEE, 1956.
12. ŠARAVANJA, Bosiljka, MALARIĆ, Krešimir, PUŠIĆ, Tanja, UJEVIĆ, Darko. Impact of dry cleaning on the electromagnetic shield characteristics of interlining fabric. *Fibres & Textiles in Eastern Europe*, 2015, **23**(1/109), 104–108.
13. ŠARAVANJA, Bosiljka, MALARIĆ, Krešimir, PUŠIĆ, Tanja, UJEVIĆ, Darko. Shield effect of functional interlining fabric. *AUTEX Research Journal*, 2015, **15**(2), 93–98, doi: 10.1515/aut-2015-0014.
14. GOOCH, Jan W., DAHER, John. K. Fundamentals of electromagnetic shielding. In *Electromagnetic shielding and corrosion protection for aerospace vehicles*. New York : Springer, 2007, doi: 10.1007/978-0-387-46096-3\_3.
15. LAITALA, Kirsi, KLEPP, Ingun Grimstad. Age and active life of clothing. In *PLATE conference*. Nottingham : Trent University, 2015, pp. 182–186.

Thomas Grethe  
FTB, Hochschule Niederrhein - University of Applied Sciences,  
Webschulstrasse 31, 41065 Moenchengladbach, Germany

---

## Biodegradable Synthetic Polymers in Textiles – What Lies Beyond PLA and Medical Applications? A Review.

*Biorazgradljivi sintetični polimeri v tekstilstvu – kaj sledi PLA in medicinskim načinom uporabe? Pregled.*

Original scientific article/Izvirni znanstveni članek

Received/Prispelo 9-2020 • Accepted/Sprejeto 10-2020

Corresponding author/Korespondenčni avtor:

Dr. Dipl.-Chem. Thomas Grethe

E-mail: thomas.grethe@hsnr.de

---

### Abstract

Biodegradable polymers are currently discussed for applications in different fields and are becoming of increasing interest in textile research. While a plethora of work has been done for PLA in medical textiles, other biodegradable polymers and their textile application fields are studied less often, presumably due to higher costs and fewer market opportunities. However, some are emerging from research to pilot scale, and are already utilized commercially in packaging and other sectors but not, unfortunately, in textiles. The commercialisation of such polymers is fuelled by improved biotechnological production processes, showing that textile applications are increasingly conceivable for the future. Additionally, commonly accepted definitions for biodegradability are probably misleading, if they are used to estimate the environmental burden of waste management or recycling of such materials. In this review, the current state of research in the field of biodegradable polymers for the application in textile materials is presented to identify emerging developments for new textile applications. It was clearly seen that PLA is most dominant in that field, while others facilitate new options in the future. The production costs of raw materials and the current patent situation are also evaluated. A special focus is placed on fibre raw materials, coatings, and additives for clothing and technical textiles. Fibre-reinforced composites are excluded, since polymers applied for the matrix component require very different properties compared to the textile materials. This represents a topic to be discussed separately. As a result, these new biodegradable polymers might serve as interesting coating materials for textiles that seem to sneak on to the textile market, as the patent search for such coating formulations suggests. Moreover, new biodegradable fibrous materials for clothing applications can be suggested, but some material properties must be addressed to render them processable on common textile machines.

Keywords: biodegradable polymers, recycling, biodegradability, PLA, textile coatings

### Izvleček

Danes so biorazgradljivi polimeri predmet razprav za rabo na različnih področjih in postajajo čedalje bolj priljubljeni v raziskavah na področju tekstilstva. Medtem ko je raziskava rabe PLA za medicinske tekstilije veliko, pa se drugi biološko razgradljivi polimeri in njihova raba v tekstilstvu verjetno zaradi večjih stroškov in manjših tržnih možnosti proučujejo manj pogosto. Nekateri od njih so že na stopnji pilotskih raziskav in jih že tržijo za embalažo in v drugih sektorjih, na področju tekstilij pa žal še ne. Trženje takšnih polimerov spodbujajo izboljšani biotehnoški proizvodni procesi, kar kaže, da bo v prihodnosti njihova raba za tekstilije bolj razširjena. Poleg

tega splošno sprejete opredelitve biorazgradljivosti verjetno vodijo do zavajajočih sklepov, če se upoštevajo le ocene obremenitve okolja z ravnanjem z odpadki ali recikliranjem takšnih materialov. V tem pregledu je podano trenutno stanje raziskav na področju biološko razgradljivih polimerov za rabo na področju tekstilij z namenom, da bi identificirali nastajajoči razvoj novih tekstilnih aplikacij. Ugotovljeno je bilo, da na področju tekstilne rabe prevladuje PLA, medtem ko drugi polimeri odpirajo nove možnosti v prihodnosti. Ocenjeni so tudi proizvodni stroški surovin in trenutno stanje patentov. Poseben poudarek je na tekstilnih surovinah, premazih in aditivih za tekstilije za oblačila in tehnične namene. Kompoziti, ojačeni z vlakni, niso zajeti, ker so zahtevane lastnosti za polimerne matrice drugačne kot za tekstilije in bi to področje moralo biti obravnavano posebej. Novi biološko razgradljivi polimeri bi se lahko uporabljali kot funkcionalni premazi na tekstilijah, saj jih, kot je mogoče razbrati iz poizvedb za tovrstne patentirane formulacije premazov, že tržijo tudi za področje tekstilij. Predlagati je mogoče tudi nova biološko razgradljiva vlakna za oblačila, vendar je treba nekatere njihove lastnosti prilagoditi, da jih bo mogoče predelovati na standardnih tekstilnih strojih.

*Ključne besede:* biorazgradljivi polimeri, recikliranje, biorazgradljivost, PLA, tekstilni premazi

## 1 Introduction

Current legislation and increasing public awareness about polymer waste call for new options in the recycling of such materials. While everyday products such as bags, nettings or packaging materials are already manufactured from biodegradable plastics to some extent, the development of textile products appears to be more challenging. In this overview, only synthetic polymers are discussed, since natural polymers like cellulose or proteins are already well-known and utilized in the textile sector. Therefore, this report will focus on manmade polymers for textile purposes. In this narrowed field, primarily three applications can be considered: fibre-reinforced composites, fibrous substrates and coatings/finishings. The first field has been already widely addressed and thus will not be discussed here. However, interesting options emerge in the latter two sectors of fibrous substrates and textile coating materials. The majority of publications for biodegradable fibrous substrates address the medical sector and, to a lesser extent, technical textiles. Thus, a systematic literature review was conducted to identify previously evaluated materials and developments, as well as new opportunities in these fields. Biodegradable textile coatings are even less prominent in the literature, so a patent search was also conducted.

Moreover, commonly accepted definitions for the term “Biodegradability” do not address environmental requirements sufficiently, since degradation products are either defined too strictly or are left completely vague. An extended definition may serve to characterize possible future products more meaningfully instead.

## 2 Definitions

The term *biodegradability* needs to be defined first, since every material will degrade in the environment over a sufficient amount of time and/or under appropriate environmental conditions. To narrow down the temporal and environmental conditions, consideration of the field of application looks promising. For the medical sector, the timeframe should be in the range of the targeted duration of therapy, which may be in the order of several weeks or less, while environmental conditions are physiological (nearly neutral pH, 37 °C, water-based surrounding, etc.). For technical textiles, the definition will obviously be different, since degradation is meant to be fulfilled by microorganisms. In this context, the OECD (Organisation for Economic Co-operation and Development) gives four classifications of biodegradability [1]:

- primary biodegradation

It is understood as the loss of certain properties of a material; consequently no complete decomposition has to occur.

- ultimate biodegradation

Here the material has to be decomposed completely into carbon dioxide, water, minerals, and potential biomass (of the microorganism involved in the degradation).

- readily biodegradable

Materials screened by strict tests on ultimate biodegradability that assume a rapid and complete biodegradation in aquatic environments.

- inherently biodegradable

Materials in this class exhibit unambiguous biodegradation behaviour in any tests.

These definitions and the associated test methods all address degradation in natural environments

and assess material degradation by direct or indirect measures of organic carbon, usually after 28 days. However, the transformation products of the degradation process and possible non-degradable residues are not taken into account by these definitions and tests, but must be assessed for a complete picture. If those residues exist and are bio-accumulative, the continuous emission of small amounts may lead to future issues, as seen in fluorinated compounds today. For this reason, such tests only deliver useful results if the material is completely degraded into carbon dioxide, biomass and water.

In a practical example, the biodegradability of mineral oils was investigated and found to be up to 70% in some cases by using a test method where the time-frame was 20 weeks (CEC-L-33 A-93). Testing was done by extracting the oil soluble phase and subsequent infrared spectroscopy using the 2950  $\text{cm}^{-1}$  band of CH-vibrations. It remained unclear what kind of residues are present in the extract and also which metabolites remained in the aqueous phase [2]. Although some sources consider this method as outdated, [3] other actual sources such as OECD 301 and the above-mentioned OECD definitions also do not evaluate residues.

For the context of this report, the OECD definitions above, except *primary biodegradability*, and an extended degradation time of up to one year, will serve valid criteria for review.

While this definition addresses only natural environmental conditions, industrial composting is also considered a form of biodegradation in the context of this report. Several standards do exist to characterize the industrial compostability of materials, usually demanding a 90 % decomposition in 180 days, [4] where the EN 13432 is probably the most common standard. An additional classification to *biodegradable* must be introduced for this review, which will be named *bionneutral degradation* (see Figure 1 for a summary of its definition). While *biodegradability* requires the complete breakdown into water, carbon dioxide and biomass, *bionneutral degradability* will include every substance that will break down into naturally occurring substances in the respective eco system. For practical application, it can be assumed that these reaction products will blend into the natural substance cycles, without significant environmental impacts. This is obviously a question of quantity as for the definition of biodegradability as well. It can be assumed that such residues will be degraded into carbon dioxide and water after some time.

#### Bionneutral degradability:

##### **Material is degradable**

- ◆ in the neutral environment, under physiological
- ◆ conditions or in an industrial process
- ◆ completely into naturally occurring substances
- ◆ which blend *neutrally* into naturally existing material cycles.

Figure 1: Concept of bionneutral degradability

Therefore, literature for this overview qualifies if it is about a manmade polymer and meets at least one of the three following criteria:

- biodegradable under physiological conditions;
- biodegradable or bionneutral degradable in natural environments with respect to [1], or materials assumed to fit in that category; and
- biodegradable in industrial composting similar to EN 13432, or materials assumed to fit in that category.

Materials that are not tested explicitly but can be assumed to fit in these categories are not excluded to ensure the widest overview on current substances.

### 3 Materials

Most synthetic biodegradable polymers can be categorized either as plant-based, microorganism-based or animal-based. The latter consists mainly of chitin and its derivatives and gelatine, which is rarely used to produce fibrous materials. However, chitosan is often utilized in antibacterial textile finishing. Polymer materials produced by microorganisms can be alginate-based materials, but also polymeric esters, proteins and other biological macro molecules, since such organisms can be easily genetically modified to produce a wide variety of materials. Plant-based polymers are mostly based on carbohydrates and can be obtained from starchy or cellulosic biomass. Polymers of greater interest in the field of biodegradable polymers are polylactic acid (PLA), and polyhydroxy alkanates (polyhydroxy fatty acids). PLA is a very common thermoplastic for many applications including melt spinning into fibres, mainly for medical applications. It is also used for the 3D-printing of structures on textile materials [5].

In general terms, most biodegradable polymers are esters, since hydrolytic cleavage of esters by enzymes is a common reaction in nature (see Figure 2). It is further assumed that aliphatic esters are of better degradability than aromatic ones, since the former may fit better in enzymatic active sites [6].



was derived from benzene in the first place. Most of the PCL available on the market is therefore of fossil origin, although biodegradable. Recent developments show processes for synthesizing capronic acid from corn stover and also discuss market opportunities for this approach [12], potentially rendering the polymer bio-based and biodegradable.

Poly(lactic acid) (PLA) and poly(butylene succinate) (PBS) are also polymers built from natural monomers, so that their decomposition products will be lactic acid, succinic acid and a diol component. Because these building blocks are available on large industrial scales, a significant cost advantage can be achieved compared to PHA.

PLA can be manufactured in three different ways: the direct condensation of lactic acid, azeotropic condensation, and ring opening polymerization after prior lactide formation (cyclic lactic acid dimers), where the last two methods are the most common ones [13]. The raw material for all processes is lactic acid, which can be produced either by chemical synthesis or the fermentation of carbohydrates. The last process is widely investigated and optimized, so that most of the lactic acid for technical applications is produced in that manner [14]. Current processes utilize starchy biomass for fermentation, but recent developments applying metabolic engineering methods open opportunities to metabolize lignocellulosic substrates, which leads to a further reduction in costs [15]. For chemical synthesis, a multi-step approach involving the addition of HCN to acetaldehyde forming lactonitrile is followed. While the chemical synthesis delivers racemic products, stereo-chemically pure substances can be obtained through fermentation [16]. Thus, homo- and copolymers of L- and D- forms can be obtained exhibiting different thermal and mechanical properties.

To obtain PBS, succinic acid is used as a commodity that is produced mainly through fermentation [17]. Substrates can vary along different types of carbohydrates, depending on the chosen microorganism. As a rule, succinic acid is only one single product among others in the metabolic cycle of a bacterium. Accordingly, metabolic engineering is used here too, to delete pathways for the production of unwanted by-products [18]. Succinic acid is then directly polymerized with butane diol to form PBS.

The above-mentioned materials can be classified, at a minimum, as bionutral degradable, since decomposition will lead to monomeric building blocks that all have natural origins.

Other polymers, copolymerized from the building blocks of non-biodegradable and biodegradable polymers, sometimes show a significant biodegradability. However, this property is highly dependent on the ratio of the different monomer types involved. If decomposition products occur that are made of the initial building blocks or even of more complex nature, the sweeping claim of a bionutral degradability cannot be made as in the aforementioned cases. In fact, it must be determined whether an ultimate degradation eventually takes place or an assessment of the residues on the environment is necessary.

In particular, numerous studies have been conducted with the aim of determining whether microorganisms and/or enzymes are capable of decomposing such copolymers [19–21], sometimes evaluating decomposition by the weight loss of the polymer [22] or solubility [23]. Although remarkable efforts considering the amount of polymer types covered were made, these works give no insight into biodegradability or bionutral degradability as long as xenobiotic monomer building blocks were involved.

Thus, copolymers involving, for example, terephthalic acid or 1,4-butanediol may be decomposable by microorganisms, but the residues must be assessed. Since both substances are xenobiotics, the bionutral degradability of such polymers cannot be generally assumed.

Although not biodegradable by any means, it is interesting to look at the relatively new substance poly(ethylene furanoate) (PEF), which is discussed as a potential alternative to poly(ethylene terephthalate) (PET). It is partially manufactured from biomass and, although its production appears to be quite ambitious, it finds itself at the brink of becoming commercially relevant, which demonstrates that such processes can possibly be implemented successfully. It is obtained through the polycondensation of poly(ethylene 2,5-furandicarboxylate) (FDCA) and ethylene glycol. Publications discussing technical production from lignocellulose [24] and investigating material properties in depth [25] date back only five years. The chemical synthesis of furan derivatives as a precursor is reported by using starch [26] and cellulose [27]. An interesting approach for synthesizing 2,5-furandicarboxylate using supported noble metal catalysts was reported recently [28]. Industrial pilot plants started up in 2011 to produce 40 t/a of 2,5-furandicarboxylate. In the years that followed, different major chemical companies developed new processes, and partially joined forces to increase the

production of FDCA [29]. In 2016, AVA Biochem announced its objective to achieve a production capacity of 30,000 tonnes/year, increasing to 120,000 tonnes/year [30]. Textile applications have not yet been reported in patent or scientific literature. The material thus seems to be new and interesting as a replacement for PET-based polyesters. This may serve as a promising model for some of the above-mentioned processes, which are not yet commercialized.

### 3.1 Thermal properties

For technical applications, the thermal properties of the materials are of crucial interest. In Table 1, glass transition temperatures and melting ranges are shown for different homopolymers. Specific requirements will evolve from targeted applications, and polymers can be adapted through either copolymerization or blending. As long as bionutral degradable components are utilized, degradability should not expect to be altered. However, secondary reactions are possible and should be assessed before larger commercialisation efforts.

*Table 1: Thermal properties of selected biodegradable polymers and PA, PP for comparison [31–34]*

Biodegradable polymer	T <sub>g</sub> (°C)	T <sub>m</sub> (°C)
PLA	45–60	150–162
PLLA (L isomer of PLA)	55–65	170–200
PCL	–60––65	58–65
PHB	5–15	168–182
PH-octanoate	–32.6	162
PBS	–28.5	116.4
PA	50	265
PP	–15	176

A wide window of technical opportunities can be utilized by copolymerization, blending and the addition of plasticizers. Such additives need to be biodegradable too, so a wide range of materials can be considered, which is not covered here, but maybe in a future overview.

## 4 Degradation pathways

The molecular biodegradation of organic substances is usually conducted using microorganisms, such

as bacteria or fungi. For degradation in natural environments, the biodiversity of such organisms is crucial for ultimate biodegradability. Testing should therefore be done using organisms of ubiquitous occurrence in the targeted eco system and under respective test conditions. For industrial composting, the conditions can be adjusted, so a wider range of polymers may be utilized, if such a recycling method is targeted. Actually, commonly accepted test methods do not focus on that issue, which addresses a regulatory gap, should these substances gain wider market relevance.

Bionutral degradation can hypothetically occur chemically, since most of the mentioned polymers will eventually be hydrolysed. However, the rapid environmental ability to hydrolyse and lifetime durability frame a solid conflict of interest, which may be solved best only for single-use products or materials for indoor applications.

Degradation by microorganisms can take place intra or extra cellular. In the latter case, enzymes are secreted in the substrate which catalyse the degradation reactions. In every case, the physical properties of the polymer also contribute to the degradation properties. Besides the molecular weight, the crystallinity of the polymer also influences the degradation rate, since amorphous phases are generally more prone to the enzymatic attack [35, 36]. Also, higher melting temperatures are usually linked to the reduced ability to achieve enzymatic hydrolysis [37].

## 5 Costs

Many promising chemical efforts have been made to develop polymers with their respective production process to potentially produce biodegradable products. The issue of costs nevertheless remains a drawback. In 2000, the costs of PLA were estimated to be approx. five times that of PP, and PHA noted even more. A comparison with other commodity plastics do not show other major differences [38]. Ten years later, prices were reported to USD 3.5 to 4.5/kg for PHB and GBP 1.5 to 2.5/kg for PLA, while a prognosis for PHA cited that price could reach as low as EUR 1 to 2/kg [39]. In late 2017, prices for commodity polymers (US export) are as follows: USD 2.7/kg for PET, USD 8.4/kg for LDPE (low density polyethylene), USD 9.2/kg for PA and USD 1.9/kg for PLA [40] However, a wide range of prices was found depending on the exporting country. This shows that at least PLA prices are similar to those of commodity

polymers. Since lactic acid for the production of PLA is mostly synthesized through fermentation and PLA is gaining market share, the reduction in price may be a scaling effect. From this, it can be estimated that prices of other polymers such as PHA/B and PBS will also be competitive, if scaling markets will allow for that. Available data for the commodity pricing of PHB is rare, but Mudliar et al. published an evaluation of production costs for a hypothetical production plant, and determined a value of USD 5.38/kg for the best investigated case [41].

## 6 Textile applications

Synthetic polymers in textiles can be considered in three fields: the fibrous material itself, coatings and finishings, and fibre-reinforced composites. The latter has already been widely investigated for example using natural fibres and bio-based or biodegradable polymer matrices. Since matrix development is a broad field and is also closely linked to the used fibres, this topic is not further discussed here. However, interesting opportunities are given in the field of coating and finishing, and in the field of fibrous raw materials.

### *Research method*

To assess the current situation regarding the use of biodegradable polymers in these fields, a systematic literature and patent review was conducted. For this, the academic search engines *Google Scholar*, *Microsoft Academic*, *Crossref*, and *Clarivate's Web of Science* were consulted, while the patent research was conducted using a search engine provided by the German patent office.

The first three databases were queried using the keywords “*biodegradable polymers*” and a search period of the last 30 years if applicable. This delivered approx. 2,000 results. The results were exported to the open source literature database *JabRef* [42] and filtered by the regular expression `title="fibre"` or `title="fabric"` or `title="textile"`, resulting in 83 hits.

*Clarivate's Web of science* was queried using the regular expression `ts=(biodegradable polymer$) AND (Ti=textile$ or ti=fibre$ or ti=fabric$)` to search within the category of *biodegradable polymer(s)* for publications with *textile(s)*, *fibre(s)* or *fabric(s)* in their titles. The results were then refined using the *Web of Science* category *Materials science*, subcategory *textiles*, delivering 81 results.

Since the initial manual overview showed a high number of papers dealing with electrospun PLA nanofibres mostly for medical applications, both results were filtered in *JabRef* using `abstract!=PLA` and `title!=PLA` and `abstract!=nanofib` and `title!=nanofib` and `abstract!=electrospun` and `title!=electrospun` and `title!=lacti`, which excludes publications with the respective substrings in the title leaving 95 results that were manually evaluated by title and abstract, delivering 47 results.

A similar approach was applied for PLA and electrospun nanofibres, delivering 45 and 15 results, respectively, regarding these topics alone.

For the topic of biodegradable coatings, *Clarivate's Web of Science* was queried using `ts=(biodegradable coating$) AND (ts=textile$ or ts=fibre$ or ts=fabric$) AND ti=biodegradable`.

A patent search was conducted exclusively for the coating topic using `ti=biodegradable UND PUB>1.1.2000 UND AB=coating UND (AB=textile? ODER AB=fibre? ODER AB=fibre? ODER AB=fabric?) NICHT ti=wound NICHT ti=medical`, delivering 132 results which were manually revised, leaving 25 hits for further evaluation. *Ti* searches in the title, *PUB* defines the publication time, *AB* searches within the abstract and the question mark acts as wildcard for one or more characters. Since the search engine is German, the operator *UND* stands for *and*, *ODER* for *or*, and *NICHT* for *not*. However, international patents from the world patent office (WPO) were also delivered by these queries.

### *Results for PLA and electrospun fibres for medical applications*

As expected, PLA and its derivatives are the most common biodegradable polymers for fibre fabrication. A wide field of application is given in medical contexts. For example, stents made from PLA and coated by a layer-by-layer technique with chitosan have been investigated [43]. Scaffolds for tissue engineering are another field where physiological biodegradation is useful. The degradation of PLA in vivo may lead to local decrease of pH due to the produced lactic acid, while medium molecular weight oligomer degradation products can also potentially initiate immune responses in some patients. It is therefore crucial to understand the influences or fabrication parameters on the degradation rate, since extrusion speed and twist of the spun PLA fibres is reported to have an impact on those, besides the chemical composition of the polymer

[44]. Further research in that direction was done by studying the influence of plasma treatment on the moisture wicking behaviour of PLA fabrics [45]. Shape memory textiles are interesting materials in the medical field, for example for self-adjusting implants, scaffolds or anchor systems for grafts. Nonwoven materials fabricated using melt blown PLA-PHB blends have shown shape memory properties while being biodegradable [46]. Biodegradable ligament replacements can be considered to be made of PLA-PCL copolymers, so their creep and stress-relaxation properties have been investigated [47]. The rheological properties of PLLA and PLDA (L and D isomers of PLA) have been studied to produce physiological degradable fibres having a potential drug delivering property [48]. Hollow PLA fibres obtained by melt spinning can serve as platforms for the delivery of drugs in the medical field, but also for the retarded release of pesticides in agricultural settings. Melt spun PLA 9 fibres have been investigated for that purpose based on different physical properties [49]. Sutures are another field where PLA is a useful polymer. The stiffness of pure PLLA was reported to be reduced by blending with PLA-co-caprolactone copolymer [50].

In addition, potentially implantable sensors made from PLA components have been reported, with the advantage of not requiring explantation after therapy [51]. A special composite made from PLA and CNTs was produced using a melt blown process to produce sensory fibres [52].

The antibacterial properties of PLA fibres coated with titanium oxide have been investigated. However, the use of such material in medical applications requires a thorough assessment of possible side effects and cytotoxicity [53].

It can often be seen that lots of effort is put into developing new medical materials or devices, but regulatory issues are not addressed in the early stages of such research. This can lead to obstacles in the subsequent development to commercialized medical products [54]. Commercially available melt spun PLA fibres often contain finishings from secondary spinning, which may hinder medical uses. Jozwicka et al. studied different PLA fibre types and found glycerol as a suitable finish for medical appliances [55].

Electrospinning is a process capable of delivering nanofibre fabrics exhibiting a high surface area and small pore sizes, but can only be utilized in small scales. This issue renders the process suitable for only specialized applications. However, the possibility of

incorporating additional substances, such as pharmaceuticals, make these materials interesting candidates for drug delivery [56, 57]. A material spun from polyethylene oxide and poly vinyl alcohol was investigated for the use as a scaffold for potential tissue engineering [58]. In addition, blends of PLA and PCL with added hydroxyl apatite were investigated as electrospun nanofibre mats [59]. Additional insights on electrospun biodegradable scaffolds are given by Sun et al. [60]. A further detailed review on biodegradable polymers for tissue engineering is also given by Xing et al. [61].

#### *Summary of medical applications of PLA*

A wide range of ambitious applications can be seen for the utilization of PLA fibres in the medical sector. Electrospun nano fibres made from physiologically degradable polymers are promising substrates for tissue engineering and drug delivery.

It is therefore obvious that lots of effort is currently directed to that area. It is furthermore known that commercial feasibility is crucial for the widely accepted clinical use of medical products. One pathway is keeping the investment burden low by addressing regulatory issues in the early stages of research. This is especially necessary for the development of new textile products in that field, since they mostly address market fields with modest profit opportunities.

#### *Results beyond PLA and medical applications*

Research in biodegradable polymers in the textile context, excluding PLA and medical textiles, has been reported in fewer publications.

A drawback of PHBV (poly hydroxy butyrate valerate) lies in its low crystallization rate, which challenges melt spinning. Hufenus et al. investigated a core shell structure with PLA in different configurations, together with a special spinneret, to overcome this issue [62]. As early as 2000, Schmack et al. investigated the melt spinning of PHB and the dependency of drawing speed on the resulting crystal structures using wide-angle X-ray scattering [63] for use in textile applications. PHB and PHBV have limitations in the melt processing, since at higher temperatures (above 160 °C as reported), cis-elimination leads to random chain breaks. Moreover, bacterially produced PHB is strongly isotactic; together with its glass transition temperature of 5–15 °C, the material shows significant brittleness. The underlying cause of high crystallinity lowers its degradation rate under physiological conditions [64]. The authors also summarized different

works dealing with spinning conditions to control the crystallisation behaviour of the spun fibres, including the use of cyclodextrins as nucleation agents.

Bicomponent fibres were also studied based on the poly butylene terephthalate/poly butylene succinate lactate system. The drawing speed influenced the core shell ratio, while the range between 65% and 35% delivered the maximum fibre strength [65]. Also, here the ultimate biodegradability can be questioned due to the terephthalic acid monomer. Although less toxic, the substance can be considered a xenobiotic.

Twarowska-Schmidt evaluated two commercially available PBS derived polyesters (poly butylene succinate adipate (PBSA) and poly butylene adipate terephthalate) in terms of their fibre forming capability in a melt spin process, as well as their degradability. For the latter, only the weight loss of the fibres was recorded, so it is highly likely that at least terephthalic acid or potentially oligomers will be found in the substrate. As expected, the aromatic ester was degraded to a higher extent, but this only remains true for higher degradation temperatures. Thus, the general rule for the aliphatic esters is only partially valid here. Although the low glass transition temperatures of the materials (-33 °C and -44 °C) can be an issue for apparel, technical applications such as those in the medical or agricultural sector are considered to be feasible [66].

The cost issue of PHA is addressed by a recent publication regarding the production of PHB in cyanobacteria [67]. However, commodity prices for PHAs, PBS or similar polymers are not available, since the materials are not yet commercialized.

### *Summary beyond PLA and medical applications*

The remaining field, after excluding medical applications, composites and PLA materials, spans mostly across PBS and different types of PHA. The production of fibrous materials out of these polymers remains challenging, when mechanical requirements set higher stakes. While the degradation over time is the key feature in drug delivery systems, technical applications require specific tensile stiffness, elongation behaviour, brittleness, etc. together with textile haptics in the final fibrous product. Furthermore, the processing properties of the polymer must be met, e.g. processing temperatures, viscosities and crystallization rate, to successfully melt spin them into these fibres. Subsequently, the produced fibres must be spun secondarily to yarns and woven, knitted or fabricated otherwise into textiles.

### *Results of biodegradable textile coatings*

During the review of literature, it became evident that almost no scientific publications exist that address the coating of textiles using biodegradable polymers, with some exceptions in the medical field utilizing chitosan. Consequently, an additional literature search was conducted, together with a patent search on that topic.

Some authors have reported the development of a super hydrophobic coating made from soya bean oil, stearic acid and ZnO [68]. This approach will probably lack an ultimate biodegradability because of the ZnO particles. A similar approach was established using poly dopamine [69]. Yarn sizing using nano cellulose and soy protein has been investigated [70]. Antibacterial coatings achieved by silicone containing cations have also been reported [71], but the ultimate biodegradability can be questioned, at least because the ultimate degradation of silicones is not defined. As a result, the most meaningful definition may state silicon oxide as end product.

The patent search delivered even fewer results, excluding medical applications. Glycerol together with gelatine for coating of natural fibres is described in KR000100647872B1 (2006). A coating for a biodegradable textile is described in FR000002879224A1 (2004) using natural rubber latex, and proteins. The coating of sheet substrates and fabrics is considered in US020050031882A1 (2005) by PHA copolymers. An aqueous dispersion of PHB is described in EP000001566409A1 and in JP002004099883A (2003) for PVA, which can potentially be applied as a coating. JP002003221786A (2002) utilizes PVA together with sulfonic acid polymers for polyester fibre finishings. However, the sulfonic acid polymer may not be ultimately biodegraded. The Canadian application CA000002410457C (2010) deals with different PHA copolymers for the coating of fabrics, threads or yarns. In JP002002121288A (2002), the use of more or less degradable plasticizers for formulating an aqueous dispersion of a biodegradable resin is claimed.

### *Summary of biodegradable textile coatings*

Scientific literature only discusses textile, biodegradable coatings for non-medical applications to a lesser extent. However, patent literature shows some evidence for commercial attention in that area, although this is also limited. The patents found likely address textile coatings more as an additional option. But aqueous coating dispersions are also discussed there, which is valuable, since the discussed polymers in

this study are thermoplastics and one would consider extrusion coating in the first place. However, a water-based coating is a common textile technique, and the associated patents allow for conceiving such an application onto textiles too.

PVC (polyvinyl chloride) is a cheap and widely utilized coating substrate for textiles, but acrylates and polyurethanes are also applied. Thus, it is somehow surprising, that nearly no biodegradable alternatives are discussed in recent literature.

## 7 Future opportunities

Younes classified different biopolymers regarding their commercialization in a review in 2017. Classes ranged from *research, pilot plant, commercial, large scale, to mature* [72] Maturity was assigned only to cellulosic materials, which is not surprising. PLA was noted as the only one in the *large scale* group. PHA/B was classified between pilot plant and commercial, and PBS in the research state. It can be assumed that cost effects play a major role here, since petrol-based and non-biodegradable polymers are usually more cost efficient. Congruent with his findings is that PLA is narrowing its price gap to petrol-based commodity polymers. In the medical sector, physiological biodegradability represents an additional function that justifies higher costs. However, public pressure and/or regulatory issues may shift parts of the textile economy to biodegradable materials.

In the sub field of geo textiles, biodegradability can be conceived as an additional function, making it an interesting application sector. The need for development is given by the tailoring of the degradation rate, depending on operational conditions. It would also be a great advantage if such geo textiles could be fabricated with a defined lifetime and if the degradation rate over time could be engineered. But as discussed previously, manufacturing requirements must also be met by such new materials. Meeting both challenges may lead to new products, which could result in higher market prices. It should be noted that different semi-finished products can be found on the path from polymer, over fibres, yarns, possibly textile surfaces or nets to the finally confectioned product. Thus, many production steps must be covered.

Other fields in technical or apparel textiles will most probably not make use of the biodegradation function directly. That function may, however, be beneficial in the recycling of these products.

The direct utilization of fibrous materials in apparel can be considered. For this, however, different issues must be addressed: Again, the material properties must be designed so that production on conventional machinery for all intermediate semi-finished products is realizable. Secondly, the degradation rate must be tailored so that it will not take place at significant rates during the normal product life. Thus, hydrolyzability issues might need to be addressed. PHAs, in particular, may release fatty acids during hydrolyzation, which can lead to olfactory discomfort, even in lower amounts that do not affect the substrate's mechanical stability. Furthermore, such new products should exhibit comfortable textile haptics. However, synthetic materials offer numerous opportunities to fine tune these kinds of properties.

The literature and patent search resulted in a significant gap in the field of textile coatings. A few research bodies are already aware of this gap [73]. It is therefore a highly interesting field for research and new developments. It is also interesting for recycling purposes to develop materials composed of similar chemical substances, since no material separation is necessary. The variety of synthetic biodegradable polymers due to the abundance of raw materials, and the ability to form blends, copolymers and introduce additives can pave a way to coated textiles, where the fibrous substrate and the coating are made at least of similar biodegradable chemistry. Biodegradable fibres, either natural or manmade, are gaining market share in the technical textiles sector, while biodegradable coatings seem to lie inevitably on the way.

Coatings can be applied on textiles through the extrusion of thermoplastics or by applying aqueous dispersions of polymers, e.g. by knife coating or other means. One research target is therefore the manufacturing of such stable and applicable dispersions with the new polymers, which includes issues such as viscosity tailoring, degradation and storage stability, and drying properties of the applied wet film.

In addition, crosslinking offers the opportunity to develop possibly biodegradable duromers, which may also help to enhance the mechanical stability of such coatings. It is thus additionally of fundamental research interest to study the degradation pathway and decomposition products of such crosslinked polymers.

Further general fields of research are the development of biodegradable plasticizers, flame retardants, crosslinkers, and other additives.

## 8 Potential textile products

Many textile products can be conceived assuming the successful outcome of the above-described research and development efforts. To start with, blended fabrics, currently made of wool and polyester for suit fabrics, offer a high-priced market segment. The replacement of petrol-based polyester by a natural, ultimately biodegradable polyester could lead to a fully biodegradable product. Even buttons could be easily made of such polymers. While zippers out of plastics may not appear very reliable, buttons can be a replacing design element here. The additional feature of ultimate biodegradability may result in a premium on the price, considering current consumer sentiments. In the area of textile coatings application fields in protective clothing, textile membranes or laminates, carpets, tents, artificial leather and others can be considered, as they offer a wide range of addressable research topics. An interesting point here is the control of the degradation rate. It could be beneficial to switch the degradation behaviour of such coatings through environmental parameters, for example temperature together with humidity, or the presence of certain auxiliary substances (e.g. pH). The materials could be rendered resilient to the influence factors of the desired application, but could be compostable on an industrial scale. However, those materials may not degrade easily in natural environments. Thus, issues of littering or abrasion of micro particles cannot be avoided by chemical means alone.

## 9 Conclusion

For the assessment of manmade biodegradable polymers, first some definitions must be commonly accepted. Some already exist, but the handling of possible degradation products may not be regulated very well, although they are of great importance from an environmental point of view.

Only a few manmade biodegradable polymers in the narrow definition of this report are already commercialized. This is especially true for the textile sector beyond medical textiles, and fibre-reinforced composites. However, the most dominant polymer of that kind is PLA. Other polymers are in the research or development state, but offer interesting opportunities for textile fabrics and coatings due to the diversity of the substance classes. Coatings, in particular, are only rarely covered in recent literature, which makes

it a promising field of further development, given that textile base substrates will be replaced by more eco-friendly substances in the future.

## References

1. OECD guideline for testing of chemicals [accessible from a distance]. *OECD* [accessed 26.10.2020]. Available on World Wide Web: <<https://www.oecd.org/chemicalsafety/risk-assessment/1948378.pdf>>.
2. HAUS, Frédérique, GERMAN, Jean, JUNTER, Guy-Alain. Primary biodegradability of mineral base oils in relation to their chemical and physical characteristics. *Chemosphere*, 2001, **45**(6–7), 983–990, doi: 10.1016/S0045-6535(01)00027-3.
3. Urteil vom 04.04.2006 - 312 O 795/05 [accessible from a distance]. *Landgericht Hamburg OECD* [accessed 26.10.2020]. Available on World Wide Web: <<https://openjur.de/u/86339.html>>.
4. WEISSHAUPT, Petra. Abfallbehandlung biologisch abbaubarer Kunststoffe. In *18. BfR-Forum Verbraucherschutz : Mikroplastik*. Dessau-Roßlau : Umweltbundesamt, 2019, 1–15. Available on World Wide Web: <<https://mobil.bfr.bund.de/cm/343/abfallbehandlung-biologisch-abbaubarer-kunststoffe.pdf>>.
5. UNGER, Lena, SCHEIDELER, Marvin, MEYER, Pia, HARLAND, Julia, GÖRZEN, Andreas, WORTMANN, Martin, DREYER, Axel, EHRMANN, Andrea. Increasing adhesion of 3D printing on textile fabrics by polymer coating. *Tekstilec*, 2018, **61**(4), 265–271, doi: 10.14502/Tekstilec2018.61.265-271.
6. MOCHIZUKI, Masatsugu, HIRAMI, Matsuo. Biodegradable fibres made from truly-biodegradable thermoplastics. In *Polymers and Other Advanced Materials*. Edited by Paras N. Prasad, James E. Mark and Ting Joo Fai. New York : Springer, 1995, 589–596, doi: 10.1007/978-1-4899-0502-4\_61.
7. REDDY, C.S.K., GHAI, R., RASHMI, KALIA, V.C. Polyhydroxyalkanoates : an overview. *Bioresource Technology*, 2003, **87**(2), 137–146, doi: 10.1016/S0960-8524(02)00212-2.
8. ANDERSON, A.J., DAWES, E.A. Occurrence, metabolism, metabolic role, and industrial uses of bacterial polyhydroxyalkanoates. *Microbiology and Molecular Biology Reviews*, 1990, **54**(4), 450–472, doi: 10.1128/mmbr.54.4.450-472.1990.

9. PEDERSON, Erik N., McCHALICHER, Christopher W.J., SRIENC, Friedrich. Bacterial synthesis of PHA block copolymers. *Biomacromolecules*, 2006, **7**(6), 1904–1911, doi: 10.1021/bm0510101.
10. FRITZSCHE, Katharina, LENZ, Robert, FULLER, Clinton R. An unusual bacterial polyester with a phenyl pendant group. *Die Makromolekulare Chemie*, 1990, **191**(8), 1957–1965, doi: 10.1002/macp.1990.021910821.
11. MOŽEJKO-CIESIELSKA, Justyna, KIEWISZ, Robert. Bacterial polyhydroxyalkanoates : still fabulous? *Microbiological Research*, 2016, **192**, 71–282, doi: 10.1016/j.micres.2016.07.010.
12. THAORE, Vaishali, CHADWICK, David, SHAH, Nilay. Sustainable production of chemical intermediates for nylon manufacture : a techno-economic analysis for renewable production of caprolactone. *Chemical Engineering Research and Design*, 2018, **135**, 140–152, doi: 10.1016/j.cherd.2018.05.026.
13. LOPES, Milena S., JARDINI, André L., FILHO, Rubens M. Synthesis and characterizations of poly (lactic acid) by ring-opening polymerization for biomedical applications. *Chemical Engineering Transactions*, 2014, **38**, 331–336, doi: 10.3303/CET1438056.
14. CASTRO-AGUIRRE, E., IÑIGUEZ-FRANCO, F., SAMSUDIN, H., FANG, X., AURAS, R. Poly(lactic acid)-mass production, processing, industrial applications, and end of life. *Advanced Drug Delivery Reviews*, 2016, **107**, 333–366, doi: 10.1016/j.addr.2016.03.010.
15. MAZZOLI, Roberto. Metabolic engineering strategies for consolidated production of lactic acid from lignocellulosic biomass. *Biotechnology and Applied Biochemistry*, 2019, **67**(1), 1–13, doi: 10.1002/bab.1869.
16. SIN, Lee Tin, RAHMAT, Abdul Razak, RAHMAN, Wan Aizan Wan Abdul. *Poly(lactic acid) : plastics desing library*. Oxford : Elsevier, 2013, pp. 71–107, doi: 10.1016/B978-1-4377-4459-0.00002-0.
17. NGHIEM, Nhuan P., KLEFF, Susanne, SCHWEGMANN, Stefan. Succinic acid : technology development and commercialization. *Fermentation*, 2017, **3**(2), 1–14, doi: 10.3390/fermentation3020026.
18. JIANG, Min, MA, Jiangfeng, WU, Mingke, LIU, Rongming, LIANG, Liya, XIN, Fengxue, ZHANG, Wenming, JIA, Honghua, DONG, Weiliang. Progress of succinic acid production from renewable resources : metabolic and fermentative strategies. *Bioresource Technology*, 2017, **245**, 1710–1717, doi: 10.1016/j.biortech.2017.05.209.
19. WITT, U., EINIG, T., YAMAMOTO, M., KLEEBERG, I., DECKWER, W.D., MÜLLER, R.J. Biodegradation of aliphatic-aromatic copolyesters : evaluation of the final biodegradability and ecotoxicological impact of degradation intermediates. *Chemosphere*, 2001, **44**(2), 289–299, doi: 10.1016/S0045-6535(00)00162-4.
20. GRIVALSKÝ, Tomáš, RYCHLÝ, Jozef, RYCHLÁ, Lyda, BUČKOVÁ, Mária, KRAKOVÁ, Lucia, PUŠKÁROVÁ, Andrea, OROVČÍK, Lubomír, PANGALLO, Domenico. Aerobic biodegradation of aromatic aliphatic copolyester induced by bacteria obtained from different environments. *Journal of Polymers and the Environment*, 2018, **26**(2), 680–690, doi: 10.1007/s10924-017-0980-y.
21. SHAH, Aamer Ali, KATO, Satoshi, SHINTANI, Noboru, KAMINI, Numbi Ramudu, NAKAJIMA-KAMBE, Toshiaki. Microbial degradation of aliphatic and aliphatic-aromatic co-polyesters. *Applied Microbiology and Biotechnology*, 2014, **98**(8), 3437–3447, doi: 10.1007/s00253-014-5558-1.
22. KLEEBERG, Ilona, HETZ, Claudia, KROPENSTEDT, Reiner Michael, MÜLLER, Rolf Joachim, DECKWER, Wolf Dieter. Biodegradation of aliphatic-aromatic copolyesters by *Thermomonospora fusca* and other thermophilic compost isolates. *Applied and Environmental Microbiology*, 1998, **64**(5), 1731–1735, doi: 10.1128/aem.64.5.1731-1735.1998.
23. TOKIWA, Yutaka, SUZUKI, Tomoo. Hydrolysis of copolyesters containing aromatic and aliphatic ester blocks by lipase. *Journal of Applied Polymer Science*, 1981, **26**(2), 441–448, doi: 10.1002/app.1981.070260206.
24. EERHART, A.J.J.E., HUIJGEN, W.J.J., GRISEL, R.J.H., van der WAAL, J.C., de JONG, E., de SOUSA DIAS, A., FAAIJ, A.P.C., PATEL, M. K. Fuels and plastics from lignocellulosic biomass via the furan pathway : a technical analysis. *RSC Advances*, 2014, **4**(7), 3536–3549, doi: 10.1039/c3ra43512a.
25. TSANAKTSIS, Vasilios, BIKIARIS, Dimitrios N., GUIGO, Nathanael, EXARHOPOULOS, Stylianos, PAPAGEORGIOU, Dimitrios G., SBIRRAZZUOLI, Nicolas, PAPAGEORGIOU,

- George Z. Synthesis, properties and thermal behavior of poly(decylene-2,5-furanoate) : a biobased polyester from 2,5-furan dicarboxylic acid. *RSC Advances*, 2015, 5(91), 74592–74604, doi: 10.1039/c5ra13324f.
26. YANG, Yu, XIANG, Xi, TONG, Dongmei, HU, Changwei, ABU-OMAR, Mahdi M. One-pot synthesis of 5-hydroxymethylfurfural directly from starch over  $\text{SO}_4^{2-}/\text{ZrO}_2\text{-Al}_2\text{O}_3$  solid catalyst. *Bioresource Technology*, 2012, 116, 302–306, doi: 10.1016/j.biortech.2012.03.081.
  27. MASCAL, Mark, NIKITIN, Edward B. Direct, high-yield conversion of cellulose into biofuel. *Angewandte Chemie - International Edition*, 2008, 47(41), 7924–7926, doi: 10.1002/anie.200801594.
  28. MOTAGAMWALA, Ali Hussain, WON, Wangyun, SENER, Canan, ALONSO, David Martin, MARAVELIAS, Christos T., DUMESIC, James A. Toward biomass-derived renewable plastics : production of 2,5-furandicarboxylic acid from fructose. *Science Advances*, 2018, 4(1), 1–9, doi: 10.1126/sciadv.aap9722.
  29. RAJESH, Rajendran Omana, GODAN, Tharangattumana Krishnan, SINDHU, Raveendran, PANDEY, Ashok, BINOD, Parameswaran. Bioengineering advancements, innovations and challenges on green synthesis of 2, 5-furan dicarboxylic acid. *Bioengineered*, 2020, 11(1), 19–38, doi: 10.1080/21655979.2019.1700093.
  30. KLÄUSLI, Thomas. Kläusli, Ava Biochem : the bioeconomy is the biggest chance we have to decarbonise our world. *Bio-based News*, 26 April 2016. Available on World Wide Web: <<http://news.bio-based.eu/klaeusli-ava-biochem-the-bioeconomy-is-the-biggest-chance-we-have-to-decarbonise-our-world/>>.
  31. WANG, Lei, ZHANG, Min, LAWSON, Tom, KANWAL, Aqsa, MIAO, Zongcheng. Poly(butylene succinate-co-salicylic acid) copolymers and their effect on promoting plant growth. *Royal Society Open Science*, 2019, 6(7), 1–11, doi: 10.1098/rsos.190504.
  32. VAN de VELDE, K., KIEKENS, P. Biopolymers : overview of several properties and consequences on their applications. *Polymer Testing*, 2002, 21(4), 433–442, doi: 10.1016/S0142-9418(01)00107-6.
  33. ELIAS, Hans-Georg. *Makromoleküle*. Weinheim : Wiley, 1992.
  34. CHENG, Qingzheng, WANG, Siqun, RIALS, Timothy G., LEE, Seung Hwan. Physical and mechanical properties of polyvinyl alcohol and polypropylene composite materials reinforced with fibril aggregates isolated from regenerated cellulose fibres. *Cellulose*, 2007, 14(6), 593–602, doi: 10.1007/s10570-007-9141-0.
  35. IWATA, Tadahisa, DOI, Yoshiharu. Morphology and enzymatic degradation of poly(L-lactic acid) single crystals. *Macromolecules*, 1998, 31(8), 2461–2467, doi: 10.1021/ma980008h.
  36. TSUJI, Hideto, MIYAUCHI, Shinya. Poly(l-lactide) : VI effects of crystallinity on enzymatic hydrolysis of poly(l-lactide) without free amorphous region. *Polymer Degradation and Stability*, 2001, 71(3), 415–424, doi: 10.1016/S0141-3910(00)00191-9.
  37. TOKIWA, Yutaka, CALABIA, Buenaventurada P., UGWU, Charles U., AIBA, Seiichi. Biodegradability of plastics. *International Journal of Molecular Sciences*, 2009, 10(9), 3722–3742, doi: 10.3390/ijms10093722.
  38. MOHANTY, A. K., MISRA, M., HINRICHSEN, G. Biofibres, biodegradable polymers and biocomposites : an overview. *Macromolecular Materials and Engineering*, 2000, 276-277(1), 1–24, doi: 10.1002/(SICI)1439-2054(20000301)276:1<1::AID-MAME1>3.0.CO;2-W.
  39. RUDNIK, Ewa. *Compostable polymer materials*. Oxford : Elsevier Science, 2008, doi: 10.1016/B978-0-08-045371-2.X5001-X.
  40. Polyethylene terephthalate (PET): production, price, market and its properties [accessible from a distance]. *Plastics Insight* [accessed 03.11.2020]. Available on World Wide Web: <<https://www.plasticsinsight.com/resin-intelligence/resin-prices/polyethylene-terephthalate/>>.
  41. MUDLIAR, S. N., VAIDYA, A. N., SURESH KUMAR, M., DAHIKAR, S., CHAKRABARTI, T. Techno-economic evaluation of PHB production from activated sludge. *Clean Technologies and Environmental Policy*, 2008, 10(3), 255–262, doi: 10.1007/s10098-007-0100-0.
  42. KOPP et al. JabRef 4.3.1 [accessible from a distance]. *JabRef* [accessed 26.10.2020]. Available on World Wide Web: <<https://builds.jabref.org/>>.
  43. REBELO, Rita, VILA, Nivea, RANA, Sohel, FANGUEIRO, Raul. Poly lactic acid fibre based biodegradable stents and their functionalization techniques. In *Natural fibres : advances in science and technology towards industrial applications*. Edited by Raul Fanguero, Sohel Rana. Dordrecht : Springer, 2016, 331–342, doi: 10.1007/978-94-017-7515-1\_25.

44. GOLDING, C., EKEVALL, E., WALLACE, S.R., MATHER, R.R. The effect of degradation on the mechanical properties of biodegradable polylactide yarns and textiles. In *Medical textiles and biomaterials for healthcare*. Edited by S.C. Anand, J.F. Kennedy, M. Miraftab and S. Rajendran. Cambridge : Woodhead Publishing, 2006, 58–66, doi: 10.1533/9781845694104.1.58.
45. ABDRABBO, A., EL-DESSOUKY, H.M., FOTHERINGHAM, A.F. Treatment of polylactic acid fibre using low temperature plasma and its effects on vertical wicking and surface characteristics. *Journal of The Textile Institute*, 2013, **104**(1), 28–34, doi: 10.1080/00405000.2012.693699.
46. WALCZAK, Joanna, SOBOTA, Michal, CHRZANOWSKI, Michal, KRUCINSKA, Izabella. Application of the melt-blown technique in the production of shape-memory nonwoven fabrics from a blend of poly(L-lactide) and atactic poly[(R,S)-3-hydroxy butyrate]. *Textile Research Journal*, 2018, **88**(18), 2141–2152, doi: 10.1177/0040517517716906.
47. GUEDES, Rui Miranda, SINGH, Anurag, PINTO, Viviana. Viscoelastic modelling of creep and stress relaxation behaviour in PLA-PCL fibres. *Fibres and Polymers*, 2017, **18**(12), 2443–2453, doi: 10.1007/s12221-017-7479-y.
48. LUKANINA, K.I., SHEPELEV, A.D., BUDYKA, A.K. Synthesis of ultrafine fibres from L- and D,L-isomers of polylactide by electrospinning. *Fibre Chemistry*, 2012, **43**(5), 332–338, doi: 10.1007/s10692-012-9357-0.
49. NAEIMIRAD, Mohammadreza, ZADHOUSH, Ali, NEISIANY, Rasoul Esmaeely, SALIMIAN, Saeed, KOTEK, Richard. Melt-spun PLA liquid-filled fibres : physical, morphological, and thermal properties. *Journal of The Textile Institute*, 2019, **110**(1), 89–99, doi: 10.1080/00405000.2018.1465336.
50. VIJU, S., THILAGAVATHI, G., GUPTA, Bhuvanesh. Preparation and properties of PLLA/PLCL fibres for potential use as a monofilament suture. *Journal of The Textile Institute*, 2010, **101**(9), 835–841, doi: 10.1080/00405000902879718.
51. LUO, Mengdi, MARTINEZ, Adam W., SONG, Chao, HERRAULT, Florian, ALLEN, Mark G. A microfabricated wireless RF pressure sensor made completely of biodegradable materials. *Journal of Microelectromechanical Systems*, 2014, **23**(1), 4–13, doi: 10.1109/JMEMS.2013.2290111.
52. KRUCINSKA, Izabella, SURMA, Beata, CHRZANOWSKI, Michal, SKRZETUSKA, Ewa, PUCHALSKI, Michal. Application of melt-blown technology in the manufacturing of a solvent vapor-sensitive, non-woven fabric composed of poly(lactic acid) loaded with multi-walled carbon nanotubes. *Textile Research Journal*, 2013, **83**(8), 859–870, doi: 10.1177/0040517512460293.
53. SAFFARI, Mohammad-Reza, MIAB, Reza Kamali. Antibacterial property of PLA textiles coated by nano-TiO<sub>2</sub> through eco-friendly low-temperature plasma. *International Journal of Clothing Science and Technology*, 2016, **28**(6), 830–840, doi: 10.1108/IJCST-12-2015-0139.
54. OBERMANN, Malin, SCHWARZ-PFEIFFER, Anne, GRETHE, Thomas. Challenges in creating electronic medical textiles – a multidisciplinary effort. In *Proceedings of the Aachen-Dresden-Denkendorf International Textile Conference*, 2017, 1–5.
55. JOZWICKA, Jolanta, GZYRA-JAGIELA, Karolina, GUTOWSKA, Agnieszka, TWAROWSKA-SCHMIDT, Krystyna, CIEPLINSKI, Maciej. Chemical purity of PLA fibres for medical devices. *Fibres & Textiles in Eastern Europe*, 2012, **20**(6B), 135–141.
56. CUI, W., QI, M., LI, X., HUANG, S., ZHOU, S., WENG, J. Electrospun fibres of acid-labile biodegradable polymers with acetal groups as potential drug carriers. *International Journal of Pharmaceutics*, 2008, **361**(1–2), 47–55, doi: 10.1016/j.ijpharm.2008.05.005.
57. QI, M., LI, X., YANG, Y., ZHOU, S. Electrospun fibres of acid-labile biodegradable polymers containing ortho ester groups for controlled release of paracetamol. *European Journal of Pharmaceutics and Biopharmaceutics*, 2008, **70**(2), 445–452, doi: 10.1016/j.ejpb.2008.05.003.
58. SUBTIRICA, Adriana-Ioana, CHIVU, Andreea Ana-Maria, BANCIU, Cristina Antonela, DINCA, Laurentiu-Christian. Nanofibres made from biocompatible and biodegradable polymers, with potential application as medical textiles. *Industria Textila*, 2018, **69**(1), 55–58.
59. NETO, Wilson Alves Ribeiro, PEREIRA, Ildeu H L, AYRES, Eliane, de PAULA, Ana Claudia Chagas, AVEROUS, Luc, GOES, Alfredo M., OREFICE, Rodrigo Lambert, BRETAS, Rosario E.S. Influence of the microstructure and mechanical strength of nanofibres of biodegradable polymers with hydroxyapatite in stem cells

- growth. Electrospinning, characterization and cell viability. *Polymer Degradation and Stability*, 2012, **97**(10), 2037–2051, doi: 10.1016/j.polyimdegstab.2012.03.048.
60. SUN, J., BUBEL, K., CHEN, F., KISSEL, T., AGARWAL, S., GREINER, A. Nanofibres by green electrospinning of aqueous suspensions of biodegradable block copolyesters for applications in medicine, pharmacy and agriculture. *Macromolecular Rapid Communications*, 2010, **31**(23), 2077–2083, doi: 10.1002/marc.201000379.
  61. XING, Z.C., HAN, S.J., SHIN, Y.S., KANG, I.K. Fabrication of biodegradable polyester nanocomposites by electrospinning for tissue engineering. *Journal of Nanomaterials*, 2011, **2011**(929378), 1–18, doi: 10.1155/2011/929378.
  62. HUFENUS, Rudolf, LUBBEN, J.M., MANIURA, Katharina, DILETTOSO, Sandro, ZINN, Manfred. Biodegradable bicomponent fibers from renewable sources. In *Proceedings of the Fibre Society 2009 spring conference. Vols. I and II*. Raleigh : The Fiber Society, 2009, 1376–1378.
  63. SCHMACK, G., JEHNICHEN, D., VOGEL, R., TANDLER, B. Biodegradable fibres of poly (3-hydroxybutyrate) produced by high-speed melt spinning and spin drawing. *Journal of Polymer Science Part B – Polymer Physics*, 2000, **38**(21), 2841–2850, doi: 10.1002/1099-0488(20001101)38:21<2841::AID-POLB130>3.0.CO;2-#.
  64. YIXIAN, Luan, CHUNJU, Gu, WENHUA, Wu, MEIFANG, Zhu. Formation and mechanical properties of high-strength poly(3-hydroxybutyrate-co-hydroxyvalerate) fibres. In *Proceedings of 2009 International Conference on Advanced Fibres and Polymer Materials. Vols. I and II*. Beijing : Chemical Industry Press, 2009, 150–153.
  65. EL-SALMAWY, A., MIYAMOTO, M., KIMURA, Y. Preparing a core-sheath bicomponent fibre of poly(butylene terephthalate)/poly(butylene succinate-co-L-lactate). *Textile Research Journal*, 2000, **70**(11), 1011–1018, doi: 10.1177/004051750007001112.
  66. TWAROWSKA-SCHMIDT, K. Evaluation of the suitability of some biodegradable polymers for the forming of fibres. *Fibres & Textiles in Eastern Europe*, 2004, **12**(2), 17–20.
  67. BANERJEE, Apurba, SINGH, Manjinder, DAS, Keshav, SHARMA, Suraj. Study of biodegradable polyesters from algal sources for use in future textile fibre applications. *AATCC Journal of Research*, 2016, **3**(1), 1–6, doi: 10.14504/ajr.3.1.1.
  68. CHENG, Quan Yong, AN, Xu Pei, LI, Yi Dong, HUANG, Cai Li, ZENG, Jian Bing. Sustainable and biodegradable superhydrophobic coating from epoxidized soybean oil and ZnO nanoparticles on cellulosic substrates for efficient oil/water separation. *ACS Sustainable Chemistry and Engineering*, 2017, **5**(12), 11440–11450, doi: 10.1021/acssuschemeng.7b02549.
  69. DONG, Xiuli, GAO, Shouwei, HUANG, Jianying, LI, Shuhui, ZHU, Tianxue, CHENG, Yan, ZHAO, Yan, CHEN, Zhong, LAI, Yuekun. A self-roughened and biodegradable superhydrophobic coating with UV shielding, solar-induced self-healing and versatile oil/water separation ability. *Journal of Materials Chemistry A*, 2019, **7**(5), 2122–2128, doi: 10.1039/c8ta10869b.
  70. XU, Xiaoyun, SONG, Kaili, XING, Bin, HU, Wenfeng, KE, Qinfei, ZHAO, Yi. Thermal-tenacity-enhanced and biodegradable textile sizes from cellulose nanocrystals reinforced soy protein for effective yarn coating. *Industrial Crops and Products*, 2019, **140**, 1–9, doi: 10.1016/j.indcrop.2019.111701.
  71. MAO, Taoyan, WEI, Yuan, ZHENG, Cheng, CHENG, Wenjing, ZHANG, Zhenqiang, ZHU, Yiting, WANG, Runhao, ZENG, Zhaowen. Antibacterial cotton fabrics coated by biodegradable cationic silicone softeners. *Journal of Surfactants and Detergents*, 2019, **22**(6), 1429–1443, doi: 10.1002/jsde.12316.
  72. YOUNES, Basel. Classification, characterization, and the production processes of biopolymers used in the textiles industry. *Journal of the Textile Institute*, 2017, **108**(5), 674–682, doi: 10.1080/00405000.2016.1180731.
  73. KLIMMT, Oliver. Projekt Abbaubare Textilbeschichtung auf PBS-Basis : BMWi INNO-KOM 49MF180159 (Laufzeit: 03.2019 – 08.2021). Freiberg: Forschungsinstitut für Leder und Kunststoffbahnen gGmbH (FILK). Available on World Wide Web: <<https://www.filkfreiberg.de/en/research/research-projects-and-publications/current-projects.html>>.

Madan Lal Regar<sup>1</sup>, Akhtarul Islam Amjad<sup>2</sup>, Shubham Joshi<sup>2</sup>

<sup>1</sup> National Institute of Fashion Technology, Department of Fashion Design, Jodhpur, Raj, 342037, India

<sup>2</sup> Uttar Pradesh Textile Technology Institute, Department of Textile Engineering, Kanpur, UP, 208001, India

---

## Effect of Solvent Treatment on Siro and Ring Spun TFO Polyester Yarn

*Vpliv obdelave s topilom na poliestrni preji, izdelani po postopku SiroSpun® in prstanskem postopku*

Original scientific article/Izvirni znanstveni članek

Received/Prispelo 7-2020 • Accepted/Sprejeto 10-2020

Corresponding author/Korespondenčni avtor:

Assist Prof Dr. Madan Lal Regar

E-mail: madan.regar@nift.ac.in

Phone: +91-7307367637

ORCID: 0000-0001-7821-4410

---

### Abstract

Yarns and fabrics can be improved through structural modifications. Using an organic solvent is a novel and popular approach for a structural modification in the field of textiles. In the present work, Siro-spun® and TFO (Two for one Twisters) polyester yarns were modified with a solvent-acid mixture of acetone and trichloroacetic acid : methylene chloride (TCAMC). Both types of yarn samples were treated in a relaxed state with various concentrations of the solvent's mixture at room temperature. The influence of the treatment with respect to linear density and TCAMC concentration on mechanical properties was investigated. Modified yarns exhibited higher breaking elongation, improved abrasion resistance and lower tenacity. It was also found that the methods of yarn manufacturing contribute significantly to the tensile behaviour of modified yarns. The improvement in elongation was higher in the treated TFO yarn. The abrasion resistance index was found to be lower in the case of siro polyester yarns. Furthermore, significant mass loss in yarn was observed after the treatment of TCAMC. Keywords: TCAMC, modification, polyester, siro, spinning

### Izvleček

Lastnosti preji in ploskovnih tekstilij je mogoče izboljšati s strukturnimi spremembami. Uporaba organskega topila je nov in priljubljen način strukturnega modificiranja tekstilij. V tej raziskavi sta bili poliestrski preji, izdelani po postopkih vitja dveh stenjev v sukano prejo (Siro-spun®) in z uporabo sukalnika z dvojnimi vitjem (Two-for-One, TFO), modificirani z mešanico organskih topil trikloroacetne kisline metilenklorida (TCAMC) in acetona. Obe preji sta bili obdelani v nevpetem stanju z različno koncentracijo mešanice topil pri sobni temperaturi. Raziskan je bil vpliv koncentracije TCAMC na mehanske lastnosti modificiranih prej z različno dolžinsko maso. Modificirane preje so imele večji pretržni raztezek, izboljšano odpornost na drgnjenje in nižjo natezno trdnost. Ugotovljeno je bilo, da sta tudi sama postopka izdelave preje pomembno vplivala na natezne lastnosti modificiranih prej. Večji raztezek je imela modificirana preja po postopku TFO. Indeks odpornosti preje na obrabo (RRI) je bil pri poliestrski preji SiroSpun nižji. Pri obdelavi s topilom TCAMC in acetona se je občutno zmanjšala tudi masa preje.

Ključne besede: TCAMC, modifikacija, poliester, SiroSpun, predenje

---

## 1 Introduction

Properties of spun yarns are mainly affected by fibre properties, process parameters and the spinning system. Fibre properties and process parameters mainly influence the appearance (melange, slub and neppy yarns), and the mechanical and physical properties [1]. The spinning system influences the yarn structure, as each spinning system tends to produce a distinctive yarn structure with distinct yarn properties. Recent refinements in spinning technologies have yielded significant improvement in the yarn structure [2]. Traditionally, two-fold yarns have been used for weaving since they are stronger and the twisting operation binds the surface fibres into the structure, the latter becoming smoother and more resistant to abrasion during weaving. Siro-spinning with 2-ply multifold yarn is another most widely used new spinning method invented by CSIRO (Division of Textile Industry Laboratories) in Australia and IWS. Such yarn is produced on a conventional ring frame with two separate strands fed in the drafting zone during the spinning process at a predetermined separation. Siro spun yarn is especially better with regard to its tensile properties, while the conventional plying process attracts attention mainly due to lower yarn hairiness [3].

Polyester fibres have been widely used in the textile industry due to its high strength, good handling and easy-care properties. However, polyester fibres exhibit low surface energy and limited chemical reactivity, which lead to poor moisture absorption and accumulation of static charge. In the recent years, various new methods have been identified to modify the surface characteristics of polyester fibres [4]. When polyester fibres are pretreated with highly interacting solvents under suitable conditions, the internal structure, especially the amorphous region, changes producing more voids, cracks etc., which facilitate the entry of dye molecules at lower temperatures. Such modified polyester fibres are developed to replace the conventional ones in a wide range of applications [5–6].

Surface modifications with after treatments of chemicals are another approach for getting a distinct structure and properties in final yarn. The surface modification of polyester suggested improving the hydrophilicity of polyester structure by enzyme treatment, esterase, thermal plasma treatment etc. [7–9]. Researchers have suggested a new approach to modify the internal structure, i.e. by using the TCAMC reagent [10–11]. It can be done at fibre, yarn and fabric stages. In the treatment, the solvent-induced modifi-

cation principle is used to modify polyester yarn. The fast treatment rate at room temperature and easy storability make the TCAMC reagent more appealing for a structural modification of yarn and fabrics [12–13]. In the present study, an attempt was made to compare the effect of a different TCAMC concentration level on the tensile and abrasion properties of TFO and siro yarn. The effect of the treatment on tensile strength and breaking extension was studied for different linear density yarns.

## 2 Materials and methods

### 2.1 Yarn preparation

For the present study, polyester (1.33 dtex (1.2 denier), 38 mm) fibre was used to produce TFO and siro yarn. Siro yarns were produced on a Lakshmi short staple spinning line with 8 mm distance between two roving strands. For TFO yarn, single yarns were spun on a Lakshmi spinning line and doubled on a Saurer compact twist TFO machine. The twist multiplier 4.2 and spindle speed were constant in both systems producing yarns.

### 2.2 TCAMC treatment

The laboratory grade trichloroacetic acid : methylene chloride ( $\text{CCl}_3\text{COOHCH}_2\text{Cl}_2$ ) and acetone ( $\text{CH}_3\text{COCH}_3$ ) were used for the surface modification of yarn samples. The pre-treatment of these yarns with a reagent was conducted in a specially made closed trough at room temperature (27 °C). The yarn specimens were immersed in the reagent of desired concentrations of 0.5%, 2.5% and 4.5% (w/v) for 2 min. The yarn to solvent ratio was 1 : 100 and the contents were stirred manually. After the treatment, the specimens were rinsed with pure methylene chloride, followed by acetone to remove any adhering reagent from the yarn. The treated yarns were squeezed and air-dried at atmospheric conditions, taking the advantage of the quick evaporation of acetone at room temperature. A total of 18 samples were made for the study. The design plan of the experiment is given in Table 1.

### 2.3 Testing methods

The yarns were conditioned for 24 hours at standard atmospheric conditions of  $65\% \pm 2\%$  RH and  $27\text{ °C} \pm 2\text{ °C}$  temperature. The number of tests for each parameter was taken to ensure the result to remain within the 95% confidence limit. For a surface analysis, images of treated and untreated samples were captured on a stereo image analyser.

Table 1: Experiment design plan

Sr. No.	Factor	Level					
		TFO			Siro		
1	Spinning system						
2	Linear density (tex/Ne)	23.6 (2/50)	29.5 (2/40)	39.4 (2/30)	23.6 (2/50)	29.5 (2/40)	39.4 (2/30)
3	TCAMC concentrations (%)	0.5	2.5	4.5	0.5	2.5	4.5

### 2.3.1 Weight loss of yarn

Leas of all yarn samples were weighed before and after the treatment, and the difference in mass of all samples was measured in terms of weight loss. Standard percentage of the mass loss was calculated using the formula given below:

$$\text{Weight loss (\%)} = \frac{\text{Yarn weight before treatment} - \text{Yarn weight after treatment}}{\text{Yarn weight before treatment}} \times 100 \quad (1)$$

### 2.3.2 Tensile testing of yarn

A Zwick universal tensile tester was used to measure the tensile properties according to ASTM D 2256. The yarns were tested at 120 mm/min extension rate using gauge length of 250 mm. At least 30 readings were taken for each sample. The effect of the treatment on tensile strength was evaluated in terms of strength loss. Strength loss (%) was calculated according to the formula:

$$\text{Strength loss (\%)} = \frac{\text{Yarn strength before treatment} - \text{Yarn strength after treatment}}{\text{Yarn strength before treatment}} \times 100 \quad (2)$$

Similarly, extension gain was evaluated with the difference in extension before and after the treatment. Extension gain was calculated according to the formula below:

$$\text{Extension gain (\%)} = \frac{\text{Yarn extension after treatment} - \text{Yarn extension before treatment}}{\text{Yarn extension before treatment}} \times 100 \quad (3)$$

### 2.3.3 Yarn abrasion resistance

The abrasion resistance of yarns was tested on a yarn abrasion tester following ASTM D-4157. The abrasion resistance was expressed in terms of the number of strokes required to rupture yarns completely. A parallel sheet consisting of 20 yarns was kept pressed at constant tension (20 g) against a cylinder wrapped with an abrader. The yarns were abraded by the cylinder surface while it oscillated at constant speed and stopped when all the yarns broke. Relative Resistances Index (RRI) was converted with the average number of strokes by including both the liner density of yarn and the applied pre-tension [3]. The RRI used to compare the resistance of yarns varying in linear density is defined by:

$$\text{RRI} = \frac{\text{No of strokes} \times \text{Pre-tension (g)}}{\sqrt{\text{Liner density (tex)}}} \quad (4)$$

The effect of the treatment on abrasive properties was evaluated in terms of change in percentage for RRI. The change in RRI was calculated with the difference in RRI before and after the treatment.

## 3 Results and discussion

This paper embodies the effect of the concentration level of trichloroacetic acid methylene chloride on weight, tensile and abrasion properties of TFO and siro yarn. The results were analysed before and after the treatment. The results were evaluated in terms of weight loss, strength loss, gain in extension and abrasion relative resistance index (RRI). An analysis of variance was performed to find the effect of the spinning system, linear density and concentration level on the analysed parameters. Table 2 shows ANOVA analysis results for the studied yarn properties.

Table 2: ANOVA result

Effect of	Weight loss		Strength loss		Increase in extension		Increase in RRI	
	F-value	p-value	F-value	p-value	F-value	p-value	F-value	p-value
S S	113.46	0.0004 (S)	124.02	0.0003 (S)	143.92	0.0003 (S)	39.74	0.0032 (S)
L D	98.46	0.0004 (S)	78.65	0.0006 (S)	86.94	0.0005 (S)	58.04	0.0011 (S)
T C	818.85	0.0001 (S)	467.74	0.0001 (S)	617.57	0.0000 (S)	392.14	0.0001 (S)
S S*L D	1.35	0.36 (NS)	3.39	0.14 (NS)	10.65	0.024 (NS)	1.10	0.42 (NS)
S S*T C	45.58	0.0018 (S)	8.68	0.0351 (S)	3.55	0.129 (NS)	5.65	0.068 (NS)
L D*T C	5.44	0.0483 (S)	8.37	0.0317 (S)	6.12	0.0500 (S)	3.53	0.124 (NS)

S S – Spinning system, L D – Linear density, T C – TCAMC concentration, RRI – Relative resistance index, S – Significant, NS – Non-significant,

It can be observed from Table 2 that the effect of the spinning system, linear density and concentration level was found significant for all evaluated properties.

### 3.1 Effect of treatment on yarn surface

Fibre arrangement in yarn being system specific, a different spinning system results in a different arrangement and/or distribution of fibres, causing variation in the product surface and performance. The studied yarn was produced on two different spinning systems; hence, the different structure resulted in the variation of yarn porosity. The effect of the TCAMC solvent on yarn surface is shown in Figure 1.

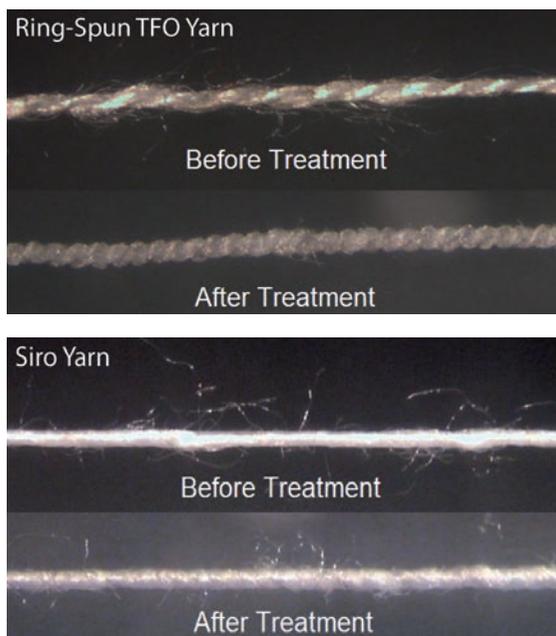


Figure 1: Effect of TCAMC treatment on yarn surface

As Figure 1 shows, the contraction was higher in the case of TFO yarn as compared to siro yarn. The TCAMC treatment causes an increase in the chain mobility and results in a structural rearrangement of polymer chains, which leads to a change in yarn structure. The surface of TFO yarn is helical while the surface of siro yarn is regular. In the case of TFO yarn, the more space is available in its structure for a rearrangement [2]. Thus, TFO yarn was more affected by the treatment and resulted in a more substantial contraction in TFO yarn.

### 3.2 Effect of TCAMC treatment on weight loss

The weight loss percentage, which is the difference between the specimen mass before and after the treatment, was observed. The weight loss (%) after the treatment for both types of yarns is presented in Figure 2.

Mass loss was observed in all types of treated yarns. Furthermore, it was found that an increment in the concentration of TCAMC increased weight loss in yarn. Higher weight loss was found in higher linear density (tex) yarns at the same level of concentration. Higher linear density of yarn possesses more surface area, which leads to a larger surface to react with TCAMC, causing greater mass reduction.

It was also observed that weight loss was higher in the case of TFO yarns compared to siro spun yarns. The inherited structural difference was the reason for higher mass loss in TFO yarns.

### 3.3 Effect of treatment on tensile strength

The strength loss percentage of both siro and TFO yarns is shown in Figure 3, which depicts that the strength of TCAMC-treated yarns decreased with

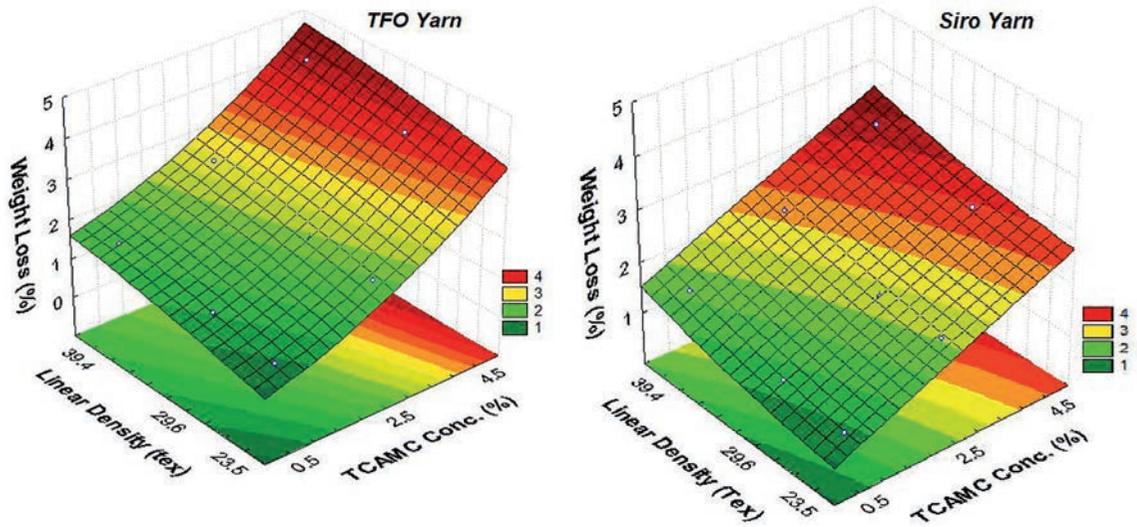


Figure 2: Effect of TCAMC concentration and linear density on weight of polyester yarns

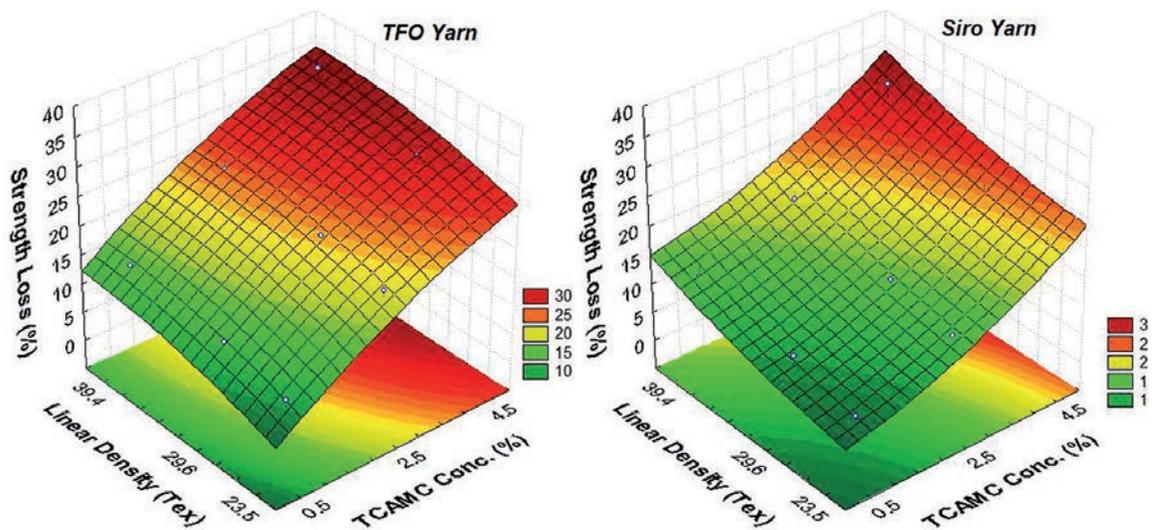


Figure 3: Effect of TCAMC concentration and linear density on strength of polyester yarns

an increase in the concentration of TCAMC. The strength loss was about 7.21–34.28% for TFO yarns and 7.27–31.66% for siro yarns, which shows that the effect of the TCAMC treatment on the strength of TFO yarn was greater than on siro yarn. The structural arrangement in both yarns was different as TFO yarn is made of two single yarns and less compact, while siro yarn is compacter and made of single entity yarns. TFO yarn possesses intra- and inter-yarn spaces, and siro yarn possess only intra-yarn spaces. Therefore, when the treatment was applied on yarn, the possibility of a solvent penetrating into TFO yarn was greater. As a consequence of greater penetration,

the surface became more modified, resulting in higher strength loss in TFO yarns.

The change in the tensile strength of polyester yarns even at lower treatment concentrations suggests that the TCAMC treatment increases the chain mobility and results in the structural rearrangement of polymer chains. Furthermore, it appears that at higher treatment concentrations, TCAMC is capable of penetrating into the crystalline domains of polyester and breaks the forces present in it, resulting in significant strength loss. It is expected that beyond 10% treatment concentrations, TCAMC would disorient crystallinity and facilitate the disintegration of fibres [11].

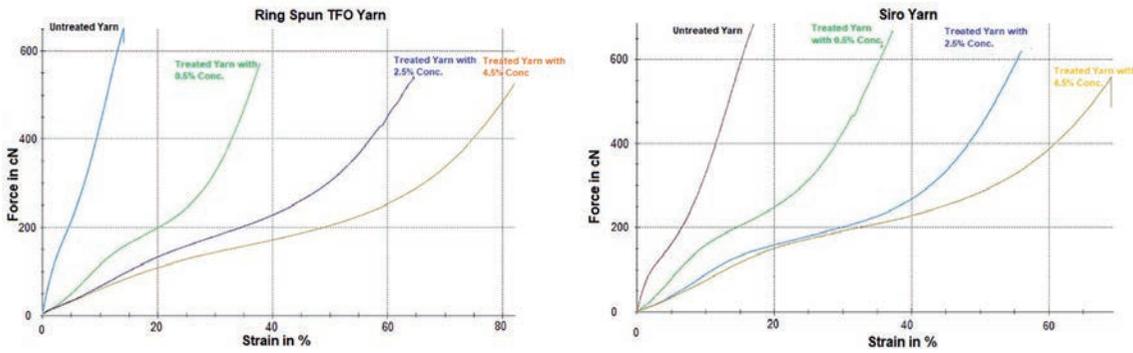


Figure 4: Load strain behaviour of untreated and treated polyester yarn (23.6 tex)

Figure 4 shows the load extension behaviour of the two yarns. Siro yarn showed marginally higher breaking load for both treated and untreated yarn compared to TFO yarn. It was also observed that the breaking extension value of treated TFO yarn was higher than that of siro yarn. Moreover, it was found that the linear density of yarn also affects the strength loss of yarn.

### 3.4 Effect of treatment on breaking extension

When yarn is subjected to load, constituent fibres share that load. The sharing of load is influenced by the arrangement of these constituent fibres. The arrangement and configuration of fibres again depends on the mode of yarn formation. Depending on the level of load in each fibre, straightening, slippage and/or breakage of fibres can take place, which is manifested as yarn extension [3]. The breaking extension of a synthetic fibre depends on the structural characteristics, e.g. configuration of chain molecules and

energy content due to chemical bonds between the atoms in the chains, and the intermolecular bonds between the chains. Figure 5 shows the increase in breaking extension values of both siro and TFO yarns with the applied treatment.

Figure 5 shows that the breaking extension increased with increased TCAMC concentration. The improvement in the breaking extension of studied yarns at lower treatment concentrations suggests that the TCAMC treatment produces some degree of molecular movement by relaxing lateral forces (intermolecular bonds), which are responsible for holding the chain molecules inside the polymer.

Furthermore, it can be seen in Figure 5 that the gain in breaking extension for TFO yarn is greater if compared to siro yarn due to the difference in the structure of TFO and siro yarn. The effect of the TCAMC treatment on the helical structure is greater, leading to more contraction in the yarn if compared to siro yarn.

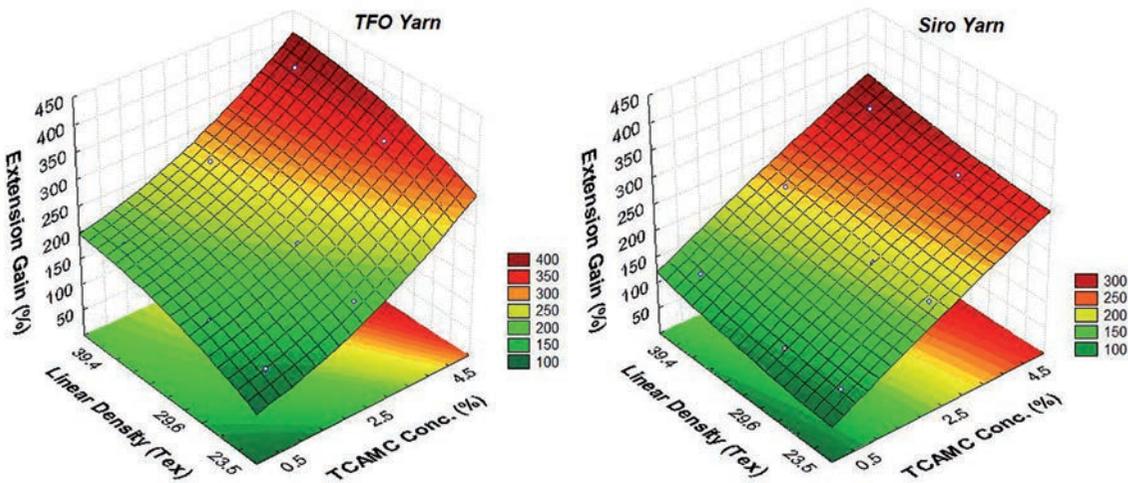


Figure 5: Effect of TCAMC concentration and linear density on breaking extension of polyester yarns

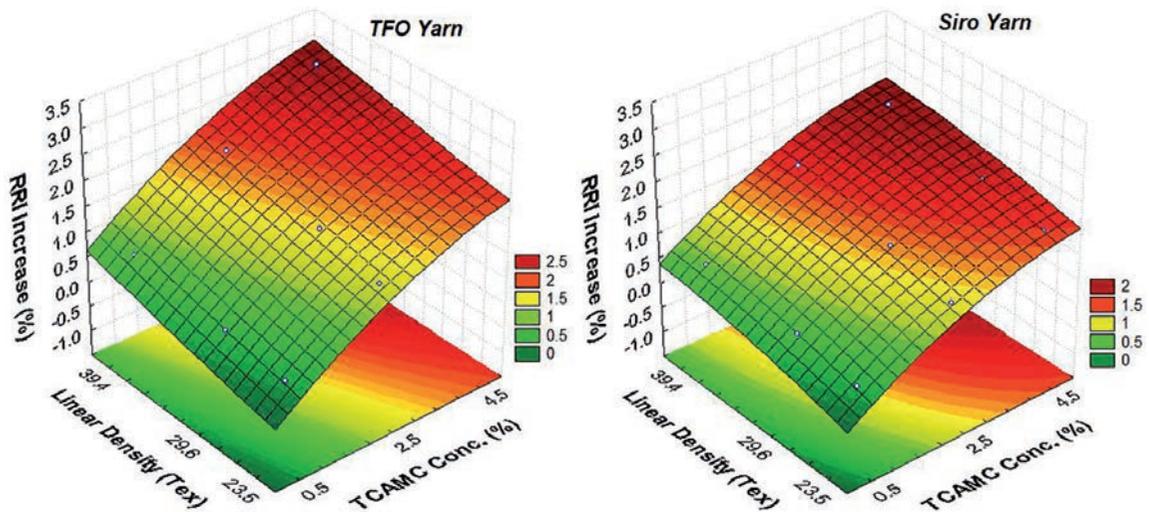


Figure 6: Effect of TCAMC treatment and linear density on RRI of polyester yarns

### 3.5 Effect of treatment on abrasion resistance (RRI)

Abrasion property is the key factor to predict how yarn performs under high abrasion resistance. A physical destruction of fibres, yarns, resulting from rubbing a surface over another surface is known as abrasion. Abrasion distorts the appearance by pulling fibre ends out from the yarn surface.

Figure 6 demonstrates that RRI increases with an increase in the TCAMC concentration. The highest relative resistance index was observed for the 4.5% concentration level. It appears that higher breaking extension of polyester yarn imparts the plasticisation effect during the treatment. The extensibility of yarn possesses more resistance to yarn abrasion and vice versa. As it can be seen in Figure 6, the increase in RRI for TFO yarn is greater than for siro yarn due to the higher breaking extension of the treated TFO yarn. The surface fibres of individual ply for TFO yarn are protected after the doubling and are relatively difficult to open. On the other hand, the surface fibres in siro yarn are likely to come out of the structure relatively easy during abrasion.

## 4 Conclusion

A solvent treatment of TCAMC on siro and TFO yarns significantly influences the extensibility, strength and abrasion properties. The strength of treated yarn decreases with increased solvent concentration. A 7.14–34.18% strength drop was found

for TFO yarn and 7.27–31.66% for siro yarns. The treatment caused a reduction in mass of both types of polyester yarn. A higher concentration of TCAMC caused greater weight reduction in yarns. Higher contraction was found in the case of TFO yarn compared to siro yarn after the treatment. The treated polyester yarns exhibited an improvement in breaking extension at a higher concentration of TCAMC. It can be concluded that TCAMC treatment modifies the structural assembly of polyester yarn by relaxing lateral forces. The treatment also influences the abrasive properties of both types of yarn. RRI for TFO yarn is higher if compared to siro yarn.

## References

1. AMJAD, A. Islam, KUMAR, Rajiv. Evaluation of mechanical and physical characteristics of eco blended melange yarns. *Tekstilec*, 2020, **63**(2), 94–103, doi: 10.14502/Tekstilec2020.63.94-103.
2. REGAR, Madan Lal, SINHA, Sujit Kumar, CHATTOPADHYAY, R. Fibre distribution and packing in Eli-Twist, Siro & ring spun TFO yarn. *Journal of The Institution of Engineers (India): E series.*, 2018, **99**(2), 177–186, doi: 10.1007/s40034-018-0126-3.
3. REGAR, Madan Lal, SINHA, Sujit Kumar, CHATTOPADHYAY, R. Comparative assessment of Eli-Twist and Siro yarn made from polyester and its blend with cotton. *Indian Journal of Fibre and Textile Research*, 2019, **44**(3), 299–305.

4. MITTAL, K. L. *Polymer surface modification: relevance to adhesion*. Vol. 3. Boca Raton : CRC Press, 2004, p.10.
5. BENDAK, A., EL-MARSAFI, S.M. Effects of chemical modifications on polyester fibres. *Journal of Islamic Academy of Sciences*, 1991, 4(4), 275–284.
6. CHIDAMBARAM, D., VENKATRAJ, R., MANISANKAR, P. Solvent-induced modifications in polyester yarns. I. Mechanical properties. *Journal of Applied Polymer Science*, 2002, 87(9), 1500–1510, doi: 10.1002/app.11531.
7. WANG, Chi., LEE, Ming Feng., WU, Yi Jiun. Solution-electrospun poly (ethylene terephthalate) fibers : processing and characterization. *Macromolecules*, 2012, 45(19), 7939–7947, doi: 10.1021/ma300121k.
8. GUPTA, B., HILBORN, J., HOLLENSTEIN, Ch., PLUMMER, C.J.G., HOURIET, R., XANTHOPOULOS, N. Surface modification of polyester films by RF plasma. *Journal of Applied Polymer Science*, 2000, 78(5), 1083–1091, doi: 10.1002/1097-4628(20001031)78:5<1083::AID-APP170>3.0.CO;2-5.
9. WU, Jindan, CAI, Guoqiang, LIU, Jinqiang, GE, Huayun, WANG, Jiping. Eco-friendly surface modification on polyester fabrics by esterase treatment. *Applied Surface Science*, 2014, 295,150–157, doi: 10.1016/j.apsusc.2014.01.019.
10. RAJENDRAN, S., RAMASAMY, S.S., MISHRA, S.P. Influence of solvent pre-treatment on dyeing performance of polyester yarns. *Indian Journal of Fibre and Textile Research*, 1997, 22(1), 44–52.
11. RAJENDRAN, S., RAMASAMY, S.S., MISHRA, S.P. Tensile behavior of polyester yarns modified by trichloroacetic acid–methylene chloride treatment. *Journal of Applied Polymer Science*, 1996, 59(1), 99–108, doi: 10.1002/(SICI)1097-4628(19960103)59:1<99::AID-APP15>3.0.CO;2-3.
12. RAJENDRAN, S., MISHRA, S.P. Chemical structural and thermal changes in PET caused by solvent induced polymer crystallization. *Polymers & Polymer Composites*, 2007, 15(2), 103–110, doi: 10.1177/096739110701500203.
13. RAJENDRAN, S., RAMASAMY, S.S., MISHRA, S.P. Abrasion resistance of modified polyester yarns. *Indian Journal of Fibre and Textile Research*, 1996, 21(4), 276–279.

Shubham Joshi<sup>1</sup>, Vinay Midha<sup>1</sup>, Subbiyan Rajendran<sup>2</sup>

<sup>1</sup> Dr B. R. Ambedkar National Institute of Technology, Department of Textile Technology, Jalandhar, 144011, India

<sup>2</sup> The University of Bolton, Institute for Material Research and Innovation, Bolton, BL3 5AB, United Kingdom

# Investigation of Durable Bio-polymeric Antimicrobial Finishes to Chemically Modified Textile Fabrics Using Solvent Induction System

*Raziskava trajnih biopolimernih protimikrobnih apretur za kemijsko plemenitenje tekstilij z uporabo indukcijskega sistema s topilom*

Original scientific article/Izvirni znanstveni članek

Received/Prispelo 8-2020 • Accepted/Sprejeto 10-2020

Corresponding author/Korespondenčni avtor:

Mr. Shubham Joshi

E-mail: shubhambmjoshi@gmail.com

Telephone: +917814086011

ORCID: 0000-0003-0292-5317

## Abstract

New technologies and materials required for developing antibacterial textiles have become a subject of interest to the researchers in recent years. This study focuses on the investigation of the biopolymeric antibacterial agents, such as neem, aloe vera, tulsi and grapeseed oil, in the trichloroacetic acid-methylene chloride (TCAMC) solvent used for the pretreatment of polyethylene terephthalate (PET) polyester fabrics. Different PET structures, such as 100% polyester, polyester/viscose, polyester/cotton and 100% texturised, are treated with four different concentrations (5%, 10%, 15% and 20%) of biopolymeric antibacterial finishes. The antibacterial activity of the treated samples is tested against both the *Staphylococcus aureus* (Gram-positive) and *Escherichia coli* (Gram-negative) bacteria. Taguchi mixed orthogonal array Design L16 ( $4^3 2^2$ ) is chosen for an experimental plan to determine the optimum conditions. Among all the fabric samples, the 100% polyester fabric treated with 20% grapeseed oil registers the highest antibacterial activity of 86%, and 73% against *S. aureus* and *E. coli* respectively. However, the antibacterial effect is reduced to 37%, and 34% respectively after 10 machine launderings. Keywords: solvent induced polymerisation, polyester and polyester blends, natural extracts, antibacterial activity, trichloroacetic acid-methylene chloride

## Izveček

Nove tehnologije in sredstva za razvoj protibakterijskih tekstilij so v zadnjih letih v središču zanimanja raziskovalcev. Ta študija se osredinja na raziskave biopolimernih protibakterijskih sredstev, kot so indijska melija, aloe vera, sveta bazilika in olje grozdnih pešk, na poliestrskih tkaninah, predhodno obdelanih s topilom trikloroocetne kisline in metilenklorida (TCAMC). Tkanine iz 100-odstotnega poliestra, poliestra/viskoze, poliestra/bombaža in iz 100-odstotnega teksturiranega poliestra so bile obdelane z biopolimernimi protibakterijskimi sredstvi štirih različnih koncentracij (5 %, 10 %, 15 % in 20 %). Protibakterijsko delovanje obdelanih vzorcev je bilo preizkušano proti bakterijama *Staphylococcus aureus* (Grampozitivna bakterija) in *Escherichia coli* (Gramnegativna bakterija). Eksperimentalni načrt za določitev optimalnih pogojev je bil izdelan z uporabo Taguchijevega mešanega pravokotnega niza L16 ( $4^3 2^2$ ). Med vsemi vzorci je imela

tkanina iz 100-odstotnega poliestra, obdelana z 20-odstotnim oljem iz grozdnih pešk, najvišjo, 86-odstotno protibakterijsko aktivnost proti *S. aureus* in 73-odstotno aktivnost proti *E. coli*. Protibakterijski učinek po desetih laboratorijskih pranjih se je zmanjšal na 37 oziroma 34 odstotkov.

Ključne besede: polimerizacija v raztopini, poliester, mešanice s poliestrom, naravni ekstrakt, protibakterijska aktivnost, trikloroocetna kislina-metilenklorid

## 1 Introduction

It is well established that textiles are used by pathogenic microorganisms to promote and spread diseases. The microorganisms also produce a range of unwanted effects, including unpleasant odour, stains, allergies, and affect the colour and tensile properties of the fabric [1–2]. Most of the textiles currently used in hospitals and hotels are prone to cross-infection or transmission of diseases caused by micro-organisms, particularly by pathogenic bacteria and fungi [2]. In view of this, prevention of microbial growth has become increasingly crucial and it demands the development of fabrics that possess a desired antimicrobial effect. Antimicrobial finishes also improve the performance and durability of textile products. With the aim to develop antimicrobial textile materials, considerable research has been carried out by making use of organic and inorganic compounds, such as Triclosan, which inhibits the growth of microorganisms using an electrochemical mode of action to penetrate and disrupt their cell walls, quaternary ammonium compounds, biguanides, amines and glucoprotamines that bind microorganisms to their cell membrane and disrupt the structure resulting in the breakdown of the cell. Organic compounds, such as metallic complex compounds based on metals like cadmium, silver, copper and mercury, cause inhibition of the active enzyme centres (inhibition of metabolism) [3–5].

However, many of the commercial antimicrobial agents, such as inorganic salts, organometallics, iodine and iodophors, phenolic compounds, ammonium salt-based compounds, heterocyclic and anionic groups, nitro compounds, urea and related compounds, formaldehyde derivatives and amines, currently available in the market are synthetic and not environment-friendly. Furthermore, a vast majority of these antimicrobial agents are of leaching type, and thus their gradual release from textiles into surroundings results in a decrease of their concentration and gradually falls under the limit of effectiveness, i.e. minimum inhibitory concentration (MIC). The release of these agents also acts as poison to a wide spectrum of bacteria and fungi [6–12].

Natural bioactive agents with antimicrobial properties have become progressively essential for producing non-toxic and environment-friendly textile products. These antimicrobial compounds, which are mostly extracted from plants (aloe vera, tea tree and eucalyptus oil (EO), neem, grapefruit seed, tulsi leaf extracts, etc.), include phenolics and polyphenols (simple phenols, phenolic acids, quinones, flavonoids, flavones, flavonols, tannins and coumarins), terpenoids, essential oils, alkaloids, lectins, polypeptides and polyacetylenes. These components possess not only antimicrobial but also antioxidant properties. Neem (*Azadirachta indica*), one of the richest sources of biologically active compounds, has attracted worldwide attention in recent years owing to its wide range of medicinal properties. [13–17]. It possesses active antimicrobial compounds, such as azadirachtin, salannin and meliantriol, which are also effective in controlling the insect growth and are used as an antifeedant. Researchers have studied the antibacterial activity of various components of the neem tree, such as oil, bark, seed extract, on the 100% cotton fabric and polyester/cotton blend fabric. Some researchers studied the antibacterial properties of neem oil in combination with other herbal oils, such as clove, tulsi and karanga on cotton textiles [11]. In another study, Joshi et al. extracted an antimicrobial agent from the seeds of a neem tree for imparting antibacterial activity to polyester/cotton blend fabric and produced a semi-durable antibacterial finish [16, 18]. The accommodation of a high amount of antimicrobial neem ingredients in the polyester structure resulted in higher antimicrobial activity without significant decrease in crystallinity and tensile properties of polyester fabric. It is concluded from previous studies that neem is effective against bacteria on cotton fabrics and it showed more microbial resistance than aloe vera. The fabric finished with neem is durable even after 15 washes [2]. Aloe vera is another well known bioactive compound used for cosmeo-finishes, antibacterial finishes and skin care agents. [19–21]. It has been reported that the increased concentration of aloe vera gel increases the antibacterial activity against both Gram-positive and

Gram-negative bacteria. It is reported that aloe vera is effectively inhibiting the microbial growth on treated cotton fabrics. Fabrics finished with a combination of aloe vera and neem are also found to be durable. [22]. Tulsi (also known as holy basil) is known for its antimicrobial, insecticidal antiprotozoal, diaphoretic and expectorant properties. The main constituents of tulsi (*Osmium basilicum*) are eugenol (70%), methyl eugenol (20%), carvacrol (3%), etc. In their research, Sathinathan et al. applied tulsi leaf (*Ocimum sanctum*) extracts on cotton fabrics by direct application, i.e. microencapsulation, resin cross-linking methods and their combinations. It is stated that fabrics treated with tulsi extract exhibit excellent antimicrobial activity, and the major component responsible for the antimicrobial properties is eugenol. The fabrics treated with the direct method showed poor durability of the finish compared to microencapsulation and resin cross-linking methods [23]. Grapeseed oil is technically known as *Vitis vinifera* and is used for thousands of years for its medicinal and nutritious properties, such as anti-inflammatory, cardio protective, antimicrobial, antifungal and anti-cancer. The main components responsible for these effects are tocopherol, linolenic acid, resveratrol, quercetin, procyanidins, carotenoids and phytosterols [24]. Several studies reported the antimicrobial activity of grapeseed oil, which is effective against both the spectrum of Gram-negative and Gram-positive bacteria [25–26]. The chemical structures of most of the above mentioned natural bioactive agents are complex and contain mixtures of multiple compounds. Selective isolation and extraction of these bioactive agents are difficult and will increase the quantity of the agent required to obtain a minimum inhibition concentration.

Most of the natural antimicrobial agents are insoluble in water and also adversely affect the physical and other desirable properties of textiles. In order to imbue the bioactive substance, various techniques have been suggested by researchers. Bioactive agents also lose their bioactivity when they react with textile materials. Hence, intensive research was carried out in order to explore and exploit the best utilisation of environment-friendly antimicrobial agents [10–11]. However, it is difficult to achieve the enhanced and durable antimicrobial property of polyester due to semi-crystalline and highly compact structure and the absence of active polar groups in the polyester structure, which facilitates crosslinking of antimicrobial agents, in addition to exhibiting low surface

energy [16–18]. Various functional finishes can easily be imparted to polyester/cotton fabric taking advantage of cotton amorphous structure. Earlier study on polyester/cotton blend using neem antibacterial agent with different crosslinking agents proved that the finished fabric retained the desired antimicrobial activity against Gram-positive bacteria for the maximum of five machine washes, while thereafter the effect is decreased on subsequent washes. [16]. Previous studies suggested many approaches to impart antibacterial activity to the polyester and polyester blended textiles, including coating, spraying, microencapsulation, grafting and insertion of dope additives into the fibre structure [27–30]. An alternative approach to enhance the antibacterial effect of polyester is to open up the compact structure of polyester by making use of suitable interacting solvents, which facilitates the easy entry of antimicrobial agents into the compact polyester structure [31–32]. This paper deals with the antibacterial effect of neem, aloe vera, tulsi, grapeseed oil on polyester and its blends with cotton and viscose, which have been previously treated with the TCAMC solvent system.

## 2 Materials and methods

Polyester (PES), polyester/viscose (PES/CV), polyester/cotton (PES/CO) and texturised polyester (texturised PES) fabric samples of 165 g/m<sup>2</sup> using Oxford weave (derivative of plain weave) are used. The characteristics of the materials are presented in Table 1. The pretreatment of the samples is carried out using various concentration of trichloroacetic acid-methylene chloride (TCAMC) solvent system. Trichloroacetic acid, methylene chloride and acetone were obtained from Sigma-Aldrich Chemical Co. Ltd. It is known that TCAMC interacts with the polyester structure and dissolves out completely at 25% in 5 minutes at room temperature (~30 °C) [31]. It is reported elsewhere that the structural modification of polyester takes place and the compact polymer structure is opened up at a lower concentration of TCAMC treatment without significantly affecting its strength [20]. The effect of different TCAMC concentration on polyester structure has been optimised [32]. The treatment is carried out in a closed trough at ~ 30 °C for 3 minutes. The treated samples are then rinsed with methylene chloride followed by acetone to remove any adhering reagent. Afterwards, the samples are wrung and dried in an open atmospheric

Table 1: Fabric characteristics

Sample no.	Fabric	Mass per unit area (g/m <sup>2</sup> )	Weave	Yarn density (warp × weft)	Linear density (warp × weft)	Tensile strength (warp × weft) (N)	Elongation (warp × weft) (%)	Thickness (mm)
1	100% PES	165	2/2 oxford weave	64 <sup>a)</sup> × 44 <sup>b)</sup>	2/30 <sup>c)</sup> × 2/30 <sup>c)</sup>	1600 × 1400	25 × 20	0.85
2	PES/CV (65/35)	165	2/2 oxford weave	64 <sup>a)</sup> × 44 <sup>b)</sup>	2/30 <sup>c)</sup> × 2/30 <sup>c)</sup>	1600 × 1400	25 × 20	0.85
3	PES/CO (65/35)	165	2/2 oxford weave	64 <sup>a)</sup> × 44 <sup>b)</sup>	2/30 <sup>c)</sup> × 2/30 <sup>c)</sup>	1600 × 1400	25 × 20	1.1
4	100% PES texturised	165	2/2 oxford weave	144 <sup>a)</sup> × 110 <sup>b)</sup>	150 <sup>d)</sup> × 150 <sup>d)</sup>	1400 × 1200	40 × 35	0.8

<sup>a)</sup> ends/cm; <sup>b)</sup> picks/cm; <sup>c)</sup> Ne; <sup>d)</sup> den

Table 2: Experimental design (Taguchi L-16 (4<sup>3</sup> 2<sup>1</sup>) Mixed orthogonal array)

Fabric	Levels		
	Antibacterial agents	Concentration (%)	M:L ratio
PES	Neem	5	01:20
PES/CV	Aloe vera	10	01:25
PES/CO	Tulsi	15	-
Texturised PES	Grapeseed oil	20	-

condition before applying the above mentioned biopolymeric finishes.

Commercially available neem, aloe vera, tulsi and grapeseed oil were chosen for this study. Table 2 shows the experimental design used in the study. Taguchi mixed orthogonal array Design L16 (4<sup>3</sup> 2<sup>1</sup>) was chosen for the experimental plan and the larger the better response (antimicrobial efficiency) was selected to determine the optimum conditions. The TCAMC treated samples were immersed in the solution at 5%, 10%, 15%, 20% concentration of neem, aloe vera, tulsi, grapeseed oil with ethanol at room temperature, resulting in active substances which are subsequently dissolved in ethanol. The treatment is carried out with the help of acetic acid at 80 °C for 20 minutes at different liquor ratio of (1:20 and 1:25). The add-on% is calculated using equation 1.

$$\text{Add-on\%} = (A - B) \times 100/B \quad (1)$$

where A represents a dry weight of biopolymer finished sample and B represents a dry weight of TCAMC treated sample.

The ASTM standard D5035 and Tinus Olsen universal tensile tester are used to test the fabric tensile strength and elongation. Fabric tear strength is tested using tongue tear tester according to the D 2261 standard. The colour values of the finished fabrics are measured using Spectra Scan 5100+ spectrophotometer (RayScan) instrument.

The antibacterial activity of untreated and treated control samples is tested qualitatively by parallel streak method (AATCC-147) and quantitatively by colony count method (AATCC 100) using *S. aureus* (Gram-positive) and *E. Coli* (Gram-negative) bacteria. In the parallel streak method, bacterial suspension in the form of streaks is placed on the plate with the help of a sterilised wire loop. Three streaks are made on the upper side of solid agar plate 1 cm apart from each other; the fabric swatches (5.08 cm × 2.54 cm or 2" × 1") of untreated and treated samples placed on the agar plates are incubated for 24 hours at 37 ± 0.5 °C. After 24 hours of incubation, the swatches are examined for any potential bacterial growth underneath and around the specimen [33].

In the colony count method, the swatches are placed separately in a previously sterilised flask containing Luria broth solution and subjected to  $37 \pm 0.5$  °C for 24 hours in a laboratory shaker for 200 rpm. After 24 hours of incubation, the bacterial suspension is diluted serially (for example  $10^2$ ,  $10^4$ ,  $10^6$  times) using sterilised water. 10  $\mu$ L of diluted bacterial suspension is spread on the plate and incubated again at  $37 \pm 0.5$  °C. After 24 hours of incubation, the agar plates are removed from the incubator to count the bacterial growth inside the plates. The percent reduction in number of colonies in the treated samples as compared to the untreated samples gives the antibacterial activity of the treated samples (equation 2) [34].

$$\text{Antibacterial activity or \% reduction} = (A - B)/B \times 100 \quad (2)$$

where A represents the bacteria colony (CFU/ml) of untreated fabric and B represents the bacterial colony of the treated fabric.

The treated fabrics are then washed in launder-o-meter according to the AATCC Test Method 61-1996 (2A) using Lissapol N, a non-ionic detergent (1% on the weight of fabric) at 40 °C for 60 minutes to check the wash fastness and staining of the treated fabric samples. It is also mentioned that 1 AATCC machine wash is equal to 5 home laundry cycles [35].

### 3 Results and discussion

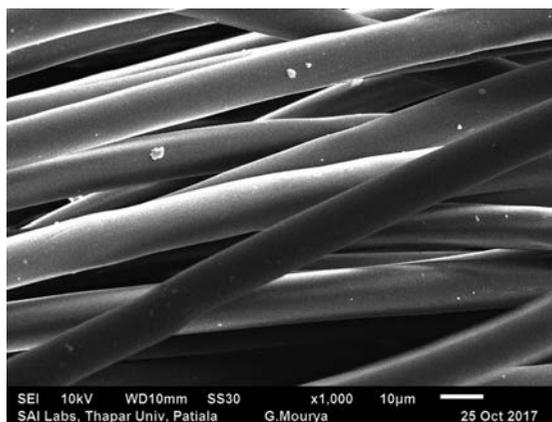
#### 3.1 Effect of biopolymer finish on add-on%

To determine the dry add-on% of the TCAMC-treated finished fabric samples, the samples are kept at 20 °C and 65% RH for 24 hours and the weight

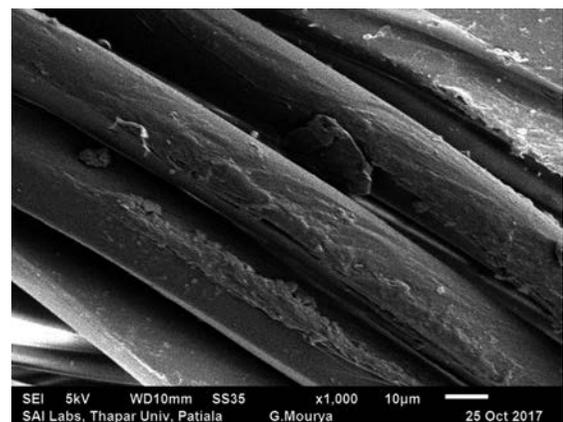
variation calculated according to equation 1. Table 3 shows that polyester fabric has weight reduction after the TCAMC treatment. After applying various finishes, the gain in weight is observed from 1.06% to 2.39%. Similar gain in weight is observed in polyester/viscose, polyester/cotton and texturised TCAMC-treated fabric samples. The scanning electron microscopy (SEM) topography of the TCAMC-treated polyester fibre is shown in Figure 1. It is evident from Figure 1 that the TCAMC induces swelling of polyester fibre and creates several voids and micro cracks.

#### 3.2 Effect of biopolymer finish on tensile and tear properties

In this study, the tensile and tear strength of biopolymer finished polyester and polyester blend fabrics, which have been previously treated with 1% TCAMC for 3 minutes, are tested and evaluated. Table 4 shows the strength loss% of all types of treated fabric samples. All four types of fabrics suffered a reduction in tensile strength ranging from 4.75 to 11.79%. The major loss in strength is due to the interaction of TCAMC reagent. Further the TCAMC-pretreated samples have been treated with biopolymeric finishes. The treatment is carried out using the exhaustion method which is carried out at high temperature. It is obvious from Table 4 that 100% texturised fabric registered a highest strength loss of 11.79%. Perhaps it offered more surface area compared to the surface area of normal polyester and its blends for the TCAMC to interact with the structure. It is evident from Figure 1 that the TCAMC reagent interacted with polyester and disturbed its compact structure, and thus suffered strength loss. In the case of functional textiles, a percentage loss of about 15%



Before TCAMC treatment



After TCAMC treatment

Figure 1: Effect of TCAMC concentration (1%) on polyester

Table 3: Add-on% of different antimicrobial agents on various fabrics

Fabric	Activity	Weight (g)			
		Neem treated	Aloe vera treated	Tulsi treated	Grapeseed oil treated
PES	Before TCAMC treatment	7.76	7.78	7.77	7.73
	After TCAMC treatment	7.62	7.67	7.58	7.52
	Weight loss (%)	<b>1.8</b>	<b>1.41</b>	<b>2.45</b>	<b>2.72</b>
	Finished	7.71	7.76	7.66	7.63
	Add -on (%)	<b>1.18</b>	<b>1.17</b>	<b>1.06</b>	<b>1.46</b>
PES/CV	Before TCAMC treatment	7.85	7.75	7.72	7.7
	After TCAMC treatment	7.59	7.51	7.26	7.54
	Weight loss (%)	<b>3.31</b>	<b>3.1</b>	<b>5.96</b>	<b>2.08</b>
	Finished	7.68	7.57	7.35	7.67
	Add -on (%)	<b>1.19</b>	<b>0.8</b>	<b>1.24</b>	<b>1.72</b>
PES/CO	Before TCAMC treatment	5.51	5.4	5.94	5.58
	After TCAMC treatment	5.34	5.22	5.75	5.33
	Weight loss (%)	<b>3.09</b>	<b>3.33</b>	<b>3.2</b>	<b>4.48</b>
	Finished	5.4	5.27	5.87	5.41
	Add -on (%)	<b>1.12</b>	<b>0.96</b>	<b>2.09</b>	<b>1.5</b>
Texturised PES	Before TCAMC treatment	5.23	5.33	5.51	5.69
	After TCAMC treatment	5.07	5.13	5.22	5.43
	Weight loss (%)	<b>3.06</b>	<b>3.75</b>	<b>5.26</b>	<b>4.57</b>
	Finished	5.13	5.39	5.28	5.56
	Add -on (%)	<b>1.18</b>	<b>1.13</b>	<b>1.15</b>	<b>2.39</b>

is acceptable, while a higher percentage may cause the deterioration of textile structure. The TCAMC and biopolymer finish treatment also influence the tearing strength of fabrics. It is observed from Table 4 that the reduction in tear strength varies from 5.44% to 11.10%, of which 100% texturised polyester showed a maximum reduction of 11.10%.

### 3.3 Effect of biopolymer treatment on antibacterial activity

Antibacterial activity of untreated polyester and TCAMC (1%) treated polyester imbued with neem, aloe vera, tulsi and grapeseed oil are evaluated qualitatively by parallel streak method as stipulated in AATCC 147. Both Gram-positive *S. aureus* and Gram-negative *E. coli* bacteria are used for antibac-

terial assessment. Figure 2 shows the results of antibacterial activity of untreated and neem-imbued TCAMC-treated fabric samples. It is obvious from Figures 2(a) and 2(f)) that a heavy growth of both *S. aureus*, and *E. Coli* bacteria respectively, is seen in the case of untreated polyester, while moderate to high bacterial resistance is observed in TCAMC-treated fabrics Figure 2(b–e) and 2(g–j). It supports the observation published elsewhere that the TCAMC reagent increases the segmental mobility of the polyester polymer and as a result structural rearrangement takes place thereby creating more voids and micro cracks to facilitate the entry of antibacterial agents inside the polymer structure [31]. Similar results are observed in Figures 3–5 for aloe vera, tulsi and grapeseed oil-imbued TCAMC-treated fabric samples.

Table 4: Tensile and tear strength of biopolymer finished fabrics (warp way)

Fabric	Treatment	Tensile strength		Tear strength	
		Mean (N)	Loss (%)	Mean (N)	Loss (%)
100% PES	Control	1600	-	150	-
	Neem	1470	8.13	139	6.96
	Aloe vera	1485	7.19	140	6.93
	Tulsi	1474	7.88	135	9.84
	Grapeseed oil	1510	5.63	142	5.49
PES/CV	Control	1600	-	125	-
	Neem	1495	6.56	116	6.8
	Aloe vera	1502	6.13	114	8.8
	Tulsi	1494	6.63	113	9.15
	Grapeseed oil	1524	4.75	118	5.44
PES/CO	Control	1600	-	125	-
	Neem	1465	8.44	115	7.84
	Aloe vera	1468	8.25	114	8.96
	Tulsi	1460	8.75	115	8.16
	Grapeseed oil	1520	5.00	114	8.8
100% texturised PES	Control	1400	-	100	-
	Neem	1235	11.79	89	10.5
	Aloe vera	1265	9.64	90	9.5
	Tulsi	1250	10.71	89	11.1
	Grapeseed oil	1295	7.5	92	7.6

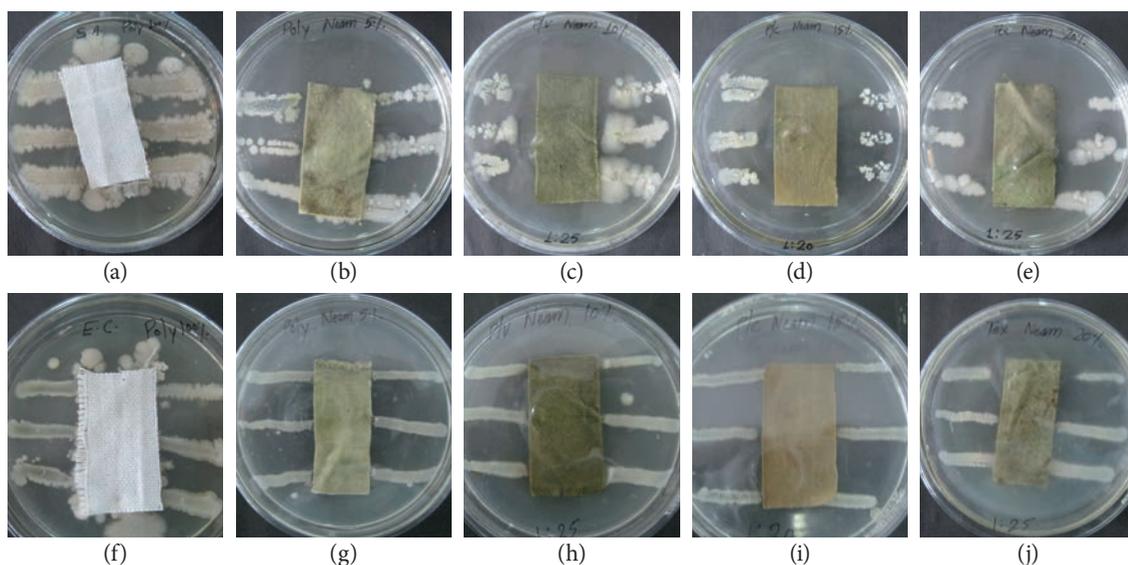


Figure 2: Antimicrobial activity of untreated and neem-imbued TCAMC-treated fabrics: Against *S. aureus* (a–e): (a) untreated 100% PES; (b) TCAMC-treated 100% PES; (c) TCAMC-treated PES/CV; (d) TCAMC-treated PES/CO; and (e) TCAMC-treated texturised PES. Against *E. coli* (f–j): (f) untreated 100% PES; (g) TCAMC-treated 100% PES; (h) TCAMC-treated PES/CV; (i) TCAMC-treated PES/CO; and (j) TCAMC-treated texturised PES

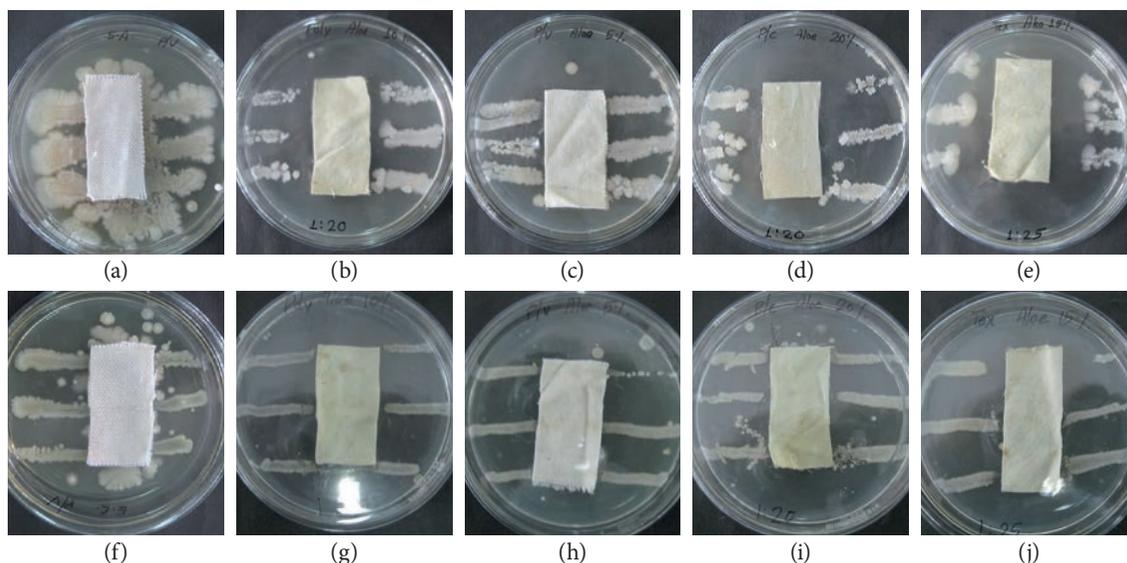


Figure 3: Antibacterial activity on untreated and aloe vera-imbued TCAMC-treated fabrics: Against *S. aureus* (a–e): (a) untreated 100% PES; (b) TCAMC-treated 100% PES; (c) TCAMC-treated PES/CV; (d) TCAMC-treated PES/CO; and (e) TCAMC-treated texturised PES. Against *E. coli* (f–j): (f) untreated 100% PES; (g) TCAMC-treated 100% PES; (h) TCAMC-treated PES/CV; (i) TCAMC-treated PES/CO; and (j) TCAMC-treated texturised PES

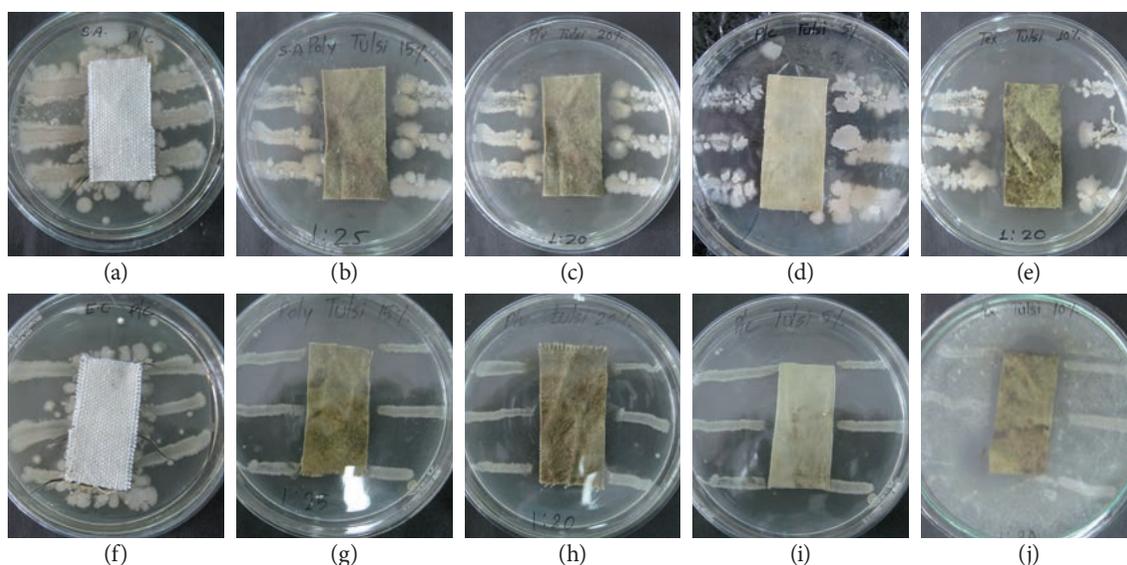


Figure 4: Antibacterial activity on untreated and tulsi-imbued TCAMC-treated fabrics: Against *S. aureus* (a–e): (a) untreated 100% PES; (b) TCAMC-treated 100% PES; (c) TCAMC-treated PES/CV; (d) TCAMC-treated PES/CO; and (e) TCAMC-treated texturised PES. Against *E. coli* (f–j): (f) untreated 100% PES; (g) TCAMC-treated 100% PES; (h) TCAMC-treated PES/CV; (i) TCAMC-treated PES/CO; and (j) TCAMC-treated texturised PES

The antibacterial activity of untreated and TCAMC-treated (with neem, aloe vera, tulsi and grapeseed oil finish) polyester fabrics is assessed quantitatively against both the Gram-positive and Gram-negative bacteria, and the results are presented in Table 5. It

is evident from Table 5 that grapeseed oil-imbued TCAMC-treated 100% polyester shows an 86% and 73% antibacterial activity against both the Gram-positive and Gram-negative bacteria. A similar trend in arresting the growth of bacteria is also evidenced

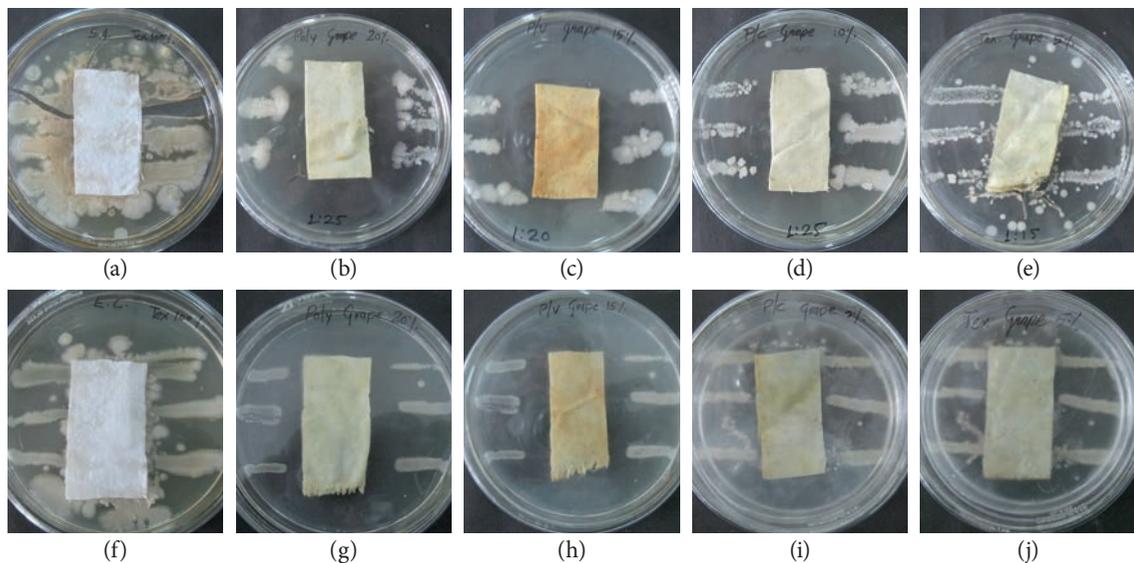


Figure 5: Antibacterial activity on untreated and grapeseed oil-imbued TCAMC-treated fabrics: Against *S. aureus* (a–e): (a) untreated 100% PES; (b) TCAMC-treated 100% PES; (c) TCAMC-treated PES/CV; (d) TCAMC-treated PES/CO; and (e) TCAMC-treated texturised PES. Against *E. coli* (f–j): (f) untreated 100% PES; (g) TCAMC-treated 100% PES; (h) TCAMC-treated PES/CV; (i) TCAMC-treated PES/CO; and (j) TCAMC-treated texturised PES

in other biopolymer-imbued TCAMC-treated fabrics. It should be noted that the TCAMC pretreatment contributed a significant role in enhancing the antibacterial activity of neem, aloe vera, tulsi and grapeseed oil treated polyester. TCAMC modifies the structure of polyester and creates more voids and microcracks in the structure to entrap the antimicrobial agents [16, 22]. It is observed that with the increase in concentration of finishes, the antibacterial activity also increases as evidenced in Figures 2–5, which show bacterial free regions. It is also obvious from

Figures 2–5 that the inhibition zone is influenced by the type of finish and its concentration, which is further reaffirmed by quantitative test results.

### 3.4 Effect of laundering on the antibacterial activity

It is observed that laundering affects the effectiveness of antibacterial finishes and the degree of antibacterial activity is reduced with increase in wash cycles (Table 5). Table 5 shows that all treated fabric samples show modest activity against both *S. aureus* and

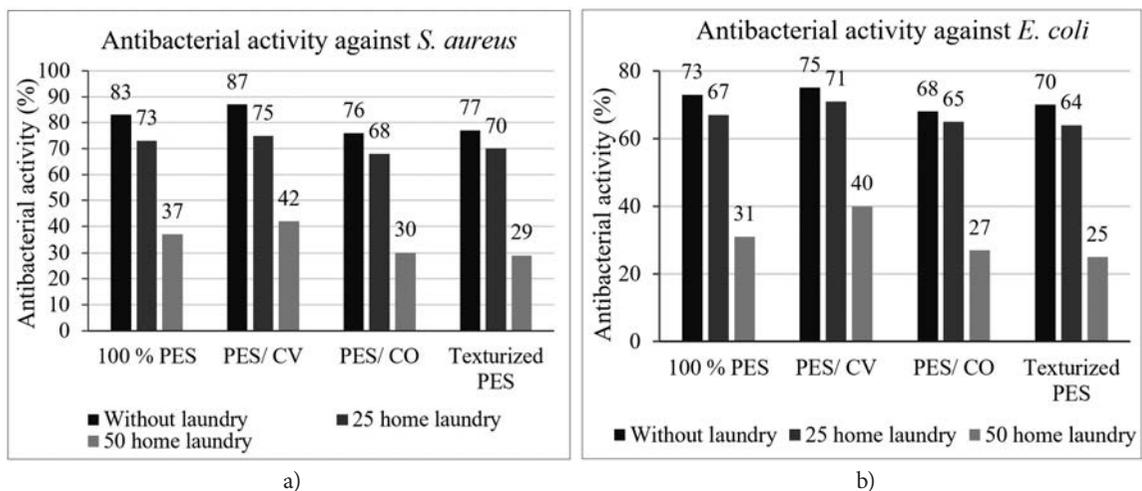


Figure 6: Antimicrobial activity of grapeseed oil-imbued TCAMC-treated fabrics against: a) *S. aureus* and b) *E. coli* after several machine washes

Table 5: Antibacterial activity of treated fabrics after various use-wash cycles (according to Taguchi design)

Fabric	Finish type	Finish concentration (%)	Liquor ratio	Without laundering			
				<i>S. aureus</i> (Gram-positive)		<i>E. coli</i> (Gram-negative)	
				CFU <sup>a)</sup>	AA	CFU <sup>a)</sup>	AA
Untreated	-	-	-	78	-	110	-
100% PES	Neem extract	5	1:20	45	<b>42</b>	65	<b>41</b>
100% PES	Aloe vera gel	10	1:20	42	<b>46</b>	60	<b>45</b>
100% PES	Tulsi extract	15	1:25	37	<b>53</b>	45	<b>59</b>
100% PES	Grapeseed oil	20	1:25	11	<b>86</b>	30	<b>73</b>
PES/CV	Neem extract	10	1:25	46	<b>41</b>	67	<b>39</b>
PES/CV	Aloe vera gel	5	1:25	48	<b>38</b>	69	<b>37</b>
PES/CV	Tulsi extract	20	1:20	36	<b>54</b>	53	<b>52</b>
PES/CV	Grapeseed oil	15	1:20	10	<b>87</b>	25	<b>77</b>
PES/CO	Neem extract	15	1:20	45	<b>42</b>	66	<b>40</b>
PES/CO	Aloe vera gel	20	1:20	44	<b>44</b>	63	<b>43</b>
PES/CO	Tulsi extract	5	1:25	34	<b>56</b>	41	<b>63</b>
PES/CO	Grapeseed oil	10	1:25	12	<b>85</b>	22	<b>80</b>
Texturised PES	Neem extract	20	1:25	35	<b>55</b>	45	<b>53</b>
Texturised PES	Aloe vera gel	15	1:25	31	<b>60</b>	46	<b>58</b>
Texturised PES	Tulsi extract	10	1:20	45	<b>42</b>	66	<b>40</b>
Texturised PES	Grapeseed oil	5	1:20	42	<b>46</b>	62	<b>44</b>

<sup>a)</sup> ml  $\times 10^8$ ; <sup>b)</sup> antibacterial activity (%)

*E. coli* even after 10 AATCC machine washes. The grape seed oil treated 100% PES and PES/CV show 34% and 32 % antibacterial activity after 10 machine washes (50 home laundry cycles) against *S. aureus* and *E. coli* bacteria. This is due to the finish concentration as lower concentration also decreases the effect of antibacterial activity. Figure 6 shows the effect of laundering on antibacterial activity for all kind of polyester fabrics treated with grapeseed oil-imbued TCAMC with 20% grapeseed oil at a liquor ratio of 1:25. It is evident that after 5 and 10 machine laundry cycles the finish is leached out and the antibacterial activity reduced accordingly.

### 3.5 Effect of biopolymer finish on colour properties

Table 6 shows the effect of biopolymeric finishes on the colour properties of all treated samples. It was observed that all fabric samples treated with neem, tulsi and grapeseed oil show a significant change in colour properties. Table 6 shows that the relative colour strength is highly influenced by the finish type and finish concentration. Relative strength increased with higher finish concentration under all conditions. It

was observed that the neem and tulsi treated fabric samples show higher colour strength values. The relative colour strength had the highest value 10994 in case of PES/CV neem. It was also observed that the increase in finish concentration from 5% to 20% leads to higher colour strength values in all cases. From Table 6, the difference in lightness ( $DL^*$ ) has been observed negative, which also confirms the darker shade appearance as compared to the control sample in all cases. The changes in hue values are represented by  $DH^*$  and its tendency towards a specific colour (such as red, yellow or blue) can be explained by the  $Da^*$  and  $Db^*$  values in Table 6.

### 3.6 Analysis and evaluation of experimental results

Analysis of the effect of each control factor fabric type, finish type, concentration and liquor ratio on the antibacterial activity of both *S. aureus* and *E. coli* with signal-to-noise (S/N) response are shown in Table 7. Normally, there are three kinds of quality characteristics in the analysis of the S/N ratio, i.e. lower -the -better, higher-the-better, and nominal-the-best. The S/N ratio is calculated based on

	5-× machine washes				10-× machine washes			
	<i>S. aureus</i> (Gram-positive)		<i>E. coli</i> (Gram-negative)		<i>S. aureus</i> (Gram-positive)		<i>E. coli</i> (Gram-negative)	
	CFU <sup>a)</sup>	AA	CFU <sup>a)</sup>	AA	CFU <sup>a)</sup>	AA	CFU <sup>a)</sup>	AA
	78	-	110	-	78	-	110	-
	48	<b>38</b>	68	<b>38</b>	68	<b>13</b>	98	<b>11</b>
	62	<b>21</b>	88	<b>20</b>	75	<b>4</b>	107	<b>3</b>
	39	<b>50</b>	58	<b>47</b>	64	<b>18</b>	94	<b>15</b>
	18	<b>77</b>	35	<b>68</b>	49	<b>37</b>	73	<b>34</b>
	51	<b>35</b>	73	<b>34</b>	67	<b>14</b>	96	<b>13</b>
	60	<b>23</b>	86	<b>22</b>	69	<b>12</b>	105	<b>5</b>
	38	<b>51</b>	57	<b>48</b>	61	<b>22</b>	90	<b>18</b>
	15	<b>81</b>	26	<b>76</b>	51	<b>35</b>	75	<b>32</b>
	49	<b>37</b>	72	<b>35</b>	61	<b>22</b>	82	<b>25</b>
	58	<b>26</b>	87	<b>21</b>	72	<b>8</b>	99	<b>10</b>
	52	<b>33</b>	76	<b>31</b>	65	<b>17</b>	94	<b>15</b>
	24	<b>69</b>	46	<b>58</b>	60	<b>23</b>	91	<b>17</b>
	46	<b>41</b>	72	<b>35</b>	58	<b>26</b>	84	<b>24</b>
	55	<b>29</b>	81	<b>26</b>	71	<b>9</b>	102	<b>7</b>
	47	<b>40</b>	68	<b>38</b>	63	<b>19</b>	92	<b>16</b>
	46	<b>41</b>	72	<b>35</b>	62	<b>21</b>	90	<b>18</b>

Table 6: Colour strength values and colour coordinates of treated fabric samples

Fabric	Treatment	Conc. (%)	DL*	Da*	Db*	Dc*	dE*	DH*	Strength (%)
100% PES	Standard	-	-	-	-	-	-	-	100
	Neem	5	-37.66	1.6	13.77	13.84	40.13	-0.77	5254
	Aloe vera	10	-10.32	0.9	15.23	15.25	18.42	-0.36	704.75
	Tulsi	15	-31.25	2.05	19.07	19.16	36.67	-0.87	5062.1
	Grapeseed oil	20	-15.54	6.69	31.24	31.85	35.52	-2.49	2221.9
PES/CV	Standard	-	-	-	-	-	-	-	100
	Neem	10	-46.79	0.91	14.51	14.53	49	-0.38	10994
	Aloe vera	5	-6.07	1.93	10.39	10.51	12.18	-1.08	400.06
	Tulsi	20	-47.26	4.66	14.98	15.51	49.8	-2.35	9897.9
	Grapeseed oil	15	-36.89	14.94	23.25	26.89	46.09	6.38	5415.9
PES/CO	Standard	-	-	-	-	-	-	-	100
	Neem	15	-28.58	3.69	16.86	17.17	33.39	-1.76	3136.9
	Aloe vera	20	-17.7	3.38	25.71	25.9	31.39	-1.31	2247.5
	Tulsi	5	-18.14	1.71	17	17.07	24.92	-0.75	1922.9
	Grapeseed oil	10	-16.5	6.88	34.15	34.75	38.54	-2.54	3211.4
100% texturised PES	Standard	-	-	-	-	-	-	-	100
	Neem	20	-39.23	1.67	16.45	16.52	42.57	-0.74	6266.7
	Aloe vera	15	-18.52	3.62	25.11	25.33	31.41	-1.43	1974.9
	Tulsi	10	-30.64	0.32	15.16	15.16	34.19	-0.06	3513.3
	Grapeseed oil	5	-11.75	0.8	22.46	22.48	25.37	-0.22	1427

the S/N analysis. This analysis focuses on finding the best combination among each process parameter for better antibacterial activity. Therefore, higher the better is the quality technique used as shown in Equation 3 given below:

$$\eta = \frac{S}{Ns} = -10 \times \log_{10}\left(\frac{1}{\sum \frac{1}{Y^2}}\right) \quad (3)$$

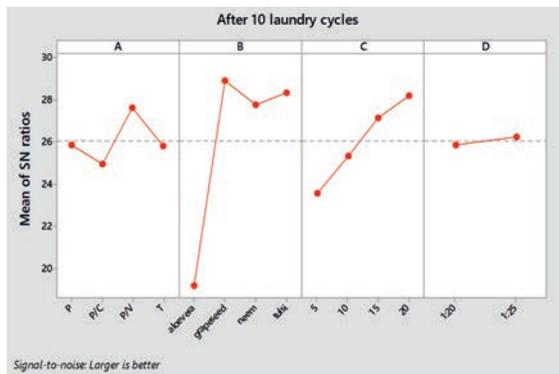
Here, Y represents the observed data in the experiment and n represents the number of observations in the experiment [35–36]. The response tables of S/N for *S. aureus* and *E. coli* are shown in Table 7. This table is made using the Taguchi technique and shows the optimal levels of control factors for the optimal process to enhance antibacterial activity. The level values of control factors for *S. aureus* and *E. coli* given in Table 7 are shown in Figure 7. Optimal process parameters of the control factors for minimising the antibacterial activity of *S. aureus* and *E. coli* can be easily determined from these graphs. The best level for each control factor is found according to the highest S/N ratio in the levels of that control factor. According to this, the levels and S/N ratios for the factors giving the best activity against *S. aureus*

value are specified as factor A (Level 3, S/N = 26.68), factor B (Level 2, S/N = 28.92), factor C (Level 4, S/N = 28.19) and factor D (Level 2, S/N = 25.79). For *E. coli*, values are specified as factor A (Level 3, S/N = 26.39), factor B (Level 2, S/N = 27.75), factor C (Level 4, S/N = 27.36) and factor D (Level 2, S/N = 24.77). In other words, the optimum activity can be achieved for both *S. aureus* and *E. coli* using polyester/viscose fabric with grapeseed oil finish at a 20% concentration at a Liquor ratio of 1:25 respectively.

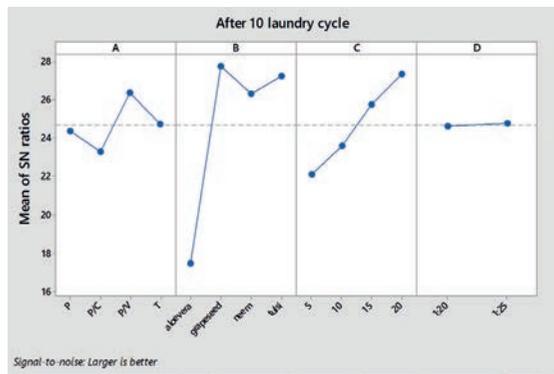
Table 8 shows the ANOVA analysis to study the effect of process parameters after 10 laundry cycles against both *S. aureus* and *E. coli*. The analysis is carried out at 5% significance and at 95% confidence level. The significance of control factors in ANOVA is determined by comparing the F values of each control factor. According to Table 8, the percentage contribution of the fabric, finish, concentration and liquor ratio on the *S. aureus* antibacterial activity is 2.80%, 66.73%, 30.06%, and 0.41 respectively. Thus, the important factors affecting the antibacterial activity are finish type (66.73%) and concertation (30.06%). Similarly, for *E. coli* the effect of laundering on fabric, finish, concentration and liquor ratio against the

Table 7: Response table for Signal to noise ratio for *S. aureus* and *E. coli*

Level	S/N ratio for <i>S. aureus</i>				S/N ratio for <i>E. coli</i>			
	A	B	C	D	A	B	C	D
1	22.89	16.55	23.89	24.38	24.37	17.48	22.09	24.62
2	24.95	<b>28.92</b>	21.10	<b>25.79</b>	23.29	<b>27.75</b>	23.58	<b>24.77</b>
3	<b>26.68</b>	26.50	27.15		<b>26.39</b>	26.32	25.74	
4	25.81	28.37	<b>28.19</b>		24.73	27.22	<b>27.36</b>	
Delta	3.78	12.37	7.08	1.40	3.10	10.27	5.27	0.15



(a)



(b)

Figure 7: Effect of 10 machine laundry cycles on antibacterial activity: a) against *S. aureus*, b) against *E. coli*

Table 8: ANOVA analysis after 10 laundry cycle

Bacteria	Variance source	Degree of freedom (DF)	Sum of squares (SS)	Mean square (MS)	F ratio	Contribution (%)
<i>S. aureus</i>	Fabric	3	136.69	45.56	0.14	2.80
	Finish	3	3280.69	1093.56	3.33	66.73
	Concentration	3	1475.19	491.73	1.50	30.06
<i>E. coli</i>	Fabric	3	60.19	20.063	0.11	1.37
	Finish	3	2953.19	984.40	5.60	69.91
	Concentration	3	1203.69	401.23	2.28	28.46

antibacterial property is shown in Table 8. The percentage contribution of the fabric, finish, concentration and liquor ratio on the *E. coli* antibacterial activity is 1.37%, 69.91%, 28.46%, and 0.26% respectively. Thus, the important factor affecting the antibacterial activity is finish type that has a maximum contribution of 66.73% and 69.91% in both cases. The trends are presented using design generated graphs in Figure 7. It is observed that the grapeseed oil finish shows better antibacterial activity against both *S. aureus* and *E. coli* compared to others finishes after 10 laundry cycles. It has been observed that as the concentration increases the durability of finishes increases. It is also observed from the study that the increase in the liquor ratio from 1:20 to 1:25 increases the antibacterial activity. It is due to the increase in the mobility of the molecule in the solution, which helps in more uniform finishing. Among different types of fabrics, polyester/cotton and polyester/viscose blends show more affinity to adhere to the molecules into the polymer structure as compared to polyester and texturised polyester.

## 4 Conclusion

Different polyester structures are treated with already optimised concentration of TCAMC solvent. The pretreatment modifies the polyester structure and creates more voids and cracks in the compact structure. In this study, the Taguchi method is used to determine optimal process parameters in the antibacterial assessment of the bio polymeric finished pretreated polyester structure. The Taguchi analysis shows that the polyester/viscose fabric with 20% grapeseed finish at a liquor ratio of 1: 25 is the optimum condition for antibacterial activity against both *S. aureus* and *E. coli*. According to the results of statistical analysis,

it is found that the finish type is the most significant parameter with contributions of 66.73% and 69.91%, respectively for *S. aureus* and *E. coli*. Grapeseed oil finish is observed to be the best antimicrobial finish; which shows 21–37% antibacterial activity even after 10 machine laundry cycles against *S. aureus* and 18–34% antibacterial activity after 10 machine laundry cycles against *E. coli*. Also, the neem, tulsi and aloe vera finish show a modest amount of antibacterial activity of about 14–26%, 13–22%, and 5–10% respectively for *S. aureus* after 10 machine laundry cycles. Similarly, the neem, tulsi and aloe vera treated samples show 11–24%, 12–18%, and 4–8% antibacterial activity after 10 machine laundry cycles against *E. coli* respectively.

## References

- ROBINSON, Tim, McMULLAN, Geoff, MARCHANT, Roger, NIGAM, Poonam. Remediation of dyes in textile effluent: a critical review on current treatment technologies with a proposed alternative. *Bioresource Technology*, 2001, 77(3), 247–255, doi:10.1016/S0960-8524(00)00080-8.
- MURUGESH BABU, K, RAVINDRA, K.B. Bioactive antimicrobial agents for finishing of textiles for health care products. *The Journal of The Textile Institute*, 2015, 106(7), 706–717, doi: 10.1080/00405000.2014.936670.
- PURWAR, R., SRIVASTAVA, C.M. Antimicrobial cellulose and cellulose derivative materials. In *Cellulose and cellulose derivatives : synthesis, modification and applications*. Edited by Md. Ibrahim H. Mondal. New York : Nova Science Publishers, 2015, 455–472, <https://novapublishers.com/shop/cellulose-and-cellulose-derivatives-synthesis-modification-and-applications/>

4. PATEL, B.H., DESAI, K.U. Corporate uniform fabrics with antimicrobial edge: preparation and evaluation methodology. *International Dyer*, 2014, **42**(2), 33–38, <https://www.researchgate.net/publication/278764455>.
5. GUGLIUZZA, A., DRIOLI, E. A review on membrane engineering for innovation in wearable fabrics and protective textiles. *Journal of Membrane Science*, 2013, **446**, 350–375, doi:10.1016/j.memsci.2013.07.014.
6. De CARVALHO, C.C. Biofilms: recent developments on an old battle. *Recent Patents on Biotechnology*, 2007, **1**(1), 49–57, doi: 10.2174/187220807779813965.
7. KIM, Y.H., NAM, C.W., CHOI, J.W., JANG, J.H. Durable antimicrobial treatment of cotton fabrics using N-(2-hydroxy) propyl-3-trimethylammonium chitosan chloride and polycarboxylic acids. *Journal of Applied Polymer Science*, 2003, **88**(6), 1567–157, doi: 10.1002/app.11845.
8. EL-TAHLAWY, K.F., EL-BENDARY, M.A., ELHENDAWY, A.G., HUDSON, S.M. The antimicrobial activity of cotton fabrics treated with different crosslinking agents and chitosan. *Carbohydrate Polymers*, 2005, **60**(4), 421–430, doi:10.1016/j.carbpol.2005.02.019.
9. BAGHERZADEH, R., MONTAZER, M., LATIFI, M., SHEIKHZADEH, M., SATTARI, M. Evaluation of comfort properties of polyester knitted spacer fabrics finished with water repellent and antimicrobial agents. *Fibers and Polymers*, 2007, **8**(4), 386–392, doi:10.1007/BF02875827.
10. DASTJERDI, R., MOJTAHEDI, M.R., SHOSHTARI, A.M., KHOSROSHAHI, A. Investigating the production and properties of Ag/TiO<sub>2</sub>/PP antibacterial nanocomposite filament yarns. *The Journal of The Textile Institute*, 2010, **101**(3), 204–213, doi:10.1080/00405000802346388.
11. JOSHI, M., ALI, S.W., PURWAR, R., RAJENDRAN, S. Ecofriendly antimicrobial finishing of textiles using bioactive agents based on natural products. *Indian Journal of Fibre & Textile Research*, 2009, **34**(3), 295–304, <http://nopr.niscair.res.in/handle/123456789/6083>.
12. PURWAR, R., JOSHI, M. Recent developments in antimicrobial finishing of textiles - a review. *AATCC Review*, 2004, **4**(3), 22–26.1
13. COWAN, M.M. Plant products as antimicrobial agents. *Clinical Microbiology Reviews*, 1999, **12**(4), 564–582, doi:10.1128/CMR.12.4.564.
14. QIN, S., XING, K., JIANG, J.H., XU, L.H., LI, W.J. Biodiversity, bioactive natural products and biotechnological potential of plant-associated endophytic actinobacteria. *Applied Microbiology and Biotechnology*, 2011, **89**(3), 457–473, doi:10.1007/s00253-010-2923-6.
15. SUBAPRIYA, R., NAGINI, S. Medicinal properties of neem leaves: a review. *Current Medicinal Chemistry*, 2005, **5**(2), 149–156, doi:10.2174/1568011053174828.
16. JOSHI, M., ALI, S.W., RAJENDRAN, S. Antibacterial finishing of polyester/cotton blend fabrics using neem (*Azadirachta indica*): a natural bioactive agent. *Journal of Applied Polymer Science*, 2007, **106**(2), 793–800, doi:10.1002/app.26323.
17. RAJENDRAN, R., RADHAI, R., BALAKUMAR, C., AHAMED, H.A., VIGNESWARAN, C., VAIDEKI, K. Synthesis and characterization of neem chitosan nanocomposites for development of antimicrobial cotton textiles. *Journal of Engineered Fibers and Fabrics*, 2012, **7**(1), 136–141, doi: 10.1177/155892501200700116.
18. ALI, W., SULTANA, P., JOSHI, M., RAJENDRAN, S. A solvent induced crystallisation method to imbue bioactive ingredients of neem oil into the compact structure of poly (ethylene terephthalate) polyester. *Materials Science and Engineering: C*, 2016, **64**, 399–406, doi:10.1016/j.msec.2016.04.002.
19. FIELDER, J.O., CARMONA, O.G., CARMONA, C.G., LIS, M.J., PLATH, A.M.S., SAMULEWSKI, R.B., BEZERRA, F.M. Application of Aloe vera microcapsules in cotton nonwovens to obtain bio-functional textile. *Journal of The Textile Institute*, 2020, **111**(1), 68–74, doi:10.1080/00405000.2019.1625607.
20. ALI, S.W., PURWAR, R., JOSHI, M., RAJENDRAN, S. Antibacterial properties of Aloe vera gel-finished cotton fabric. *Cellulose*, 2014, **21**(3), 2063–2072, doi: 10.1007/s10570-014-0175-9.
21. SATHIANARAYANAN, M.P., BHAT, N.V., KOKATE, S.S., WALUNJ, V.E. Antibacterial finish for cotton fabric from herbal products. *Indian Journal of Fibre & Textile Research*, 2010, **35**(1), 50–58, <http://nopr.niscair.res.in/handle/123456789/7662>.
22. RAJENDRAN, S., MISHRA, S.P. Chemical, structural and thermal changes in PET caused by solvent induced polymer crystallisation. *Polymers*

- Polymer Composites*, 2007, **15**(2), 103–110, doi: 10.1177/096739110701500203.
23. SIMONČIČ, B., TOMŠIČ, B. Structures of novel antimicrobial agents for textiles—a review. *Textile Research Journal*, 2010, **80**(16), 1721–1737, doi: 10.1177/0040517510363193.
24. SOTO, M.L., FALQUÉ, E., DOMÍNGUEZ, H. Relevance of natural phenolics from grape and derivative products in the formulation of cosmetics. *Cosmetics*, 2015, **2**(3), 259–276, doi: 10.3390/cosmetics2030259.
25. BAYDAR, N.G., ÖZKAN, G., SAĞDIÇ, O. Total phenolic contents and antibacterial activities of grape (*Vitis vinifera L.*) extracts. *Food Control*, 2004, **15**(5), 335–339, doi: 10.1016/S0956-7135(03)00083-5.
26. AL-HABIB, A., AL-SALEH, E., SAFER, A.M., AFZAL, M. Bactericidal effect of grape seed extract on methicillin-resistant *Staphylococcus aureus* (MRSA). *The Journal of Toxicological Sciences*, 2010, **35**(3), 357–364, doi: 10.2131/jts.35.357.
27. MONTAZER, M., AFJEH, M.G. Simultaneous x-linking and antimicrobial finishing of cotton fabric. *Journal of Applied Polymer Science*, 2007, **103**(1), 178–185, doi: 10.1002/app.25059.
28. RAJENDRAN, R., BALAKUMAR, C., SIVAKUMAR, R., AMRUTA, T., DEVAKI, N. Extraction and application of natural silk protein sericin from *Bombyx mori* as antimicrobial finish for cotton fabrics. *The Journal of The Textile Institute*, 2012, **103**(4), 458–462, doi: 10.1080/00405000.2011.586151.
29. DASTJERDI, R., MONTAZER, M., SHAHSAVAN, S. A new method to stabilize nanoparticles on textile surfaces. *Colloids Surfaces A : Physicochemical and Engineering Aspects*, 2009, **345**(1–3), 202–210, doi: 10.1016/j.colsurfa.2009.05.007.
30. RAJENDRAN, S., RAMASAMY, S.S., MISHRA, S.P. Tensile behavior of polyester yarns modified by trichloroacetic acid–methylene chloride treatment. *Journal of Applied Polymer Science*, 1996, **59**(1), 99–108, doi: 10.1002/(SICI)1097-4628(19960103)59:1<99::AID-APP15>3.0.CO;2-3.
31. SIVAKUMARAN, S., RAJENDRAN, S. A quick, room temperature method for polyester-cellulosic blend analysis. *Textile Research Journal*, 1986, **56**(3), 213–214, doi: 10.1177/004051758605600310.
32. 32 ALI, S.W., RAJENDRAN, S., JOSHI, M. Synthesis and characterization of chitosan and silver loaded chitosan nanoparticles for bioactive polyester. *Carbohydrate Polymers*, 2011, **83**(2), 438–446, doi: 10.1016/j.carbpol.2010.08.004.
33. AATCC Test Method 147–1993. Antibacterial activity assessment of textile materials : parallel streak method. Research Triangle Park : AATCC, 1993, 261–262.
34. AATCC Test Method 100–2004. Antibacterial activity assessment of textile materials : percentage reduction method. Research Triangle Park : AATCC, 2004, 149–150.
35. KIVAK, T. Optimization of surface roughness and flank wear using the Taguchi method in milling of Hadfield steel with PVD and CVD coated inserts. *Measurement*, 2014, **50**, 19–28, doi: 10.1016/j.measurement.2013.12.017.
36. ENGIN, A.B., ÖZDEMİR, Ö., TURAN, M., TURAN, A.Z. Color removal from textile dye-bath effluents in a zeolite fixed bed reactor : determination of optimum process conditions using Taguchi method. *Journal of Hazardous Materials*, 2008, **159**(2–3), 348–353, doi: 10.1016/j.jhazmat.2008.02.065.

Md. Reazuddin Repon<sup>1,2</sup>, Nure Alam Siddiquee<sup>1</sup>, Mohammad Abdul Jalil<sup>3</sup>, Daiva Mikučionienė<sup>2</sup>, Md. Rezaul Karim<sup>4</sup>, Tarikul Islam<sup>5</sup>

<sup>1</sup> Khwaja Yunus Ali University, Department of Textile Engineering, Sirajgang-6751, Bangladesh

<sup>2</sup> Kaunas University of Technology, Faculty of Mechanical Engineering and Design, Department of Production Engineering, Kaunas Studentu 56, LT-51424, Kaunas, Lithuania

<sup>3</sup> Khulna University of Engineering and Technology, Department of Textile Engineering, Khulna-9203, Bangladesh

<sup>4</sup> Port City International University, Department of Textile Engineering, Chittagong-4202, Bangladesh

<sup>5</sup> Jashore University of Science and Technology, Department of Textile Engineering, Jashore-7408, Bangladesh

---

## Flame Retardancy Enhancement of Jute Fabric Using Chemical Treatment

*Izboljšanje ognjevarnosti jutne tkanine s kemično obdelavo*

Original scientific article/Izvirni znanstveni članek

Received/Prispelo 9-2020 • Accepted/Sprejeto 10-2020

Corresponding author/Korespondenčni avtor:

**Md. Reazuddin Repon**

E-mail: reazmbstu.te@gmail.com; md.repon@ktu.edu

Phone: +370-66227098

ORCID: 0000-0002-9984-7732

---

### Abstract

This work aims to improve the flame retardancy of jute fabric. Raw and bleached plain weave jute fabric was used in this work. Flame retardants borax, diammonium phosphate and thiourea were applied in different concentrations in a raw and bleached jute fabric with the padding method. The influences of flame retardant finishing on the vertical flammability behaviour and tensile properties as well as wash resistance were investigated. Flame spread time was found to significantly increase when these simple flame retardant finishing agents were used. It was found that the borax-treated raw and bleached specimens exhibited higher flame spread time among all. The assessment of physical properties such as weight gain percentage and breaking load along warp and weft direction of the control and treated fabrics revealed that the increase of flame retardant finishing weight gain caused a decrease in breaking load. Furthermore, the specimens treated with borax and diammonium phosphate flame retardant showed better results than thiourea for flame retardancy and wash durability. These flame retardant jute fabrics have industrial protective textile applications as brattice cloth in mines and many other potential fields of application, e.g. flame retardant kitchen apron, furnishings for public hall, theatre and hospital, etc.

Keywords: flame retardant, jute fabric, finishing, wash durability, breaking load

### Izveček

Namen raziskave je bil izboljšati ognjevarnost jutne tkanine. Surova in beljena jutna tkanina sta bili impregnirani z zaviralci gorenja boraksom, diamonijevim fosfatom in tiosečnino v različnih koncentracijah. Z vertikalnim testom gorljivosti je bil proučevan učinek ognjevarne obdelave na gorljivost, prav tako pa tudi njen vpliv na natezne lastnosti in odpornost pri pranju. Ugotovljeno je bilo, da se je z uporabo teh preprostih ognjevarnih sredstev čas širjenja plamena znatno podaljšal. Najdaljši čas širjenja plamena so imeli surovi in beljeni vzorci, obdelani z boraksom. Ocena fizikalnih lastnosti, kot sta odstotek povečanja mase in pretržna obremenitev v smeri osnove in votka, kontrolne in obdelane tkanine so pokazali, da se je pri povečani količini ognjevarnega sredstva zmanjšala pretržna obremenitev. Pri obdelavi

*z boraksom in diamonijevim fosfatom so bili doseženi boljši rezultati zaviranja gorenja in obstojnosti na pranje kot s tiosečnino. Tako obdelane jutne tkanine so primerne za industrijske tekstilne pregrade v rudnikih in na številnih drugih področjih, kot so ognjevarni kuhinjski predpasniki, ognjevarne dekorativne tekstilije za notranjo opremo javnih prostorov, v gledališčih in bolnišnicah ipd.*

*Ključne besede: zaviralec gorenja, juta, plemenitenje, obstojnost pri pranju*

## 1 Introduction

Jute is a lignocellulosic fibre with a different percentage of hemicellulose (22–24%),  $\alpha$ -cellulose (58–60%) and lignin (12–14%) as the key constituents with other elements as well [1]. Despite jute being one of the most important biodegradable, eco-friendly, anti-static and annually renewable agro products, it is nowadays facing tough competition with synthetic fibres both home and abroad. Therefore, for the competitiveness of this environment friendly fibre, it is necessary to diversify its uses and develop new products for domestic and industrial purposes that can at least partially overcome the present unfavourable situation of jute [2–5]. In South Asia, jute is the most versatile natural fibre gifted

to man by nature. At present, jute can be defined as an eco-friendly natural fibre with multipurpose application prospects ranging from low value geotextiles to high value products, e.g. fancy bags, carpets, home furnishings, composites, papers, particle boards, car components, fashion accessories and gift articles [6]. Apart from its conventional use as packaging material, jute fabrics are now extensively used in furnishing clothes and home textiles as this fibre has good spinnability [7–10]. Due to its versatility, jute is the second most important bast fibre after cotton. However, it ignites easily and is frequently involved in fire. Jute cellulose undergoes decomposition upon ignition, forming highly explosive volatile compounds, mainly laevoglucose with the spread of fire causing injuries and fatalities



a)



b)



c)



d)

Figure 1: Jute: a) plant, b) fibre, c) yarn and d) fabric

in fire accidents [11, 12]. Since consumers are now increasingly becoming aware of a safe lifestyle, expectations and demand for diversified value-added flame retardant jute products are steadily increasing. The flame retardant jute product demand is also on an increase due to its prolonged life cycle and boosted application for home textile and furnishing purposes [13].

Different chemical formulations have been reported for preparing flame retardant jute fabrics. It has been found that different inorganic salts, borax-boric acid composition, hydrated metal oxide like sodium silicate, different phosphorous and nitrogenous compounds and their combinations have been mostly used to make fire-retardant jute [12–16]. Studies also have been reported on the application of nitrogen and sulphur based thiourea on jute fabric, and their flame retardancy properties have been evaluated [17, 18].

This study reports on the application of borax, diammonium phosphate and thiourea for developing flame retardant functionality on a jute fabric (cf. Figure 1). The thermal behaviour in terms of vertical flammability was investigated, and the weight gain and breaking load changes of the jute fabric were reported.

## 2 Experimental

### 2.1 Materials

A raw and bleached plain weave jute fabric commonly known as a hessian cloth with mass per unit area of 241 g/m<sup>2</sup> was fabricated for this research. The fabric construction is shown in Table 1 and its breaking load is stated in Table 2. After the bleaching, fabric mass per unit area became 232 g/m<sup>2</sup>.

Table 1: Raw jute fabric specifications

Fabric structure	Yarn count (tex)		Fabric density (cm <sup>-1</sup> )		Mass per unit area (g/m <sup>2</sup> )
	Ends	Picks	Ends	Picks	
Plain weave	210.54	210.54	6.2 ± 0.2	5.2 ± 0.2	241

Table 2: Jute fabric breaking load

Fabric type	Breaking load (kg)	
	Warp	Weft
Raw jute	32	30
Bleached jute	25	23

### 2.2 Chemicals

Flame retardant finishes such as borax (Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub> × 10 H<sub>2</sub>O, white solid), diammonium phosphate ((NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub>, white powder) and thiourea (CH<sub>4</sub>N<sub>2</sub>S, white solid) were obtained from Tradesia International Pvt. Ltd., Singapore. Other essential chemicals particularly hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>) and sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) were procured from Redox Chemical Industries Ltd., Sri Lanka. All reagents were laboratory grade and were used without further purification.

### 2.3 Methods

#### 2.3.1 Bleaching process

The bleaching was conducted according to the exhaust method using an infrared lab dyeing machine (Xiamen Rapid, China) at 85 °C for 60 min. Then, the bleaching bath was cooled to 40 °C. Samples were washed at room temperature and neutralised with 0.5 g/L acetic acid for 10 min. The specimens were air-dried in a flat dryer machine (Mesdan, Italy). For both the bleaching and neutralising, the material to liquor ratio was 1 : 40. The recipe for bleaching the jute fabric is tabulated in Table 3.

Table 3: Recipe of bleaching of jute fabric

Chemicals/Parameters	Amount
Hydrogen peroxide, 35% (g/L)	1.5
Sodium silicate (g/L)	3
Sodium carbonate (g/L)	3
Wetting agent (g/L)	1
pH	11
Material : liquor	1 : 40
Temperature (°C)	85
Time (min)	60

### 2.3.2 Padding process

All single chemicals with different concentrations were applied to both raw and bleached jute fabrics. The prepared solution was poured into the padding bath of a two-bowl horizontal padding machine (Switzerland Mathis shares technology Ltd., China) and the fabric was impregnated to yield a 95% wet pick up. The pressure was set to 0.3 MPa and the bowl rotation was  $20 \text{ m}^{-1}$  to get the 95% pick up. Each sample was passed two times through the padding bath for better results. The nip pressure was controlled to ensure the required pick-up percentage. The latter can be calculated by using Equation 1. After the padding, the samples were dried using a stenter machine at  $100 \text{ }^\circ\text{C}$  for 2 min.

$$\text{Pick up} = \frac{\text{Wet weight of sample (g)} - \text{Dry weight of sample (g)}}{\text{Dry weight of sample (g)}} \times 100 (\%) \quad (1)$$

## 2.4 Characterisation

### 2.4.1 Weight gain measurement

Weight gain can be determined by using the oven-dry weight method, taking the oven-dry weight of a sample before and after the treatments, expressing the results as a percentage of the initial oven-dry weight of the material taken. Weight gain was calculated with Equation 2.

$$\text{Wet gain} = \frac{W_2 (\text{g}) - W_1 (\text{g})}{W_1 (\text{g})} \times 100 (\%) \quad (2)$$

where  $W_1$  and  $W_2$  indicate the oven-dry weight of the untreated and treated fabric samples, respectively.

### 2.4.2 Tensile strength

Warp and weft breaking strength of untreated and treated jute fabric samples was determined according to the EN ISO 13934-2 standard test method [19] by using a universal strength tester (Titan 3, James Heal, England). The sample size was  $20 \text{ cm} \times 10 \text{ cm}$  for the breaking strength measurement.

### 2.4.3 Vertical flammability test

Raw and chemical-treated bleached jute fabric samples with  $30.48 \text{ cm} \times 7.62 \text{ cm}$  ( $12 \text{ inch} \times 3 \text{ inch}$ ) in size were prepared and placed in a specimen holder for the measurement. Afterwards, the specimens were exposed to a standard flame at  $90^\circ$  angle for  $12 \text{ s} \pm 0.2 \text{ s}$  and left for burning. The flame spread time after the burning was measured in line with the ASTM D6413 standard test method [20].

### 2.4.4 Wash durability of fabrics

The wash durability of the chemical-treated samples (sample size  $30.48 \text{ cm} \times 7.62 \text{ cm}$ ) was tested by washing the samples in a bowl taking 500 ml of water. Each wash cycle was performed for 30 min at  $45 \text{ }^\circ\text{C}$  followed by drying at  $80 \text{ }^\circ\text{C}$  for 10 min to simulate the Samsung auto (WF8500NHS) laundering machine. The flame spread time of fabrics was repeatedly measured after different wash cycles and the average value was reported.

### 2.4.5 Sampling

The samples are identified as stated in Table 3.

Table 3: Sample identification of jute fabric

Fabric type	Flame retardant finish	Amount (%)	Sample identification code
Raw jute fabric	–	–	RR
	Borax	4	RB1
		6	RB2
		8	RB3
		12	RB4
		4	RD1
	Diammonium phosphate (DAP)	6	RD2
		8	RD3
		12	RD4
		4	RU1
	Thiourea	6	RU2
		8	RU3
		12	RU4

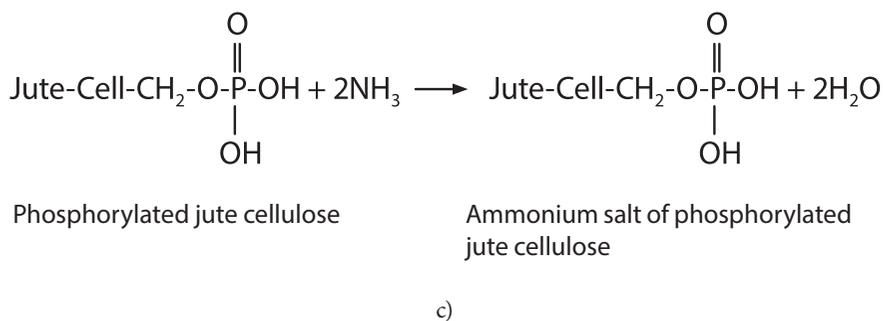
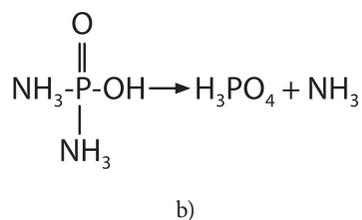
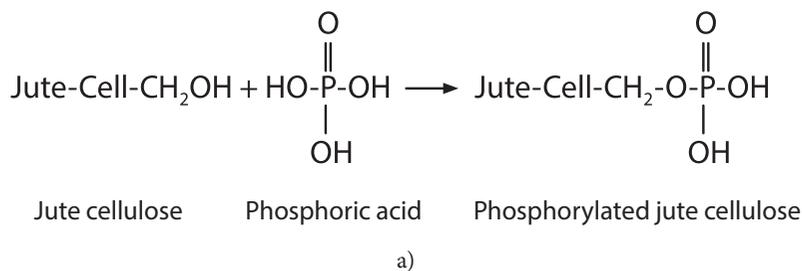
Fabric type	Flame retardant finish	Amount (%)	Sample identification code
Bleached jute fabric	–	–	BR
	Borax	4	BB1
		6	BB2
		8	BB3
		12	BB4
	Diammonium phosphate (DAP)	4	BD1
		6	BD2
		8	BD3
		12	BD4
	Thiourea	4	BU1
		6	BU2
		8	BU3
		12	BU4

#### 2.4.6 Atmospheric conditioning

All physical properties of raw, bleached chemical-treated jute fabrics were evaluated after the conditioning in a standard testing atmosphere, i.e.  $65\% \pm 2\%$  relative humidity (RH) and  $27\text{ }^\circ\text{C} \pm 2\text{ }^\circ\text{C}$  temperature for 48 hours [21].

#### 2.4.7 Probable reactions mechanism

$\text{H}_3\text{PO}_4$  reacts with the hydroxyl groups in jute fibres at high temperature. The following reactions illustrate the postulated mechanism leading to the configuration of phosphorylated jute.



### 3 Results and discussion

#### 3.1 Weight gain percentage

Raw and bleached jute fabrics were subjected to a treatment with three different flame retardant formulations under specific treatment conditions and the weight gain percentage of the treated fabrics was evaluated. The results of the weight gain percentage are reported in Figures 2 and 3.

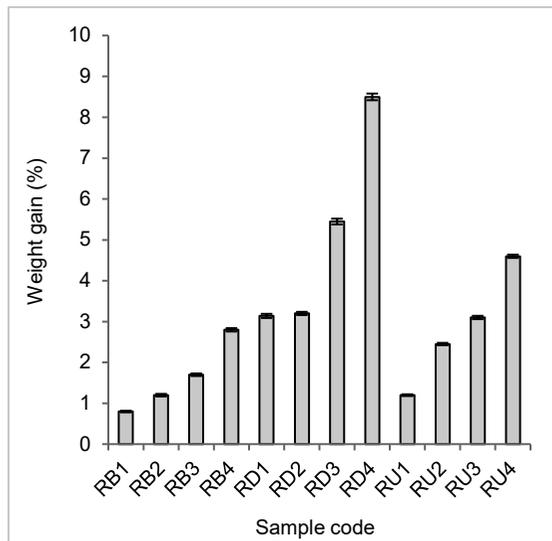


Figure 2: Weight gain changes of raw jute fabric during application of different concentrations of flame retardant finish

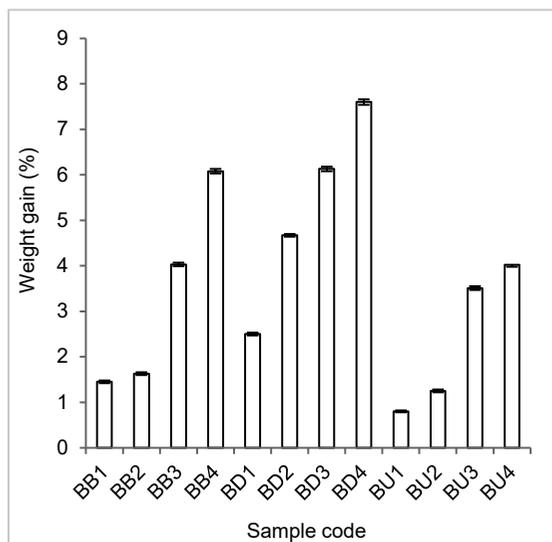


Figure 3: Weight gain changes of bleached jute fabric during application of different concentrations of flame retardant finish

From Figures 2 and 3, it can be observed that with increased loading percentage of chemicals in all formulations, the weight gain gradually increased in jute fabric as well. The DAP-treated samples showed higher weight gain compared to the borax- and thiourea-treated samples. Both raw and bleached jute fabrics showed similar behaviour after the treatment with flame retardant chemicals. A probable reason for this strength gain may lie in the formation of additional binder film on the substrate.

Regarding raw jute fabrics, the sample order of weight gain was  $RB4 > RB3 > RB2 > RB1$  after being treated with borax in a different amount of chemical loading,  $RD4 > RD3 > RD2 > RD1$  for the samples treated with DAP and  $RU4 > RU3 > RU2 > RU1$  for the samples treated with thiourea (cf. Figure 2). Due to the 2%, 6%, 8% and 12% borax concentration treatment of the raw jute fabric, the weight gain increased by 0.08%, 1.20%, 1.70% and 2.80% for the samples RB1, RB2, RB3 and RB4. The weight gain showed a 3.14%, 3.20%, 5.45% and 8.50% increase for the samples RD1, RD2, RD3 and RD4 compared to the raw jute fabric. The flame spread time increased by 1.20%, 2.45%, 3.10% and 4.60% for the samples RU1, RU2, RU3 and RU4 compared to the raw jute fabric. The same results were recorded for RB2 and RU1, despite the fact that RB2 was treated with 6% and RU1 with 2% chemical concentration (cf. Figure 2).

Regarding the bleached jute fabrics, the weight gain of samples followed in the order  $BB1 < BB2 < BB3 < BB4$  after being treated with borax in a different amount of chemical loading,  $BD1 < BD2 < BD3 < BD4$  for the samples treated with DAP and  $BU1 < BU2 < BU3 < BU4$  for the samples treated with thiourea (cf. Figure 3). Due to the 2%, 6%, 8% and 12% borax concentration treatment of bleached jute fabric, the weight gain was by 1.45%, 1.63%, 4.03% and 6.08% higher for the samples BB1, BB2, BB3 and BB4. The weight gain was by 2.50%, 4.67%, 6.13% and 7.60% higher for the samples BD1, BD2, BD3 and BD4 compared to the bleached jute fabric. The weight gain showed a 0.80, 1.25%, 3.51% and 4.02% increase for the samples BU1, BU2, BU3 and BU4 compared to the bleached jute fabric (cf. Figure 3). The same results were observed for RB1 and BU1 at 2% chemical concentration, RB1 being the sample of raw jute fabric treated with borax and BU1 the sample of bleached jute fabric treated with thiourea (cf. Figures 2 and 3). At 2% chemical concentration, the weight gain was by 0.08%, 3.14% and 1.20% higher for the samples RB1, RD1 and RU1 compared to the raw jute fabric,

and by 1.45%, 2.50% and 0.80 higher for the samples BB1, BD1 and BU1 compared to the bleached jute fabric (cf. Figures 2 and 3). At 6% chemical concentration, the flame spread time showed a 1.20%, 3.20% and 2.45% increase for the samples RB2, RD2 and RU2 compared to the raw jute fabric, and a 1.63%, 4.67% and 1.25% increase for the samples BB2, BD2 and BU2 compared to the bleached jute fabric (cf. Figures 2 and 3). At 8% chemical concentration, the flame spread time grew by 1.70%, 5.45% and 3.10% for the samples RB3, RD3 and RU3 if compared to the raw jute fabric, and by 4.03%, 6.13% and 3.51% for the samples BB3, BD3 and BU3 if compared to the bleached jute fabric (cf. Figures 2 and 3). At 12% chemical concentration, the flame spread time increased by 2.80%, 8.50% and 4.60% for the samples RB4, RD4 and RU4 if compared to the raw jute fabric, and by 6.08%, 7.60% and 4.02% for the samples BB4, BD4 and BU4 compared to the bleached jute fabric (cf. Figures 2 and 3). Finally, it was concluded that the weight gain percentage of raw and bleached jute fabrics increased together with increased chemical concentration. The highest value was observed at DAP treated samples.

### 3.2 Flammability properties

The flame spread times of raw and bleached jute fabric were tested after using a different flame retardant finish in different concentrations. The results are presented in Figures 4 and 5.

It was found that the raw jute fabric without the treatment with a flame retardant finish catches fire and burns continuously within 18 s, while the bleached jute fabric catches fire and burns continuously within 21 s. It became evident that the flame spread time of the jute fabric increased due to the bleaching and treatment with a flame retardant finish. Figures 4 and 5 demonstrate that the flame spread time gradually increased in all cases with increased chemical concentration. The borax- and DAP-treated fabrics showed higher flame spread time compared to those treated with thiourea. Both raw and bleached jute fabrics showed similar behaviour after being treated with flame retardant chemicals.

Several chemical reactions are responsible for the flame retardancy effect of the jute fabric treated with a different formulation [7]. The effect of borax on the improvement of the flame retardancy of jute can be explained with the insulating layer theory. Borax acts by the endothermic release of water and melts to form a sodium borate coating. Chemically, borax

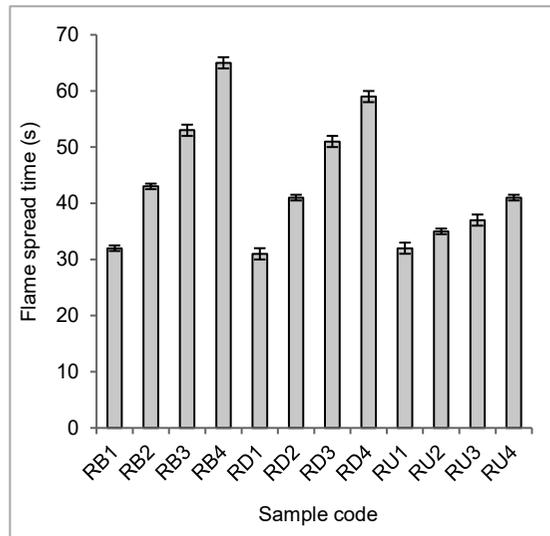


Figure 4: Flame spread time changes of raw jute fabric during application of different concentrations of flame retardant finish

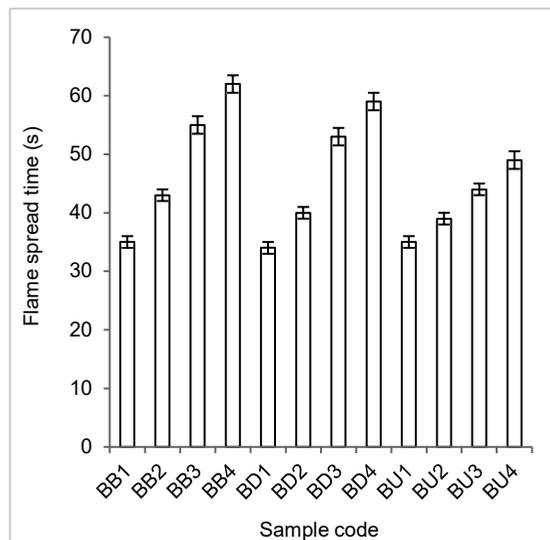


Figure 5: Flame spread time changes of bleached jute fabric during application of different concentrations of flame retardant finish

can also react with the primary hydroxyl group of cellulose polymers to give a borate ester and block the release of flammable gases. In the case of the DAP-treated jute fabric, at high temperature, DAP forms a phosphoric acid, which causes the material to char, forming a thick glassy layer of carbon. This carbonated char stops the decomposition process (pyrolysis) and prevents the release of flammable gases, essentially cutting off fuel to the flame. It also

provides a barrier between the material and the heat source. In the case of thiourea-treated jute fabric, at high temperatures, it enables the formation of stable molecular compounds that stop the decomposition process (pyrolysis) and prevent the release of flammable gases. It also releases inert nitrogen gases that inhibit the chain reaction leading to combustion.

Regarding raw jute fabrics, the sample order was RB4 > RB3 > RB2 > RB1 after the treatment with borax in a different amount of chemical loading, RD4 > RD3 > RD2 > RD1 for the samples treated with DAP and RU4 > RU3 > RU2 > RU1 for the samples treated with thiourea (cf. Figure 4). Due to the 2%, 6%, 8% and 12% borax concentration treatment of the raw jute fabric, the flame spread time showed a 77.78%, 138.89%, 194.44% and 261.11% increase for the samples RB1, RB2, RB3 and RB4. The flame spread time was by 72.22%, 127.78%, 183.33% and 227.78% higher for the samples RD1, RD2, RD3 and RD4 if compared to the raw jute fabric. The flame spread time increased by 77.78%, 94.44%, 105.56% and 127.78% for the samples RU1, RU2, RU3 and RU4 in comparison with the raw jute fabric. The same results were observed for RB1 and RU1 at 2% concentration of chemicals. On the other hand, the same results were recorded for RD2 and RU4 even though RD2 was treated at 6% concentration of chemicals, and RU4 at 12% concentration of chemicals (cf. Figure 4).

Regarding bleached jute fabrics, the sample order was BB1 < BB2 < BB3 < BB4 after the treatment with borax in a different amount of chemical loading, BD1 < BD2 < BD3 < BD4 for the samples treated with DAP and BU1 < BU2 < BU3 < BU4 for the samples treated with thiourea (cf. Figure 5). After the bleached jute fabric being treated with 2%, 6%, 8% and 12% borax, the flame spread time was by 66.67%, 104.76%, 161.90% and 195.24% higher for the samples BB1, BB2, BB3 and BB4. The flame spread time showed a 61.90%, 90.48%, 152.38% and 180.95% increase for the samples BD1, BD2, BD3 and BD4 if compared to the bleached jute fabric. The flame spread time increased by 66.67%, 85.71%, 109.52% and 133.33% for the samples BU1, BU2, BU3 and BU4 compared to the bleached jute fabric. The same results were observed for BB1 and BU1 at 2% concentration of chemicals (cf. Figure 5).

At 2% concentration of chemicals, the flame spread time was by 77.78%, 72.22% and 77.78% higher for the samples RB1, RD1 and RU1 in comparison with the raw jute fabric, and by 104.76%, 90.48% and 85.71% higher for the samples BB1, BD1 and BU1 if compared

to the bleached jute fabric (cf. Figures 4 and 5). At 6% concentration of chemicals, the flame spread time showed a 138.89%, 127.78% and 94.44% increase for the samples RB2, RD2 and RU2 when compared to the raw jute fabric, and a 66.67%, 61.90% and 66.67% increase for the samples BB2, BD2 and BU2 when compared to the bleached jute fabric (cf. Figures 4 and 5). At 8% chemical concentration, the flame spread time rose by 194.44%, 183.33% and 105.56% for the samples RB3, RD3 and RU3 if compared to the raw jute fabric, and by 161.90%, 152.38% and 109.52% for the samples BB3, BD3 and BU3 if compared to the bleached jute fabric (cf. Figures 4 and 5). At 12% chemical concentration, the flame spread time showed a 261.11%, 227.78% and 127.78% increase for the samples RB4, RD4 and RU4 if compared to the raw jute fabric, and a 195.24%, 180.95% and 133.33% increase for the samples BB4, BD4 and BU4 if compared to the bleached jute fabric (cf. Figures 4 and 5). The flame retardancy of the jute fabric improved after the flame retardant finishing agent was applied. The concentrations of chemicals also had a beneficial effect on flame retardancy. The raw and bleached jute fabrics were shown to improve their flame retardant behaviour.

### 3.3 Breaking load

Raw and bleached jute fabrics were subjected to a treatment with three different flame retardant formulations under specific treatment conditions, and the changes of the breaking load of the treated samples were evaluated. The results of breaking load both in warp and weft direction are reported in Figures 6 and 7.

Figures 6 and 7 show that both warp and weft direction breaking load values gradually decreased with the increase of loading percentage of chemicals in all formulations. It may be presumed that due to a mild acidic hydrolysis of jute cellulose, the breaking load falls.

Regarding raw jute fabrics, the sample order of breaking load was RB1 > RB2 > RB3 > RB4 after being treated with borax in a different amount of chemical loading, RD1 > RD2 > RD3 > RD4 for the samples treated with DAP and RU1 > RU2 > RU3 > RU4 for the samples treated with thiourea in warp direction. A similar scenario was observed in weft direction (cf. Figure 6). After the raw jute fabric being treated with 2%, 6%, 8% and 12% borax, the breaking load was by 3.12%, 6.25%, 10.93% and 12.50% lower for the samples RB1, RB2, RB3 and RB4 in warp

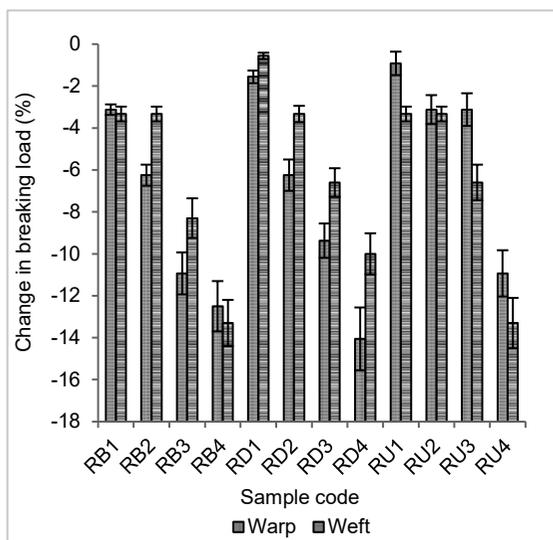


Figure 6: Breaking load changes of raw jute fabric during application of different concentrations of flame retardant finish

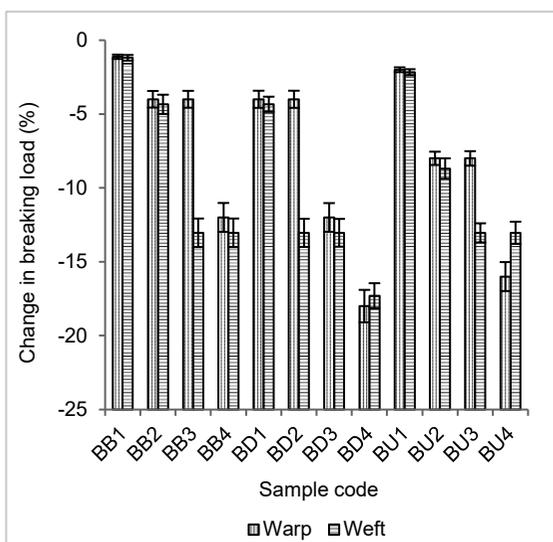


Figure 7: Breaking load changes of bleached jute fabric during application of different concentrations of flame retardant finish

direction, and by 3.33%, 3.33%, 8.30% and 13.30% lower in weft direction. The breaking load showed a 1.56%, 6.25%, 9.37% and 14.06% decrease for the samples RD1, RD2, RD3 and RD4 in warp direction, and a 0.56%, 3.33%, 6.60% and 10.0% decrease in weft direction if compared to the raw jute fabric. The breaking load fell by 0.92%, 3.12%, 3.12% and 10.93% for the samples RU1, RU2, RU3 and RU4 in warp direction, and by 3.33%, 3.33%, 6.60% and

13.30% in weft direction when compared to the raw jute fabric. Under the same amount of breaking load, a 6.25% decrease in warp direction was recorded for RB2 and RD2, a 3.33% decrease for RB1, RB2, RD2, RU1 and RU2, a 6.60% decrease for RD3 and RU3, and a 13.30% decrease for RB4 and RU4 in weft direction (cf. Figure 6).

Regarding bleached jute fabrics, the sample order of breaking load was  $RB1 > RB2 = RB3 > RB4$  after being treated with borax in a different amount of chemical loading,  $RD1 = RD2 > RD3 > RD4$  for the samples treated with DAP and  $RU1 > RU2 = RU3 > RU4$  for the samples treated with thiourea in warp direction. A regular decreasing scenario was observed in weft direction for all samples (cf. Figure 7). The breaking load was by 1.120%, 4.0%, 4.0% and 12.0% lower for the samples BB1, BB2, BB3 and BB4 in warp direction, and by 1.19%, 3.34%, 13.04% and 13.04% lower in weft direction after treating the bleached jute fabric with 2%, 6%, 8% and 12% concentration borax (cf. Figure 7). The breaking load showed a 4.0%, 4.0%, 12.0% and 16.0% fall for the samples BD1, BD2, BD3 and BD4 in warp direction, and a 4.34%, 13.04%, 13.04% and 17.30% fall in weft direction compared to the bleached jute fabric (cf. Figure 7). The breaking load decreased by 2.0%, 8.0%, 8.0% and 16.0% for the samples BU1, BU2, BU3 and BU4 in warp direction, and by 2.17%, 8.69%, 13.04% and 13.04% in weft direction if compared to the bleached jute fabric (cf. Figure 7). Under the same amount of breaking load, there was a 4.0% decrease recorded for BB2, BB3, BD1 and BD2, and an 8.0% decrease for BU2 and BU3 in warp direction, and a 13.04% decrease for BB3, BB4, BD2, BD3, BU3 and BU4 in weft direction (cf. Figure 7). At 2% concentration of chemicals, the breaking load showed a 3.12%, 1.56% and 0.92% fall for the samples RB1, RD1 and RU1 in warp direction, and a 3.33%, 0.56% and 3.33 fall for the same samples in weft direction when compared to the raw jute fabric, and a 1.12%, 4.0% and 2.0% fall for the samples BB1, BD1 and BU1 in warp direction, and a 1.19%, 4.34% and 2.17% fall for the same samples in weft direction in comparison with the bleached jute fabric (cf. Figures 6 and 7). At 6% chemical concentration, the breaking load showed a 6.25%, 6.25% and 3.12% increase for the samples RB2, RD2 and RU2 in warp direction, and a 3.33%, 3.33% and 3.33 decrease for the same samples in weft direction if compared to the raw jute fabric. The breaking load was by 4.0%, 4.0% and 8.0% lower for the samples BB2, BD2 and BU2 in warp direction, and by 4.34%, 13.04% and 8.89% lower

for the same samples in weft direction compared to the bleached jute fabric (cf. Figures 6 and 7). At 8% chemical concentration, the breaking load was by 10.93%, 9.37% and 3.12% lower for the samples RB3, RD3 and RU3 in warp direction, and by 8.30%, 6.60% and 6.60% lower for the same samples in weft direction if compared to the raw jute fabric. For the samples BB3, BD3 and BU3, it was lower by 4.0%, 12.0% and 8.0% in warp direction, and by 13.04%, 13.04% and 13.04% in weft direction in comparison with the bleached jute fabric (cf. Figures 6 and 7). At 12% chemical concentration, the breaking load showed a 12.50%, 14.06% and 10.93% fall for the samples RB4, RD4 and RU4 in warp direction, and a 13.30%, 10.0% and 13.30% fall for the same samples in weft direction if compared to the raw jute fabric. It was by 12.0%, 18.0% and 16.0% lower for the samples BB4, BD4 and BU4 in warp direction, and by 13.04%, 17.30% and 13.04% for the same samples in weft direction compared to the bleached jute fabric (cf. Figures 6 and 7). A lower breaking load in weft direction is responsible for a lower density of picks compared to the density of ends.

### 3.4 Wash durability

The washability of the chemical-treated samples after several wash cycles (1–5) was evaluated and the results are shown in Figure 8.

After wash cycle 1, the treated samples experienced a slight decrease in flame spread time compared to the unwashed samples. This was probably due to

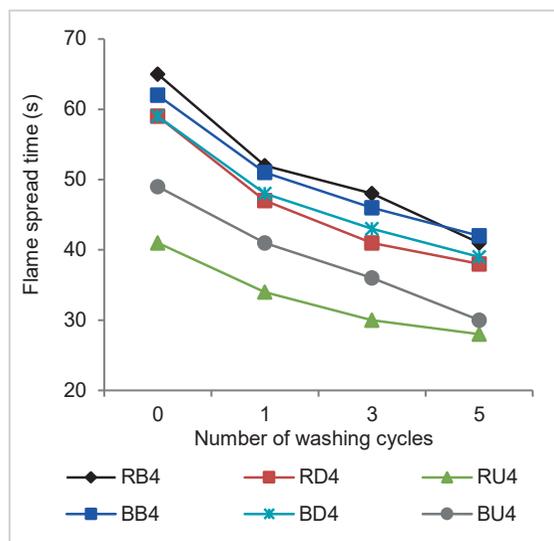


Figure 8: Effect of wash performance on flame spread time of jute fabric treated with flame retardant finish

the removal of unfixed chemical substances from the fabric surface after the washing. Furthermore, increasing the number of wash cycles to 3 and 5 resulted in a decrease of flame spread time. The latter can be attributed to the surface damage of coated layers caused by rigorous stirring during the washing [1]. Nevertheless, a moderate decrease in the flame spread time pattern was observed after the wash cycle 5, which is comparable to the raw and chemical-bleached unwashed jute samples, suggesting that the chemical-treated jute samples have moderate wash durability.

## 4 Conclusion

This study describes the added value of a jute fabric by imparting flame retardancy by using borax, diammonium phosphate and thiourea. It was found that the flame spread time of a jute fabric significantly improved through the application of these simple flame retardant agents. The weight gain percentage increased with the increase of concentration of chemicals whereas the breaking load decreased. It was also established that the specimens treated with borax and diammonium phosphate flame retardant showed better results than those treated with thiourea. Since borax, diammonium phosphate and thiourea are abundantly available and the application process is simple, the scope of applying these flame retardant finishes to impart flame retardancy in jute fabrics is appealing, better wash durability further reinforcing the use in wearable textile applications.

### Declaration of conflict of interest

The authors declared no potential conflicts of interest concerning the research, authorship and/or publication of this article.

### Funding

The authors received no financial support for the research, authorship and/or publication of this article.

## References

- SAMANTA, A. K., BHATTACHARYA, R., CHOWDHURY, R. Fire retardant chemical finishing of jute fabric using sulfamate and urea mixture. *AASCIT Journal of Materials*, 2015, 1(4), 98–110, <http://article.aascit.org/file/pdf/9740743.pdf>.

2. BASAK, S., SAMANTA, K. K., CHATTO-PADHYAY, S. K., DAS, S., NARKAR, R., DSOUZA, C., SHAIKH, A. H. Flame retardant and antimicrobial jute textile using sodium metasilicate nonahydrate. *Polish Journal of Chemical Technology*, 2014, **16**(2), 106–113, doi: 10.2478/pjct-2014-0039.
3. JALIL, M. A., SINHA, R. C., MAHABUBUZ-ZAMAN, A. K. M., HOSSAIN, M. M., IDRIS, M. A. Study on physical and structural properties of jute-PALF blended yarn spun by apron draft spinning. *Research Journal of Textile and Apparel*, 2015, **19**(3), 9–15, doi: 10.1108/RJTA-19-03-2015-B002.
4. MASRANI, S., MCKIERNAN, P. Accounting as a legitimising device in voluntary price agreements: The Dundee jute industry, 1945–1960. *Critical Perspectives on Accounting*, 2011, **22**(4), 415–433, doi: 10.1016/j.cpa.2011.02.001.
5. CHAUDHURI, S., PRAHALAD, K. K. C., VATHSALA, S. Patterns of diversification in larger indian enterprises. *Vikalpa*, 1982, **7**(1), 23–40.
6. JALIL, M. A., AFROZ, F. M., HOSSAIN, A., & SIDDIKA, A. Y. E. S. H. A. Analysis of physio-mechanical properties of jute-PALF Union fabrics. *International Journal of Mechanical Engineering*, 2015, **4**(3), 23–28, <http://oaji.net/articles/2015/1877-1434624316.pdf>
7. SAMANTA, A. K., BHATTACHARYA, K. Simultaneous dyeing and fire-retardant finishing of jute fabric using an acid dye and selective FR finishing chemicals. *Textiles and Light Industrial Science and Technology*, 2015, **4**(1), 1–16, doi: 10.12783/tlist.2015.0401.01.
8. BASU, G., ROY, A. N. Blending of jute with different natural fibres. *Journal of Natural Fibers*, 2008, **4**(4), 13–29.
9. MIAZI, M. O. G., MAHABUBUZZAMAN, A. K. M., MANIRUZZAMAN, M., JALIL, M. A., SHAHID, M. A. Effect of twist on productivity of fine jute yarn produced by modified ring spinning machine. *Journal of Innovation, Development and Strategy*, 2009, **3**(4), 14–16, <http://ggfjournals.com/assets/uploads/14-16.pdf>.
10. *Tribology of natural fiber polymer composites*. Edited by Navin Chand and Mohammed Fahim. Cambridge : Woodhead Publishing, 2008, 1–58.
11. KHANH, V. T. H., HUONG, N. T. Influence of crosslinking agent on the effectiveness of flame retardant treatment for cotton fabric. *Industria Textila*, 2019, **70**(5), 413–420, doi: 10.35530/IT.070.05.1610.
12. BASAK, S., SAMANTA, K. K., CHATTO-PADHYAY, S. K., NARKAR, R., BHOWMICK, M., DAS, S., SAIKH, A.H. Fire retardant and mosquito repellent jute fabric treated with thio-urea. *Journal of Textile Association*, 2014, **74**(5), 273–282.
13. SAMANTA, A. K., BISWAS, S. K., BAGCHI, A., BHATTACHARJEE, R. Semi-durable fire retardant finishing of jute fabric and its thermal behaviour. *Journal of the Institution of Engineers (India), Part TX: Textile Engineering Division*, 2011, **91**(1), 18–26, [https://shodhganga.inflibnet.ac.in/bitstream/10603/162359/16/16\\_publication.pdf](https://shodhganga.inflibnet.ac.in/bitstream/10603/162359/16/16_publication.pdf).
14. NAGIEB, Z. A., NASSAR, M. A., EL-MELIGY, M. G. Effect of addition of boric acid and borax on fire-retardant and mechanical properties of urea formaldehyde saw dust composites. *International Journal of Carbohydrate Chemistry*, 2011, 1–6, doi: 10.1155/2011/146763.
15. BASAK, S., CHATTOPADHYAY, S. K., SAMANTA, K. K., DAS, S., NARKAR, R. Fire retardant jute fabric using water glass. *Man-Made Textiles in India*, 2013, **41**(11), 386–390.
16. BASAK, R.K., SAHA, S.G., SARKAR, A.K., SAHA, M., DAS, N.N., MUKHERJEE, A.K. Thermal properties of jute constituents and flame retardant jute fabrics. *Textile Research Journal*, 1993, **63**(11), 658–666, doi: 10.1177/004051759306301107.
17. SAMANTA, A. K., SINGHEE, D., BASU, G., MAHALANABIS, K. K. Hydrogen peroxide and potassium per-oxo-disulphate combined room temperature bleaching of jute, cotton and jute-cotton union fabrics - an energy-efficient ecofriendly process. *Indian Journal of Fibre and Textile Research*, 2007, **32**(2), 221–231.
18. Wakelyn, P.J. Environmentally friendly flame resistant textiles. In *Advances in fire retardant materials*. Edited by A.R. Horrocks and D. Price. Cambridge : Woodhead Publishing, 2008, pp. 188–212, doi: 10.1533/9781845694701.1.188.
19. ISO 13934-2. Textiles - Tensile properties of fabrics. Part 2 : Determination of maximum force using the grab method. Geneva : ISO Copyright Office, 2014, 1-9.
20. D6413/D6413M-15 Standard test method for flame resistance of textiles (vertical test). West Conshohocken : ASTM International, 2015, doi: 10.1520/D6413\_D6413M-15.
21. BOOTH, J.E. *Principle of textile testing*. London : Heywood Books, 1968, p. 101.

# SHORT INSTRUCTIONS FOR AUTHORS OF SCIENTIFIC ARTICLES

## Scientific articles categories:

- **Original scientific article** is the first publication of original research results in such a form that the research can be repeated and conclusions verified. Scientific information must be demonstrated in such a way that the results are obtained with the same accuracy or within the limits of experimental errors as stated by the author, and that the accuracy of analyses the results are based on can be verified. An original scientific article is designed according to the IMRAD scheme (Introduction, Methods, Results and Discussion) for experimental research or in a descriptive way for descriptive scientific fields, where observations are given in a simple chronological order.
- **Review article** presents an overview of most recent works in a specific field with the purpose of summarizing, analysing, evaluating or synthesizing information that has already been published. This type of article brings new syntheses, new ideas and theories, and even new scientific examples. No scheme is prescribed for review article.
- **Short scientific article** is original scientific article where some elements of the IMRAD scheme have been omitted. It is a short report about finished original scientific work or work which is still in progress. Letters to the editor of scientific journals and short scientific notes are included in this category as well.

**Language:** The manuscript of submitted articles should be written in UK English and it is the authors responsibility to ensure the quality of the language.

**Manuscript length:** The manuscript should not exceed 30,000 characters without spacing.

**Article submission:** The texts should be submitted only in their electronic form in the format \*.doc (or \*.docx) and in the format \*.pdf (made in the computer program Adobe Acrobat) to the address: [tekstilec@a.ntf.uni-lj.si](mailto:tekstilec@a.ntf.uni-lj.si). The name of the document should contain the date (year-month-day) and the surname of the corresponding author, e.g. 20140125Novak.docx. The articles proposed for a review need to have their figures and tables included

in the text. The article can also be submitted through a cloud-based file transfer service, e.g. "WeTransfer" ([www.wetransfer.com](http://www.wetransfer.com)).

**Publication requirements:** All submitted articles are professionally, terminologically and editorially reviewed in accordance with the general professional and journalistic standards of the journal Tekstilec. Articles are reviewed by one or more reviewers and are accepted for publication on the basis of a positive review. If reviewers are not unanimous, the editorial board decides on further proceedings. The authors can propose to the editorial board the names of reviewers, whereas the editorial board then accepts or rejects the proposal. The reviewers' comments are sent to authors for them to complete and correct their manuscripts. The author is held fully responsible for the content of their work. Before the author sends their work for publication, they need to settle the issue on the content publication in line with the rules of the business or institution, respectively, they work at. When submitting the article, the authors have to fill in and sign the Copyright Statement ([www.tekstilec.si](http://www.tekstilec.si)), and send a copy to the editors by e-mail. They should keep the original for their own personal reference. The author commits themselves in the Copyright Statement that the manuscript they are submitting for publication in Tekstilec was not sent to any other journal for publication. When the work is going to be published depends on whether the manuscript meets the publication requirements and on the time reference the author is going to return the required changes or corrections to the editors.

**Copyright corrections:** The editors are going to send computer printouts for proofreading and correcting. It is the author's responsibility to proofread the article and send corrections as soon as possible. However, no greater changes or amendments to the text are allowed at this point.

**Colour print:** Colour print is performed only when this is necessary from the viewpoint of information comprehension, and upon agreement with the author and the editorial board.

**More information on:** [www.tekstilec.si](http://www.tekstilec.si)

