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Engineering Power

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EDITOR-IN-CHIEF'S WORD

Dear Readers,

The Croatian Academy of Engineering remains dedicated to bringing you the latest insights through Engineering Power, largely due to the remarkable efforts of our editor, Prof. Bruno Zelić.

This issue spotlights cutting-edge research in textile technology, featuring papers from the University of Zagreb on topics like ultrasonic welds, hospital textile treatments, polyester microplastics, and genetically engineered fibers. Modern textile tech is crucial for sustainable fashion, as emphasized by the European Technology Platform's strategic programs.

Additionally, we're excited to share a brief report on the Academy's recent 30th anniversary celebration.

Editor-in-Chief
Vedran Mornar, President of the Croatian Academy of Engineering

EDITOR'S WORD

Dear readers,

I am pleased to introduce the Engineering Power issue, edited by Prof. Tanja Pušić, Ph.D. Four original scientific publications offer findings from research undertaken at the Faculty of Textile Engineering of the University of Zagreb. The fifth article provides a brief overview of the activities of the European Technology Platform for the Future of Textiles and Clothing (Textile ETP). This edition concludes with a brief recap of the Academy's regular annual assembly, which commemorated HATZ's 30th anniversary, as well as the list of HATZ awards recipients in 2022.

I hope you like reading it!

Editor
Bruno Zelić, Vice-President of the Croatian Academy of Engineering

FOREWORD

Dear readers,

Three original scientific papers, one scientific review and one report were selected for this issue of Engineering power. All papers are thematically related to the Research Strategy of the University of Zagreb Faculty of Textile Technology, for the period 2021-2027, which has development potential and is part of the strategic programmes of Europe Technology Platform.

The first paper presents the contribution of a group of innovators in the field of ultrasonic technology, discussing functional relationships between the fracture forces of ultrasonic welds and the speed as a function of the electrical power of the ultrasonic generator. The authors presented 43 parameters categorised into three main groups: polymer material parameters, acoustic parameters and technological parameters. The second paper was prepared by a group of authors as a contribution to the project HrZZ-UIP-2017-05-8780, HPROTEX in the field of functionalization of hospital textiles. The influence of pretreatment of cotton fabrics blended with polyester with sodium hydroxide on chitosan functionalisation was studied. The effects of functionalization and durability in three washing cycles were evaluated using physico-chemical methods, physico-mechanical methods and antimicrobial activity. The third paper is related to the project HrZZ-IP-2020-02-7575, InWaShed-MP, which includes research on polyester textiles and the problem of microplastics in washing wastewaters. Polyester knitted fabrics and wastewater from innovative and standard washing processes analysis is based on the surface properties of the fabric and the composition of the wastewater, with a focus on the content of particles released from the knitted fabric during ten washing cycles. The fourth paper is a scientific review of genetically engineered/modified fibres for 21st century textiles and fashion, providing an overview of the achievements in this field and their use in the context of contemporary thinking and sustainable textiles and fashion. According to the author's concluding remarks, the era of biotextiles represents a new epoch that is being realized not only through genetic design but also through synthetic biology.

Finally, the European Technology Platform for the Future of Textiles and Clothing (Textile ETP) was presented as the largest European network for textile research and innovation. The ETP has launched 7 strategic programs for the period between 2020 and 2030: 1. circular economy; 2. bio-based fibres; 3. sustainable chemistry; 4. smart textiles; 5. high-performance technical textiles; 6. digital textile production; 7. digital EU fashion production.

Guest-Editor
Tanja Pušić, University of Zagreb Faculty of Textile Technology

CONTENT

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Functional relationships between the breaking forces of ultrasonic welds and the speed in relation to the electrical power of the ultrasonic generator

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Abstract

Welding thermoplastic polymer materials with ultrasonic welding machines (UWM) and an ultrasonic rotary sonotrode (URS) is a high-tech, long-term process welding technology with fast welding speed, high weld breaking forces, low energy consumption, and simplicity of operation. Many writers have concentrated on a few characteristics, primarily the welding duration, welding speed, and ultrasonic generator power. This study present s43 parameters grouped into three categories : polymer material parameters, acoustic parameters and technological parameters. Functional correlations are provided between ultrasonic weld breaking forces and the speed at which specimens are welded utilizing a rotary ultrasonic sonotrode in relation to the electrical power of the ultrasonic generator

Keywords: *process parameter, welding, ultrasonic, rotary sonotrode*

1. Introduction

Thermoplastic polymer ultrasonic welding is becoming increasingly common. It is characterized by comparatively low energy consumption per welded joint, welding speed, high weld breaking force, ease of use, and environmental friendliness. Airtight and watertight ultrasonic welds can also be approved. Since weld tightness is a critical property, ultrasonic technology is most commonly used for long welds in continuous mode in the manufacture of face masks, protective clothing, disposable hospital gowns, filters, aerospace, sails, marine, automotive and other applications.

The three parameters most frequently discussed in scientific papers are the power of the ultrasonic generator for the URS, the compression force of the URS on the material, and the welding duration.

Many scientists list power welding mode, amplitude, pressure, and URS or anvil gap as welding parameters. E. Sancaktr specifies amplitude, welding speed, power and frequency parameters [1]. Z. Kiss et al. specify amplitude, compression force, frequency and welding time [2]. A. K. Makawana and V. R. Patel specify amplitude, welding pressure, and welding duration as the most important process parameters [3]. W. Shi and T. Little specify welding duration, power, speed, amplitude, and compression force as process parameters [4]. O. Atalay et al. give only strength and compressive force as process parameters [5]. V. N. Kmelev et al [6] provide the greatest overview of the process parameters for the plunge technique, particularly in terms of the thermal characteristics of the materials.

2. Ultrasonic welding parameters

Tab.1 displays the ultrasonic welding parameters of thermoplastic polymer materials. They are thermoplastic polymer material properties, acoustic parameters, and technical parameters that are governed by the UWM.

3. Mathematical correlation of parameters

A counter roller is located at the bottom of the welder and has a specified density of the material from which it is manufactured as well as the speed of sound waves in it. In the URS, ultrasonic vibrations of high intensity are generated to introduce heat into the thermoplastic polymer material. Since reflection occurs at each discontinuity of acoustic impedances encountered by an ultrasonic wave, an ultrasonic wave of intensity is also partially reflected at the discontinuity (URS/material) with an intensity, while a larger part penetrates the material with an intensity of (I_1). Equation (1), proposed by E. Dieulasaint and D. Royer [16], may be used to represent the intensity of the ultrasonic oscillations (I0) of the URS, i.e., the average power of the ultrasonic wave:

$$I_0=2\cdot\pi^2\cdot f^2\cdot A_0^2\cdot Z_0 \qquad (1)$$

where is:

f - frequency of ultrasonic vibrations of the sonotrode [Hz]

A_0 - sonotrode vibration amplitude [m]

Z_0 - acoustic impedance of the ultrasonic rotary sonotrode [kg/m^{2}s]

The ultrasonic power (P_α) developed in the thermoplastic polymer material is calculated by the product of the intensity of the ultrasonic waves converted into the heat of the polymer material (I_α) and the effective area of the URS, which consists of the width (w_s) and length (l_s) of the URS imprint on the polymer material:

$$P_\alpha=I_\alpha\cdot w_s\cdot l_s \qquad (2)$$

Tab. 1. The parameters of thermoplastic polymer ultrasonic welding

Ultrasonic welding parameters

Polymer material parameters

1. intensity of ultrasonic oscillations
2. reflection of ultrasound pressures coefficient
3. ultrasound intensity absorption coefficient
4. ultrasound intensity reflection coefficient between the material to be welded and the counter roller
5. ultrasound intensity reflection coefficient between the URS and the material to be welded
6. ultrasound intensity absorption coefficient on the discontinuity of URS/material
7. ultrasound intensity absorption coefficient on the material/counter roller discontinuity
8. functional dependence of the decrease in ultrasound pressure in the material to be welded depending on the thickness of the material
9. ultrasound pressure
10. functional dependence of the decrease in ultrasound intensity in the material with thickness of the material
11. coefficient of reflection of ultrasound intensities on the discontinuity

Acoustic parameters

1. intensity of ultrasonic oscillations
2. coefficient of reflection of ultrasound pressures
3. ultrasound intensity absorption coefficient
4. ultrasound intensity reflection coefficient between the material to be welded and the counter roller
5. ultrasound intensity reflection coefficient between the URS and the material to be welded
6. ultrasound intensity absorption coefficient on the discontinuity of the URS/material
7. ultrasound intensity absorption coefficient on the material/counter roller discontinuity
8. functional dependence of the decrease in ultrasound pressure in the material to be welded depending on the thickness of the material
9. ultrasound pressure
10. functional dependence of the decrease in ultrasound intensity in the material with thickness of the material
11. coefficient of reflection of ultrasound intensities on the discontinuity

Technological parameters

1. frequency of ultrasonic vibrations of the URS
2. specified electrical power of the UWM generator
3. electrical power of the UWM generator
4. effective power of the URS
5. force with which URS act on two layers of thermosplastic polymer materials with total thickness
6. specific density of URS material
7. speed of ultrasound propagation in the URS
8. URS vibration amplitude
9. acoustic impedance of the URS
10. specific material density from which the counter-roller is made
11. speed of ultrasound propagation of the backing material for welding
12. acoustic impedance of the backing material for welding
13. URS ultrasound intensity
14. radius of the URS
15. width of the URS
16. length of the imprint of the URS on the material
17. angular velocity of the rotation of the URS
18. linear speed of the edge of the URS
19. ultrasonic welding time depending on the ratio of the length of the imprint of the URS
20. linear speed of the sonotrode edge
21. delay time of the start of welding due to heating the beginning of the welded joint

When equation (1) is supplemented with the reflection and absorption coefficient, the resulting equation (3) is:

$$P_\alpha=2\cdot\pi^2\cdot f^2A_0^2\cdot Z_0\cdot w_s\cdot l_s\cdot(1-R_1)\cdot(1-e^{-4\cdot\mu_A\cdot d}+R\cdot e^{-4\cdot\mu_A\cdot d}-R\cdot e^{-8\cdot\mu_A\cdot d}) \quad (3)$$

R_1 – ultrasound intensity reflection coefficient between the URS and the polymeric material to be welded

μ_A – acoustic damping factor of the material [m^{-1}]

d – material thickness [mm]

R – coefficient of reflection of ultrasound intensities on the discontinuity

When ultrasonic energy is delivered into a thermoplastic polymeric material for the first time, the temperature begins to rise as the energy is used to raise the temperature of the material from its starting temperature to its melting point. The density of the material (ρ), the thickness of the two layers of material with individual material thickness d, the width (w_s) and length (l_s) of the URS imprint on the polymer material, the specific heat of the material (c), and the difference between the melting temperature and the initial temperature are the factors that determine the energy required to reach the melting temperature of the material [7].

The following is the mathematical equation for heating the material from room temperature to melting temperature:

$$Q_H=2\cdot\rho\cdot d\cdot w_s\cdot l_s\cdot c\cdot(T_2-T_1) \quad (4)$$

The latent melting heat of fusion of the welded polymer material (Q_L) is equal to the product of the density of the material (ρ), the thickness of the two layers of material with individual thickness (d), the width (w_s) and length (l_s) of the URS on the polymer material, and the latent heat of the material (L):

$$Q_L=2\cdot\rho\cdot d\cdot w_s\cdot l_s\cdot L \quad (5)$$

The total heat (Q_T) required for welding polymer materials is the sum of specific heating and latent heat of fusion

$$Q_T=2\cdot\rho\cdot w_s\cdot l_s\cdot[c\cdot(T_2-T_1)+L] \quad (6)$$

where is:

T_1 - ambient temperature and melting temperature

T_2 - melting temperature

The necessary welding time (t) for applying ultrasound to thermoplastic polymer materials is provided by equation (7).

Even after removing some action factors, equation (7) retains great accuracy in terms of practical applicability. The intricate structure of Equation (7) indicates that numerous factors influence the welding of polymeric materials. Ultrasonic welding of polymer materials involves complex interdependencies between technical and technological parameters, which are completely explained and revealed by the equation above and all of its derivatives [7].

Based on experience and experiments, it was found that the most important parameters of the comprehensive model are the specified power of the ultrasonic generator and welding times, while other parameters are required to achieve a certain accuracy of the acoustic mathematical model of ultrasonic welding time.

$$t=\frac{\rho\cdot[c\cdot(T_2-T_1)+L]}{\pi^2\cdot f^2A_0^2\cdot Z_0\cdot(1-R_1)\cdot(1-e^{-4\cdot\mu_A\cdot d}+R\cdot e^{-4\cdot\mu_A\cdot d}-R\cdot e^{-8\cdot\mu_A\cdot d})} \quad (7)$$

4. Experimental and results

The tests were performed using the Pfaff UWM, model 8310 [17] with URS for welding thermoplastic polymer materials, shown in Fig. 1. An InfiniVisions MSO-X 3024A oscilloscope from Agilent Technologies [18] and an MTI-2100 photon sensor [19] were also used for the measurements. The MTI-2100 photon sensor, a dual-channel optical measurement system that performs non-contact measurements of displacements and vibrations, was used in this work. Displacements ranging from 0.25 nm to 5.08 mm can be measured at frequencies from DC to over 150 kHz. The frequency response ranges from DC to 100 kHz. The typical normal sensitivity of the probes is of the order of 0.025 μm/mV [7].

The properties of the PVC material used are: thickness d = 20 μm; specific heat (at T_1 = 289 K and T_2 = 485 K) c = $1\cdot10^3$ J/mm^{3}K; specific melting heat L=$163\cdot10^3$ J/kg; specific density $\rho_0 = 1.4\cdot10^3$ kg/m^3. PVC foil was used to create 15x50 mm measuring specimens. The aforementioned UWM, which has an adjustable electrical power of the ultrasonic generator of 50 – 100 % , or converted to a power of 200 to 400 W, was used to weld the test specimens. Five welding speeds were also used, including 0.077 m/s, 0.097 m/s, 0.125 m/s, 0.146 m/s, and 0.227 m/s.

The acoustic impedance of the sonotrode $Z_0 = 4.1\cdot10^7$ kg/m^2 s, the acoustic impedance of the material to be welded $Z_1 = 3.2 \cdot10^6$ kg/m^{2}s, and the acoustic damping factor of the material $\mu_A = 0.37$ m^{-1}.

The ultrasound intensity reflection coefficient between the material to be welded and the counter roller and the ultrasound intensity reflection coefficient between the URS and the material to be welded amount to 0.73.

The maximum breaking forces (F_p) of the test specimens for the highest welding speeds (0.077 m/s, 0.097 m/s, 0.125 m/s, 0.146 m/s, and 0.227 m/s) are important for validating the mathematical model. For each maximum, the vibration amplitude must be read based on the specified declared power (D_p) of the ultrasonic generator [7]. Based on the data acquired, functional correlations were established between ultrasonic weld breaking forces and the stated claimed power of the ultrasonic generator, as well as the speed at which the specimens were welded with the URS (Fig. 2).

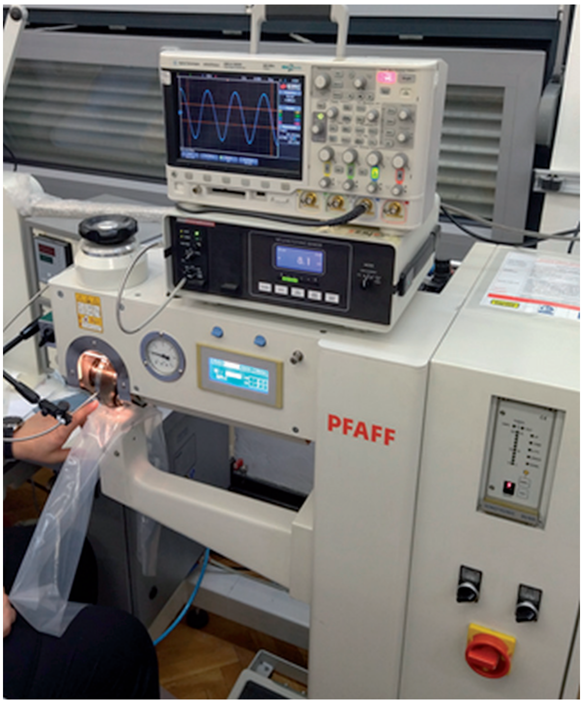

Fig. 1. Measuring set-up on the UWM

As the applied ultrasonic energy increases, the material melts more and the weld has a larger breaking force. The breaking force of ultrasonic welds first increases as the claimed power of the ultrasonic generator increases. The breaking force can be raised until it reaches its limit, at which point the injected energy becomes too high, resulting in excessive melting of the polymer material, damage to the weld, and a sudden decline in breaking force.

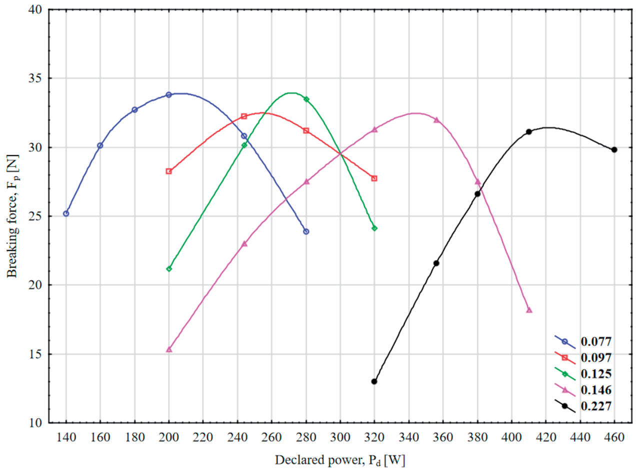

Fig. 2. Functional relationships between the breaking forces of ultrasonic welds and the speed in relation to the electrical declared power of the ultrasonic generator

Fig. 2 shows that the maximum breaking force (F_p) for the polymer material used is between 32 and 34 N for different powers of the ultrasonic generator and welding speeds [7].

5. Conclusion

This study discusses the use of ultrasonic welding. The article presents and discusses the delicate interweaving of technological and technical elements of ultrasonic welding of polymer materials. The optimal welding parameters and typical changes in the functional dependency of breaking forces on the ultrasonic energy introduced into the weld have been determined. The results show that the applied ultrasonic energy resulted in low breaking forces, that there were optimum ranges of applied ultrasonic energy that resulted in maximum joint breaking forces, and that there were ranges with too much applied ultrasonic energy. As a result, the welds deteriorated. The newly established mathematical model and the experimental results agreed well, with minor variations for practical applications.

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Anita Tarbuk, Sandra Flinčec Grgac, Tihana Dekanić, Ivana Čorak, Stefana Begović

The Influence of Cotton/Polyester Blend Fabric Pre-treatment on Chitosan Functionalization

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Abstract

In this work, the influence of cotton/polyester blend fabric pretreatment on chitosan functionalization was investigated. For this purpose, a cotton/polyester blend fabric in twill weave was pretreated with 1.5 M NaOH and then functionalized with chitosan. Three wash cycles were performed to determine the durability. The zeta potential was measured with an electrokinetic analyzer EKA (Anton Paar) before and after functionalization, as well as after the first and third wash cycles, so that the presence of chitosan could be determined. The changes in mechanical properties - thread density, change in mass per unit area, tensile strength and elongation, spectral properties - whiteness, yellowing index and deviation from the white standard, moisture management and antimicrobial efficacy were determined according to standardized methods. Alkali pretreatment was discovered to enhance the amount of accessible hydroxyl groups of the cotton component and carboxyl groups of the polyester component in the blend, as well as the bonding of chitosan and hence wash resistance. Chitosan improves the blend's strength and antibacterial efficiency.

***Keywords**: cotton-polyester blend, chitosan, zeta potential, moisture management.*

1. Introduction

Cotton fiber is a cellulose fiber with excellent absorbency and comfort. Natural impurities such as pectin, waxes, organic acids, proteins, and minerals are eliminated during the scouring process, but colored compounds - pigments - remain. Chemical bleaching of pigments results in fiber whiteness, although cellulose oxidative damage might occur, resulting in reduced fiber strength. Therefore, alkali treatment, i.e. mercerization process can be done to obtain better strength and brightness, and even higher absorption ability [1-4]. Another possibility is blending with synthetic fibers, i.e. polyester or polyamide for that purpose. Polyester fibers, on the other hand, have exceptional strength, resistance to chemicals, light and microorganisms, dimensional stability and fast drying, but due to high degree of crystallinity (65-85%) it has low moisture absorption and no swelling. As a result, the surface of polyester fibers can be changed using hydrolysis (alkali, enzyme), aminolysis, or plasma treatment, or combined with natural fibers such as cotton or wool [5, 6].

Since modification or treatment results in change of the number of surface-active groups of fiber, i.e. blocking and/or adding, their dissociation results in different thickness and distribution of the electric double, resulting in a change in fabric interface phenomena [1, 2, 7]. The change in the interface phenomenon between cotton and polyester fibers in monofilament fabrics has been studied in detail, but the dependence on the structure of the fabric as well as its alkaline modification in the blend was first investigated in the HRZZ project UIP-2017-05-8780 [8-12].

Today, there is an increased need for fabrics that, in addition to offering comfort, also have protective features, and given the recent COVID epidemic, antimicrobial effectiveness is particularly in demand. In addition to commercially available antimicrobial agents, natural active ingredients based on plant extracts, essential oils and animal origin are increasingly being employed in the functionalization of textiles [13].

Over the last decade, researchers' attention has shifted to the use of chitosan in textile processing, namely to offer an antibacterial impact and limit the release of textile particles into water during the washing process [10-12, 14]. Chitosan is a linear polysaccharide of natural origin obtained by partial alkaline N-deacetylation of chitin (over 50%). It consists of an acetylated part (N-acetyl-D-glucosamine) and a deacetylated part (beta-(1,4)-D-glucosamine) [15, 16]. Due to its antimicrobial activity, nontoxicity, biocompatibility, and biodegradability, it is a valued agent that is increasingly used in various fields. Its main applications are in medicine for antibacterial wound dressings, in drug delivery systems and to boost immune defenses [15-22]. Chitosan is most commonly used in the finishing of wool, cotton and polyester fibers and achieves antimicrobial activity against various bacteria and fungi [23-25]. The antimicrobial properties of chitosan result from its polycationic nature and depend on the degree of deacetylation, molecular weight and pH [26]. Positively charged amino groups of chitosan can bond to negatively charged bacterial surfaces, resulting in protein degradation and disruption of the cell membrane, increasing its permeability, and eventually leading to bacterial cell death. The higher the degree of deacetylation of chitosan, the greater its antimicrobial efficacy. The main disadvantage of chitosan as an antimicrobial agent for textiles is the dependence of its activity on temperature and the limitation of its pH activity to acidic conditions, as well as its weak adhesion to cellulose fibers. Water-soluble quaternized N-chitosan and carboxyalkylated chitosan derivatives are most commonly used as antimicrobial agents, showing antimicrobial activity in a wide pH range. Polycarboxylic acids and imidazolidinone derivatives are mainly used to form a solid bond between chitosan and cellulose fibers. In the presence of a crosslinking agent, the hydroxyl groups of chitosan and cellulose form covalent bonds with the carboxyl groups of the polycarboxylic acid, forming crosslinks between chitosan and cellulose, which significantly improves the resistance of textile materials treated in this way to care cycles [22, 27].

To improve the bonding of chitosan, for the purposes of this study a cotton-polyester blended fabric in twill weave was pretreated with 1.5 M NaOH and then functionalized with chitosan. Three wash cycles were performed to determine the durability. The zeta potential was measured with an electrokinetic analyzer EKA (Anton Paar) before and after functionalization, as well as after the first and third wash cycles, so that the presence of chitosan could be determined. The changes in mechanical properties - thread density, change in mass per unit area, tensile strength and elongation, spectral properties - whiteness, yellowing index and deviation from the white standard, moisture management and antimicrobial efficacy were determined according to standardized methods. Alkali pretreatment was found to increase the number of available hydroxyl groups of the cotton and carboxyl groups of the polyester component in the blend, and to increase the bonding of chitosan and thus wash resistance. Chitosan contributes to better strength and antimicrobial efficacy of the blend.

2. Material and Methods

The cotton/polyester blended fabric 50/50 supplied by Čateks d.o.o. (Čakovec, Croatia) was used. It is a 3/1 twill weave fabric with 38 ends per cm and 19 picks per cm with a mass per unit area of 160 g/m^2. The fabric was scoured and bleached under industrial conditions.

The fabric was pre-treated with 1.5 M NaOH with addition of 4 g/l surfactant Subitol MLF (CHT-Bezema) by exhaustion method at 80 °C for 20 min in the drum of Turbomat P4502 (Mathis) at LR 1:10. Afterwards, the fabrics were rinsed in hot, warm and cold distilled water; neutralized with 1% HCl, then again rinsed in distilled water and air dried.

Chitosan functionalization was performed using the pad dry cure method on Benz Stenter. Fabric was impregnated in a bath of 3 g/l chitosan dissolved in acetic acid, then dried at 110 °C for 4 min and cured at 170 °C for 45 s. After treatment, the fabrics were washed in distilled water at 60 °C for 30 min to remove unbound chitosan.

Three washing cycles were performed according to ISO 6330:2021 *Textiles - Domestic washing and drying procedures for textile testing* in Polycolor, Mathis, at 60 °C, 40 min with the program Washtest 60 using 2 g/l of EMPA ECE reference detergent 98 without optical brightener (Testfabrics Inc.).

Fabric labels and treatments are listed in Table 1.

Table 1. Fabric labels and treatments

Label	Treatment
19M	Cotton/polyester blended fabric
L	Pretreatment with 1.5 M NaOH
K	Chitosan functionalized fabric
0	Washed with distilled water
19MK1	1 washing cycle
19MK3	3 washing cycles

Fabric characterization was accomplished using electrokinetic analysis with the Electrokinetic Analyzer EKA (Anton Paar) and the stamp cell [28]. By using the streaming current approach, the zeta potential (ZP) was determined as a function of the pH (2-9) of the electrolyte, 0.001 mol/l KCl. The Helmholz-Smoluchowski equation [7] was used to compute the zeta potential. The IEP (isoelectric point) was also calculated [7, 28].

The tensile properties were used to examine the changes in mechanical characteristics by determining maximum force and elongation at break according to ISO 13934-1:2013 *Textiles - Tensile properties of fabrics - Part 1: Determination of maximum force and elongation at maximum force using the strip method* using the TensoLab dynamometer (MESDAN-LAB). The fabric count was determined according to ASTM D3775-17e1 *Standard Test Method for End (Warp) and Pick (Filling) Count of Woven Fabrics,* and the mass per unit area according to ISO 3801:1977 *Textiles - Woven fabrics - Determination of mass per unit length and mass per unit area* by weighing on a digital balance ALJ 220-5DNM (KERN) with an accuracy of 0.0001 g.

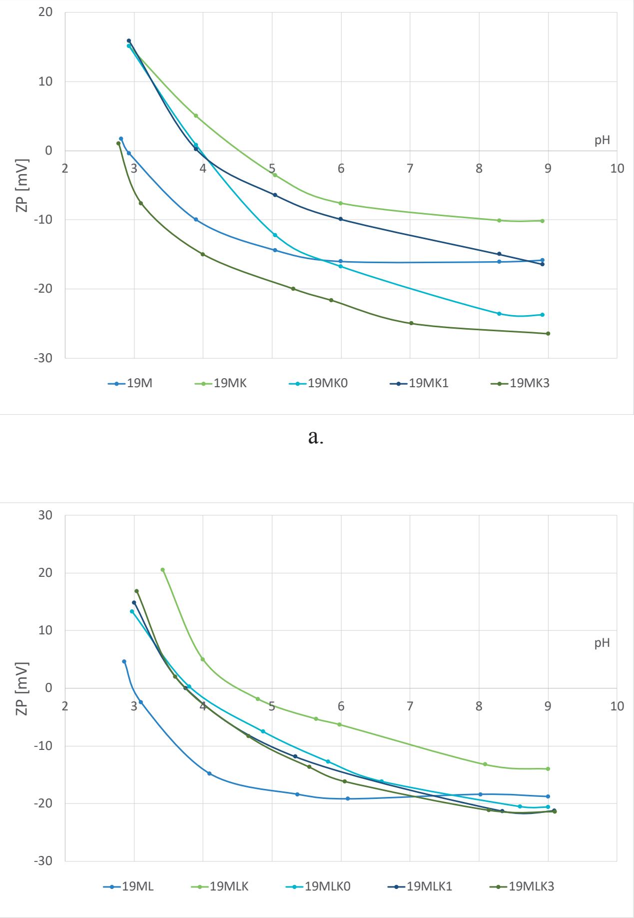
The spectral remission was evaluated using a remission spectrophotometer Spectraflash SF 300 (Datacolor). Degree of whiteness according to CIE (W_{CIE}) was calculated automatically according to ISO 105 J02:1997 *Textiles - Tests for colour fastness - Part J02: Instrumental assessment of relative whiteness* and Yellowing Index (YI) according to DIN 6167:1980-01 *Description of yellowness of near-white or near-colourless materials*. Tint deviation (TD) from the white standard and its coloristic meanings according to Griesser [29] were determined as well.

The moisture management properties were determined according to AATCC TM 195-2017 *Liquid Moisture Management Properties of Textile Fabrics* using the Moisture Management Tester MMT M290 (SDL Atlas).

The antimicrobial activity was determined according to AATCC TM 147-2016 *Antibacterial Activity of Textile Materials: Parallel Streak*. Activity was determined to Gram-positive bacteria *Staphylococcus aureus* ATCC 6538 (*S. aureus*), Gram-negative bacteria *Escherichia coli* ATCC 8739 (*E. coli*), and microfungi *Candida albicans* ATCC 10 231 (*C. albicans*).

3. Results and Discussion

In this work, the influence of cotton/polyester blend fabric pretreatment on chitosan functionalization was investigated. The characterization of fabrics was performed by electrokinetic analysis through the results of ZP presented in Figure 1. From the results, it can be seen that the ZP of the cotton/polyester fabric (19M) at pH 9 is about -15 mV. The negative charge is due to the dissociation of the hydroxyl and carboxyl groups of the fibers. In cotton due to the dissociation of the hydroxyl groups and in polyester due to the carboxyl groups and its hydrophobic surface [7]. Cotton shows an IEP at pH lower than 2.5, whilst polyester has IEP at pH 4. Blending fibers in the fabric causes a change and the IEP is at pH 3. With alkaline hydrolysis (19ML), the ZP decreases to -18 mV because the swelling of the material is greater, and the IEP shifts to pH 3.5.



a.

b.

Fig. 1. Zeta potential (ZP) of cotton/polyester blended fabrics vs. pH of 0.001 M KCl, (a.) before and (b.) after pretreatment and chitosan functionalization up to three washing cycles

According to the literature [7], hydrophilic surfaces may adsorb water (hydration) and swelling, and so have a greater zeta potential. Cotton swelling [4] and the availability of active groups are both caused by alkali pretreatment. When polyester is hydrolyzed, the number of surface-active groups increases.

With the addition of chitosan, the surface of the fabric becomes more positive (ZP -10 mV), indicating a decrease in free active groups, and the positive charge of the chitosan comes to the fore ($-NH_3$ groups dissociate). The IEP rises to 4.5-5. This effect is particularly noticeable in materials that have been alkali pretreated/hydrolyzed, indicating greater chitosan bonding (19 MLK). Washing removes unbound chitosan, although in most cases, the ZP stays more positive after washing. This is supported by the IEP values, which have remained nearly constant.

The results from the measurement of the degree of whiteness (W_{CIE}) shown in Table 2 show that the treatment did not affect the whiteness of blended textiles. Fabrics are whiter after alkali pre-treatment, but chitosan functionalization remains the same. It is to emphasize that the white standard has no discernible difference in tint.

Tables 3 and 4 present the findings of mechanical parameters, such as breaking force and elongation, fabric count, and mass per unit area of cotton/polyester fabrics before and after alkali pre-treatment.

Table 2. Fabric whiteness (W_{CIE}), Yellowing Index (YI), Tint Deviation (TD) and its coloristic meaning

Fabric	W_{CIE}	YI	TD/ coloristic meaning
19M	79.9	0.14	No appreciable deviation in tint from the white standard
19MK	75.9	1.59	
19MK0	77.9	0.80	
19MK1	78.5	0.62	
19MK3	79.0	0.45	
19ML	86.8	-0.03	
19MLK	82.7	1.45	
19MLK0	81.3	1.68	
19MLK1	83.9	0.89	
19MLK3	83.1	1.09	

Table 3. Fabric count and mass per unit area

Fabric	warp [No/cm]	weft [No/cm]	m [g/m^2]
19M	38.3	17.8	157
19MK	38.8	18.3	161
19MK0	39.4	18.4	165
19MK1	37.8	18.3	163
19MK3	37.8	18.7	163
19ML	38.5	18.1	188
19MLK	39.2	18.3	192
19MLK0	39.8	18.7	196
19MLK1	39.8	18.4	193
19MLK3	39.6	18.2	192

The declared warp count was 38 ends/cm, and it did not alter after the alkali pre-treatment or chitosan functionalization. Slightly lower values were discovered for the claimed weft count of 19 picks/cm and mass per unit area. However, the fabric shrank, but the number of weft threads and mass per unit area rose. The cause of this is swelling of the cellulose component during wet processing. Because just the cellulose component of the blend swells, the increase is small, only 2-4%. It is also clear that chitosan treatment does not affect the mass of the fabric. During the first wash, there is more shrinkage and an increase in mass, confirming that the treatment is wash-resistant.

Table 4. Fabric count and mass per unit area

Fabric	F [N]	ΔF [%]	e [%]	Δε [%]
19M	1202	0.00	12.29	0.00
19MK	1204	0.17	14.70	19.59
19MK0	1278	6.32	15.45	25.69
19MK1	1241	3.24	16.60	35.05
19MK3	1283	6.74	17.05	38.70
19ML	1069	-11.06	14.69	19.52
19MLK	1151	-4.24	16.35	33.01
19MLK0	1195	-0.58	18.69	52.08
19MLK1	1156	-3.83	17.62	43.35
19MLK3	1152	-4.16	18.54	50.86

Polyester fibers are known to have excellent mechanical properties. According to the breaking force data, alkali treatment of polyester causes hydrolysis, and the tensile strength is reduced by 11% (19ML). The cotton component swells, whereas the polyester component shrinks due to alkaline hydrolysis.. The elasticity of the two components, and hence of the fabric itself, rises by 19% (19ML). The reason for this is regulated hydrolysis of polyester, cotton swelling, and shrinkage, all of which contribute to fabric strength retention.

Fabric exhibits improved strength and elongation following chitosan functionalization. The tensile strength increases because chitosan is coated in functionalization, which strengthens the fabric. It is also important to emphasize that with an increasing number of wash cycles, there is no loss of strength, but rather an increase in elongation.

Table 5 displays the findings of liquid moisture management qualities as mean values and coefficients of variation (CV) for each measured parameter for the Top surface (T) and Bottom surface (B). The results obtained are: Wetting Time (WT), Absorption rate (AR), Maximum wetted radius (MWR), Spreading speed (SS), Accumulative One-way Transport Capability (R) and Overall (liquid) Moisture Management Capability (OMMC).

Table 5. Moisture management properties of cotton/polyester fabrics before and after alkali pre-treatment, chitosan functionalization and washing

Fabric		**19M**		**19ML**	
Parameter		**Mean**	**CV**	**Mean**	**CV**
WT (s)	**T**	2.375	0.0994	2.25	0.0418
	B	2.5	0.0781	2.3437	0.0399
AR (%/s)	**T**	56.4877	0.0197	53.534	0.008
	B	62.0323	0.0087	55.833	0.0048
MWR (mm)	**T**	25	0	25	0
	B	25	0	26.667	0.1083
SS (mm/s)	**T**	6.7875	0.031	7.1172	0.0178
	B	6.7519	0.0291	7.1183	0.0429
R (%)		162.527	0.0986	115.42	0.0999
OMMC		0.6307	0.0295	0.5611	0.0229
Type		MMF		MMF	

Fabric		**19MK**		**19MLK**	
Parameter		**Mean**	**CV**	**Mean**	**CV**
WT (s)	**T**	14.7917	0.367	4.1663	0.1751
	B	8.6877	0.0856	4.193	0.0901
AR (%/s)	**T**	8.986	0.1362	24.6493	0.2524
	B	30.628	0.2314	38.9672	0.0999
MWR (mm)	**T**	15	0	20	0
	B	18.3333	0.1575	20	0
SS (mm/s)	**T**	0.8578	0.3295	3.4827	0.1448
	B	1.1997	0.2553	3.6911	0.1102
R (%)		613.373	0.0658	267.976	0.3033
OMMC		0.5776	0.0632	0.655	0.0793
Type		MMF		MMF	

Fabric		**19MK0**		**19MLK0**	
Parameter		**Mean**	**CV**	**Mean**	**CV**
WT (s)	**T**	9.7707	0.1662	3.0937	0.0302
	B	8.5157	0.1568	3.1877	0.0509
AR (%/s)	**T**	10.9331	0.1623	45.4939	0.0264
	B	40.9454	0.1321	49.553	0.0463
MWR (mm)	**T**	11.6667	0.2474	21.6667	0.1332
	B	20	0	20	0
SS (mm/s)	**T**	1.1233	0.1184	5.4066	0.0832
	B	2.0801	0.0765	4.9708	0.0244
R (%)		506.918	0.1134	161.667	0.0625
OMMC		0.676	0.0418	0.5951	0.0155
Type		MMF		MMF	

Fabric		19MK1		19MLK1	
Parameter		**Mean**	**CV**	**Mean**	**CV**
WT (s)	**T**	2.6567	0.0539	2.6663	0.0914
	B	3.0003	0.0312	2.854	0.0525
AR (%/s)	**T**	53.6087	0.006	51.502	0.0087
	B	57.8656	0.0255	54.2083	0.0338
MWR (mm)	**T**	20	0	25	0
	B	25	0	23.3333	0.1237
SS (mm/s)	**T**	5.9568	0.0162	6.3929	0.043
	B	6.1673	0.0481	6.0904	0.1402
R (%)		151.316	0.1717	119.379	0.3465
OMMC		0.6066	0.0536	0.561	0.0909
Type		MMF		MMF	

Fabric		19MK3		19MLK3	
Parameter		**Mean**	**CV**	**Mean**	**CV**
WT (s)	**T**	2.474	0.0403	2.9067	0.1116
	B	2.63	0.0033	3.1563	0.0684
AR (%/s)	**T**	54.5092	0.0131	45.9961	0.043
	B	58.1552	0.0009	51.0711	0.0218
MWR (mm)	**T**	23.3333	0.1237	20	0
	B	25	0	21.6667	0.1332
SS (mm/s)	**T**	6.8659	0.0509	5.3753	0.0743
	B	6.8977	0.0137	5.5287	0.1458
R (%)		175.328	0.0966	124.4	0.1131
OMMC		0.6341	0.0295	0.5579	0.0301
Type		MMF		MMF	

**Variation coefficient (CV); Wetting Time (WT); Absorption rate (AR); Maximum wetted radius (MWR); Spreading speed SS for top (T) and bottom (B) surface; Accumulative One-way Transport Capability (R); Overall (liquid) Moisture Management Capability (OMMC)*

The wetting time measured at the MMT is the time it takes for the top and bottom surfaces of the cloth to start wetting [31, 32]. Table 5 shows that the untreated cotton/polyester blend fabric (19M) has an extremely short wetting time WT<2.3 and a large wetting radius. In this blend these two components combine the best of both: the good absorbency of cotton and the high capillarity of the hydrophobic surface of polyester. In the case of fabrics treated with alkali, the wetting time is < 2.5 s. This is due to the change in the surface and the better absorption of the polyester components, so that a small amount of water bonds to the new surface groups of the polyester fibers. Measurements confirm the hydrophilicity of all cotton/polyester samples: the wetting time is very fast and the maximum wetting radius MWR is 25 mm. For this reason, the spreading rate, which represents the accumulated rate of surface wetting from the center of the sample where the discharge solution descends to the largest wetted radius, is somewhat lower. The accumulative one-way transport capability (R) represents the difference between the areas of the upper and lower curves of the moisture content of the surface of the sample as a function of time. All fabrics benefit from efficient transport.

Total (Liquid) Moisture Management Capability (OMMC) is estimated by adding three measurable qualities together: Liquid absorption rate at the bottom, one-way liquid transport capability, and maximum moisture spreading rate at the bottom. This is a measure of the fabric's ability to carry liquid moisture. OMMC is present in all fabrics and is a great indicator of moisture management fabric and does not change with alkali treatment.

The addition of chitosan changes the properties. It is evident that the wetting time increases following chitosan treatment. Additionally, there is a distinction between untreated and alkali-pretreated samples. The MWR value decreases for non-alkali pre-treated samples while remaining nearly unchanged for alkali pre-treated samples. Regardless of these differences, excellent transport is achieved, and all fabrics are of the "Moisture Management Fabric" (MMF) type. The fabric type is also unaffected by the wash cycles.

Since the presence of chitosan was also detected after 3 washing cycles, the antimicrobial efficacy was evaluated according to AATCC TM 147-2016 for *S. aureus*, *E. coli,* and *C. albicans*. The activity as example is shown in Fig. 2 and the results are listed in Table 6.

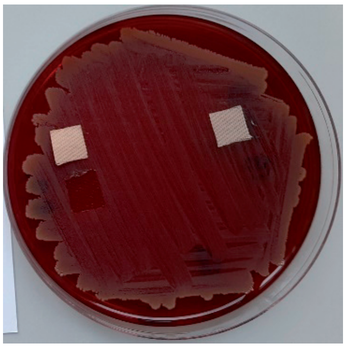


Figure 2. The antimicrobial activity – an example

It is clear from the results in Table 6 that both nonfunctionalized fabrics (19M, 19ML) showed no activity against Gram-positive bacteria *Staphylococcus aureus*, Gram-negative bacteria *Escherichia coli*, and microfungi *Candida albicans*. It is also clear that chitosan functionalization resulted in high antimicrobial activity. Although there is no the zone of inhibition because chitosan is not leaching from fabric, there are no bacterial colonies directly under the sample in the contact area, indicating that the fabrics have antimicrobial activity (Fig. 2).

Table 6. The antimicrobial activity of fabrics before and after functionalization with chitosan and 3 washing cycles

Fabric	*S. aureus*	*E. coli*	*C. albicans*
19M	-	-	-
19MK	+/-	+/-	+/-
19MK0	+/-	+/-	+/-
19MK1	+/-	+/-	+/-
19MK3	+/-	+/-	+/-
19ML	-	-	-
19MLK	+/-	+/-	+/-
19MLK0	+/-	+/-	+/-
19MLK1	+/-	+/-	+/-
19MLK3	+/-	+/-	+/-

\+ *antimicrobial activity (zone of inhibition can be observed);*
+/– *antimicrobial activity (no colonies beneath);*
– *no antimicrobial activity*

The antimicrobial activity of chitosan is attributed to its polycationic nature. It most likely interacts with the anionic membrane of the microorganism, resulting in a change in permeability that causes cell death by leakage of intracellular plasma. Therefore, the electropositive character of chitosan plays an important role in antimicrobial activity. It should be emphasized that the antimicrobial activity obtained remains after 3 washing cycles, indicating a sufficient amount of chitosan in the fabric structure.

4. Conclusions

In this work, the influence of cotton/polyester blend fabric pretreatment on chitosan functionalization was investigated. For this purpose, a cotton/polyester blend fabric in twill weave was pretreated with 1.5 M NaOH and then functionalized with chitosan. Three wash cycles were performed to determine the durability.

Alkali pretreatment was found to increase the number of available hydroxyl groups of the cotton and carboxyl groups of the polyester component in the blend, and to increase the bonding of chitosan and thus wash resistance. Chitosan contributes to higher strength and antimicrobial efficacy of the blend. The results confirm hydrophilicity of cotton/polyester fabrics and fabric type is “Moisture Management Fabric”.

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Assessment of polyester knitted fabrics and effluents from standard and innovative washing processes

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Abstract

The purpose of this research was to establish the link between the surface of pristine polyester knitted fabrics before and after alkali hydrolysis modification, as well as the composition of effluents following standard and innovative washing processes. Using the streaming potential method, substantial variations in the surface of the pristine knitted fabrics after ten washes in the standard and innovative washing processes were discovered in comparison to the alkali-hydrolysed knitted fabrics. The results of the tests carried out with the selected processes establish a relation between the properties of pristine and the alkaline hydrolysed knitted fabrics and the composition of the effluents and filter cakes. The results revealed that the innovative washing process has the potential for further investigations into the release of microfibers from knitted polyester fabrics.

Keywords: *polyester knit, alkali hydrolysis, washing, microfibers*

1. Introduction

The global issue of the past few decades has been the presence of a variety of microscopic particles, such as spheres, fragments, and debris, or fibres, in the environment from different sources, including wastewater, air, soil, and sediments. Microplastic (MP) is a collective name for small plastic fragments with an approximate length of 1 to 5 mm or for fibres with a length of 3 to 15 mm and a length to diameter ratio > 3 [1, 2]. Microfibres (MFs) are similar in size to microplastics, but their composition is not exclusively limited to plastics. Microfibres of natural origin are biodegradable, but the functionalisation of textiles made from natural fibres can slow down or prevent biodegradation, which can be harmful to aquatic organisms [3]. The fragments from synthetic materials such as polyamide (PA), polyester (PES), polypropylene (PP), polyacrylonitrile (PAN), polyethylene (PE) and polyurethane (PU) in the form of flexible foams pose a significant environmental threat to internal organs due to their accumulation [4, 5]. Studies indicate that synthetic textiles are a source of secondary MPs, i.e. microplastics produced by fragmentation, weathering/ageing or maintenance of larger items [6]. Among these sources, household clothes washing has the greatest potential for the formation of microplastics and microfibers [7-9]. Many structural factors influence the formation and release of microfibres when washing synthetic textiles, including type, geometry, yarn type, processing, etc. [4]. The extent of the changes depends on the factors of the washing process, which are determined by a Sinner cycle (chemicals, temperature, time, mechanical agitation) [10].

Accordingly, intensive research has been conducted on the washing process of synthetic textiles over the last decade, with some of the research devoted to factors in the washing process [11, 12], prevention of particle release [13, 14], installation of filters in household washing machines [15, 16] and methodology of analysis of released particles [17, 18]. For a long time obtaining comparable and accurate results was difficult due to the lack of standardised methods, protocols and approaches. The American Association of Textile Chemists and Colorists, AATCC, released the first technique for evaluating particle/fibre fragment release from home washing in 2021, AATCC TM212-2021 [19] which goes some way to resolving the inconsistencies in the protocol for conducting the washing process and the method for analysing the released particles that are dubious when assessing the extent of pollution in the effluent from the washing process. The procedure defines key terms that are often the subject of discussion in scientific and professional communities when it comes to defining released particles, namely: Fibre, Fibre Fragment and Microfibre [20].

Further progress followed in 2023 with the announcement of three parts of the international standard ISO 4484 dedicated to microplastics from textile sources [21-23]. Standard ISO 4484-1 describes a protocol and gravimetric method for material loss of all types from fabrics under washing test conditions [21]. Detergent has been left out of the test method due to clogging of filters during the filtration procedure, stick to fibers and filters, which can interfere with fiber fragment release by adding mass and lead to misinterpretation of results. Despite these facts, the washing process in water, without detergent, has a different effect on fiber fragment release than washing with detergent. ISO 4484-2 [22] specifies a method for determining microplastics (from the textile industry) collected in various matrices (such as textile process wastewater, clothes washing water, textile process air emissions, and textile process solid waste). ISO 4484-3 [23] specifies a method for measuring the collected material mass released from the outlet hose of a standard washing machine, during the washing operation, as specified in EN ISO 6330 [24].

The research contribution to this interdisciplinary topic from 2020 is provided by the project HRZZ-IP-02-2020, 7575 “Assessment of microplastic shedding from polyester textiles in washing process” [25]. The research area of this project focuses on the assessment of microplastics of textile origin released into the environment using innovative washing process and chitosan treatment of polyester fabrics in an eco-friendly manner.

The standard method, EN ISO 6330, is adjusted in the innovative washing process to adapt the washing parameters for synthetic textiles, polyester and polyester/cotton fabrics, and knitted fabrics. Surface modification using the biopolymer chitosan is used in the environmentally friendly treatment of polyester and polyester/cotton fabrics and knitted fabrics to minimize MP shedding from synthetic textiles into the environment. Alkaline hydrolysis of polyester textiles is one of existing methods for modifying the surface of polyester to optimize its interaction with chitosan. This topochemical reaction in sodium hydroxide solution is saponification, which occurs through the hydrolysis of ions attached to the carbonyl group in the polyester chain [26-28].

This research focuses on the investigation of pristine and alkaline hydrolysed polyester knitted fabric as a donors of textile origin particles in standard and innovative washing processes with reference detergent at 60 °C. The effects of these processes were investigated using the fabric surface properties before and after ten washing cycles, the composition of the effluents collected after ten cycles, and the filter cake.

2. Experimental

Material

Double faced interlock standard polyester knit, MRF-0008, Wfk, Germany with a mass per unit area of 139 g/m^2, Dh= 16 stitches/cm and Dv = 21 stitches/cm was used in the research.

Alkali hydrolysis

The fabric was alkaline hydrolysed in a Mathis laboratory apparatus for 30 minutes in a 2 % sodium hydroxide solution at 98 °C. This was followed by two hot rinse water cycles and two cold rinse water cycles.

Washing process

The washing of pristine and alkali-hydrolysed PES knitted fabric was performed in SDL Atlas Rotawash equipment using the 2A protocol of standard EN ISO 6330: 2021[24] in a solution of 1.25 g/L ECE A phosphate-free reference detergent [29] at 60 °C with a bath ratio of 1:7. The fabric was rinsed four times with cold water at a temperature of 20 °C and a bath ratio of 1:8 after each cycle. The innovative process follows the 2A protocol for washing, with modifications made for gradual cooling throughout the four times rinsing. The first cycle is done at 50 °C, the second at 40°C, the third at 30°C, and the fourth at 20 °C. After each individual wash and rinse cycle, the effluents were collected as composite samples from 10 washings. Table 1 lists the characteristics of PES knitted fabrics, both before and after ten washes.

Table 1. Designation and description of samples

Labels	Description of standard polyester knit
P_PES	Pristine
P_PES-St_10	Pristine 10 times washed by standard process
P_PES-In_10	Pristine 10 times washed by innovative process
P_PES_AH	Alkali hydrolysed
P_PES_AH-St_10	Alkali hydrolysed 10 times washed by standard process
P_PES_AH-In_10	Alkali hydrolysed 10 times washed by innovative process

Characterisation of the polyester knitted fabrics

The fabric surface before and after alkaline hydrolysis and 10 washing cycles was characterised by determining the zeta potential as a function of pH 1 mmol/L KCl using the streaming potential method in the SurPASS electrokinetic analyser, A. Paar, Austria. Polyester knitted fabrics with a higher percentage of breaks and wrinkles are a potential donor of particles in the wash and rinse cycles, so their surface and appearance [30] before and after 10 wash cycles of the standard and innovative processes was evaluated by a panel of four examiners and presented as an average value. The surface appearance of the pristine and 10 times washed samples after cyclic rubbing of 125, 600, 1000, 2000, 5000 and 7000 cycles before and after washing in dry condition was evaluated according to standard method [31]. The surface of the polyester knitted fabrics previously coated with gold and palladium for a period of 90 s was examined with a scanning electron microscope (SEM) tt. Tescan, MIRA /LMU, Czech Republic using magnifications of 1,000x.

Characterization of washing effluents

The effluent was separated into a filtrate and a filter cake using membrane filtration; a glass fibre membrane with a pore size of 0.7 µm was selected for this purpose.

Since microplastic emissions are not directly monitored, according to the Best Available Techniques (BAT) reference document for the textile industry should be considered as total suspended solids (TSS) [32, 33]. Accordingly, the analysis of the composite effluent samples was performed by determining TSS [34], turbidity (NTU) according to [35] and pH [36]. In addition, the particle size distribution (PSD) was determined by the laser diffraction method using the particle size analyser PSA 1090 LD, A. Paar, Graz, Austria [37]. All measurements were taken in triplicate and the results were reported as mean values.

To facilitate sampling of the filter cake sample using a digital microscope, the fibrous formations on the surface were counted and labelled as a preliminary measure for the examination of the filter cake sample by pyrolysis-gas chromatography-mass spectrometry (Py-GC/MS). Measurements were carried out using a micro-furnace pyrolyzer (EGA/Py-3030D, Frontier Laboratories, Ltd.) equipped with an auto-shot sampler (AS-1020E, Frontier Laboratories, Ltd.). The pyrolyzer was interfaced directly to a split/splitless injection port of a GC/MS instrument (GCMS Shimadzu QP2010 Plus). The GC injection port was connected to a quadrupole mass detector through a separation column (Ultra ALLOY+-5, 30 m×0.25 mm i.d., coated with 0.5 µm film thickness of 5% diphenyl 95 % dimethylpolysiloxane, Frontier Laboratories, Ltd.) and a vent-free GC/MS adapter (Frontier Laboratories, Ltd.). The detailed analytical conditions are listed in Table 2.

Table 2. Analytical conditions for Py-GC/MS

Instrument	Parameters	Settings
Pyrolyzer	Furnace temperature	600°C
	Interface temperature	300°C
GC	Injection port temperature	300°C
	Column oven temperature	40°C (2 min hold) - 320°C (20°C min^{-1}, 16 min hold
	Flow Control mode	Pressure
	GC/MS interface temperature	300°C
	Injection mode	Split (split ratio: 1:16)
	Carrier gas	Helium (column flow rate: 0.87 mL min^{-1})
MS	Ion source temperature	250°C
	Ionization method	Electron ionization (EI), 70 eV
	Scan range	*m/z 29– 350*

The pyrolysis temperature was preheated at 600 °C and the resulting pyrolyzates of the sample of 0.2 mg, placed into the deactivated stainless steel sample cup.

The qualifications and identifications of peaks in the chromatograms were confirmed by comparing the mass spectrum of each peak in the chromatogram with those in data search libraries of F Search all in one ver. 3.8 (Frontier Laboratories Ltd., Japan) and NIST/EPA/NIH (NIST 17).

3. Results and discussion

The effects of standard and innovative washing processes on the surface properties of polyester knitted fabrics were analysed by streaming potential, wrinkle appearance, resistance to surface fuzzing and pilling and scanning electron microscopy. Zeta potential values of pristine and alkali-hydrolysed PES knitted fabric before and after 10 washing cycles according to standard and innovative procedures in variation of the pH of 1 mmol/L KCl are shown in Fig. 1.

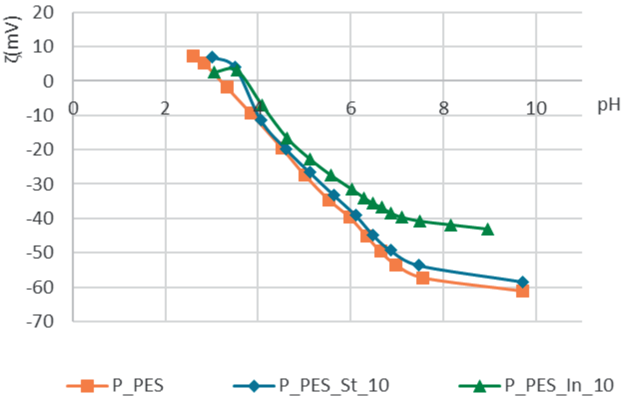

Fig. 1a. Zeta potential of pristine knitted fabrics before and after 10 washing cycles according to standard and innovative process in variation of the pH of 1 mmol/L KCl

Figure 1a shows the zeta potential of pristine (P_PES) knitted fabric sample as shown by a typical titration curve [38]. The zeta potential values of polyester knitted fabrics washed according to standard process (P_PES-St_10) are nearly equal to the values pristine samples. Polyester knitted fabrics washed 10 times using an innovative process (P_PES-In_10) had a lower negative impact than the original sample (P_PES). When compared to the standard wash, the knitted fabric that went through the new method exhibited a lower negative charge, which might imply that its hydrophilicity was improved.

Zeta potential of alkali-hydrolysed PES knitted fabric before and after 10 washing cycles according to standard and innovative process in variation of the pH of 1 mmol/L KCl is shown in Fig. 1b.

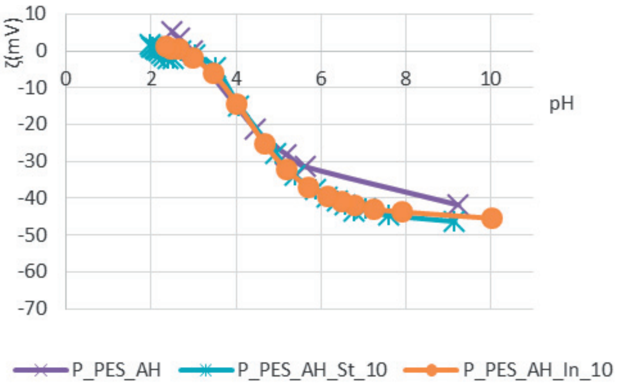

Fig. 1b. Zeta potential of alkali-hydrolysed knitted fabrics before and after 10 washing cycles according to standard and innovative process in variation of the pH of 1 mmol/L KCl

The influence of the alkaline hydrolysis process (AH) on the surface of the fabric is demonstrated by decreasing the negative value of the zeta potential compared to the original PES fabric in the observed pH range, and the value of the isoelectric point (IEP) of the alkaline hydrolysed fabric confirms the modification of the surface.

Titration curves for alkaline hydrolysed polyester fabric were identical when washed 10 times using the standard procedure (P_PES_AH-St_10) and the innovative method (P_PES_AH-In_10). The differences between the two alkaline processes, hydrolysis and standard washing process, are confirmed by the obtained ratios of the zeta potential titration curves. The degree of modification of the polyester fabric during alkaline hydrolysis is more reflected in the change of surface properties [26, 39, 40], and both the standard and innovative alkali washing processes have no influence on the surface of the alkaline hydrolysed samples.

SEM micrograph of knitted fabrics before and after standard and innovative washing processes are shown in Fig. 2.

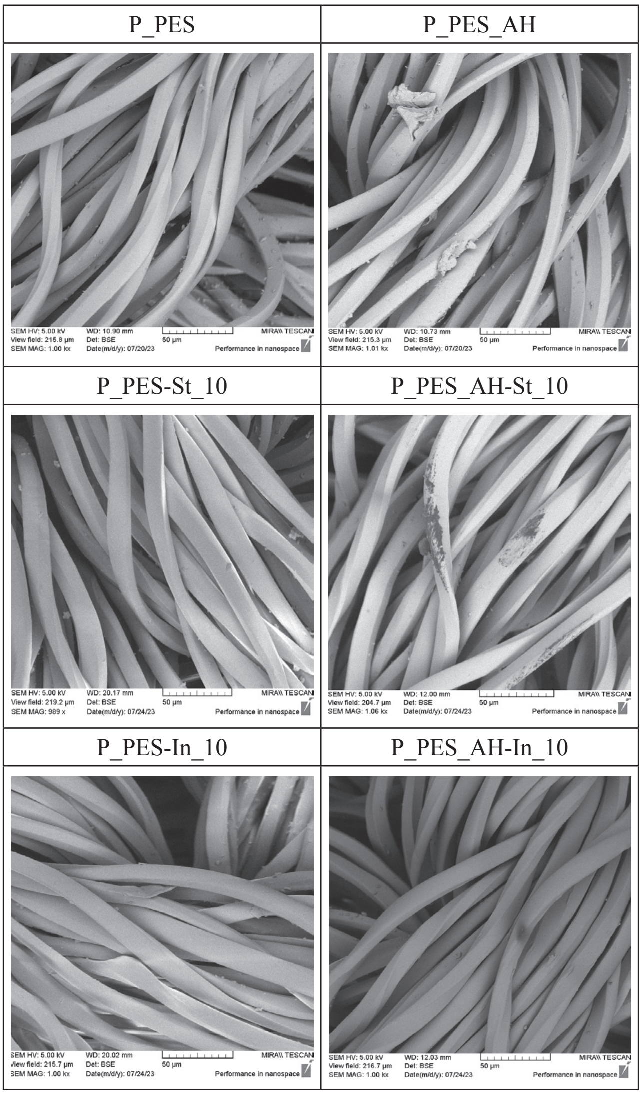
P_PES
P_PES_AH
P_PES-St_10
P_PES_AH-St_10
P_PES-In_10
P_PES_AH-In_10

Fig. 2. SEM micrographs of knitted fabrics under magnification of 1,000x

Micrographs of PES knitted fabrics before and after 10 cycles of the standard and innovative washing processes reveal no modifications, showing that the washing procedures have no prominent effect on the surface of PES knitted fabrics (Fig. 2). The micrographs of the alkaline hydrolysed knitted fabric after the standard and innovative washing processes differ. The surface of this sample is partially peeled after the standard washing process, in contrast to the sample washed using the innovative process, where no surface changes are visible.

Despite this, all knitted fabrics score a 5 for resistance to surface fuzzing and pilling, which is evaluated both before and after 10 washing cycles using standard and innovative methods. Grades for wrinkle appearance of assessed after ten cycles washings of PES knitted fabrics are summarised in Table 3.

Table 3. Surface appearance grades

P_PES-St_10	P_PES-In_10	P_PES_AH-St_10	P_PES_AH-In_10
2-3	3	1-2	1-2

Grade: 1- surface with many wrinkles; 5 – smooth surface

The results (Table 3) demonstrate that alkaline hydrolysed PES knitted fabrics washed using standard and innovative processes have a greater level of wrinkling than PES knitted fabrics washed using these processes.

Characterisation of the washing effluents

Composite effluents of 10 cycles washing were evaluated by TSS, NTU, pH listed in Table 4, and particle size distribution as shown in Fig. 3 and Fig. 4.

Table 4. Characteristics of 10 cycles washing effluents

Effluent	TSS (mg/L)	NTU	pH
P_PES-St_10	128.3	70.2	7.8
P_PES-In_10	150.5	68.4	8.4
P_PES_AH-St_10	113.3	48.7	7.8
P_PES_AH-In_10	171.5	42.2	8.1

When the results of the characterization of the composite effluents from 10 cycles washing PES knits (P_PES) and alkaline hydrolysed (P_PES_AH) according to the standard and innovative processes are compared (Table 4), a higher content of total suspended solids, TSS, can be observed in the effluent of the innovative process. The surfactant components in the detergent are primarily responsible for the turbidity of the effluent. The obtained results show that the turbidity value of alkaline hydrolysed knitted fabrics is lowered. The surface is modified and the structure is opened up by alkaline hydrolysis. This modification improves the surface ability to interact with detergent ingredients that affect turbidity. The pH of the composite phase effluents from the innovative washing process (pH ~ 8) is higher than that from standard washing.

Figure 3 shows the PSA analysis results as a particle size distribution curve in effluents from 10 cycles of washing PES knitted fabrics using standard and innovative methods, whereas Figure 4 shows effluent after washing alkaline hydrolysed PES knitted fabrics using the same processes.

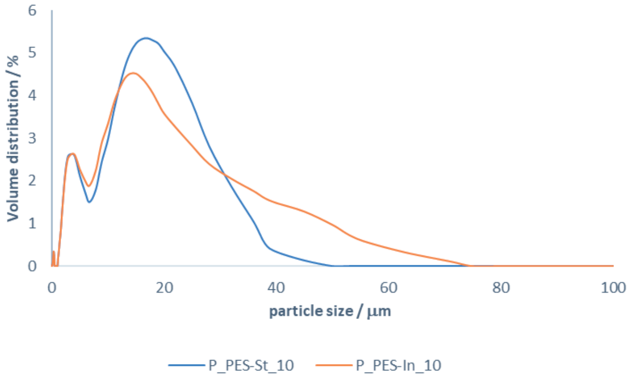

Fig. 3. Volume distribution of particle sizes in the effluent from 10 cycles washing PES knitted fabric (P_PES) using the standard and innovative processes

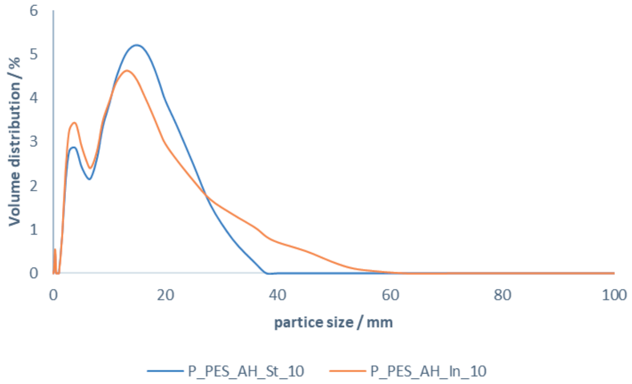

Fig. 4. Volume distribution of particle sizes in the effluent from 10 cycles washing alkaline hydrolysed PES knitted fabric (P_AH_PES) using the standard and innovative processes

The particle size distribution curves for effluents from the standard and innovative washing processes of PES knitted fabrics (P_PES and P_PES_AH) differ in shape, with the innovative process exhibiting a greater range of particle sizes and the lowest volume distribution.

The characteristic diameters D_{10}, D_{50}, D_{90}, average diameter (A) and their ratio as Span value and k parameters of the curve computed from the acquired distribution curves using the computer support of the PSA instrument are presented in Table 5.

Table 5. Characteristic parameters for PSD curves

Parameter	P_PES-St_10	P_PES-In_10	P_PES_AH-St_10	P_PES_AH-In_10
D_{10} (μm)	2.014	2.029	1.866	1.794
D_{50} (μm)	10.776	10.130	8.641	7.473
D_{90} (μm)	23.748	28.836	19.924	21.446
Average (μm)	12.246	13.544	10.216	10.345
k	1.117	1.091	1.122	1.073
Span	2.017	2.646	2.090	2.630

The mean particle diameter in the effluent from washing alkali hydrolysed knitted fabric has reduced, and equivalent values are seen in the effluent from standard and innovative processes. The alkaline hydrolysis affects the particles of smaller diameter in comparison to pristine polyester knitted fabric. According to the reported ratios of characteristic diameters, indicated as span value, the wastewater from the innovative washing process has a higher value. The lowest value of the shape factor k is typical for the effluent from the innovative process involving both PES knitted fabrics.

Filter cakes produced by filtering effluents from 10 cycles of standard and innovative washing processes (PES -St_10; PES -In_10; AH -St_10; AH -In_10) were analysed using Py - GC/MS. The extracted ion chromatographs (EIC), with particular mass values of the selected samples were compared. When comparing the EICs, no significant difference was found between the analysed samples of the cake filter sample after 10 cycles of the standard and innovative washing processes. For the comparison EIC with filter cake samples (PES-St_10; PES-In_10; AH-St_10; AH-In_10) the polyester (AATCC & ISO) multifibre test fabric # 10A (DW) textile standard was used. Pyrolytic decomposition of the textile standard revealed that benzoic acid is one of its main components. The characteristic mass spectrum and EIC of benzoic acid with the intensities of the main m/z values of the polyester (Terylene) textile standard is shown in Fig. 5.

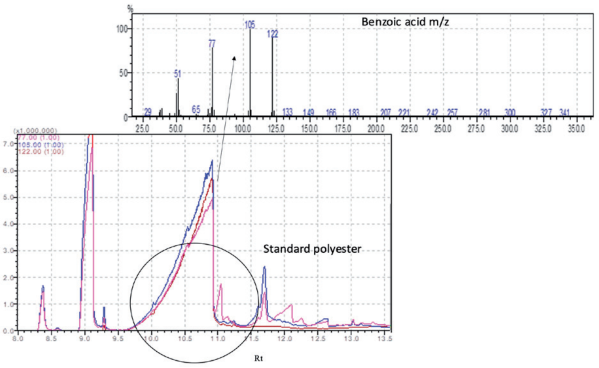

Fig. 5. EIC of standard polyester Multifiber test fabric # 10A (DW) with characteristic mass fragments m/z of benzoic acid

A match may be detected after a specified retention period by monitoring and comparing the EICs, with specific m/z values typical for benzoic acid, of the standard samples with the filter cake samples, Figs. 6-9.

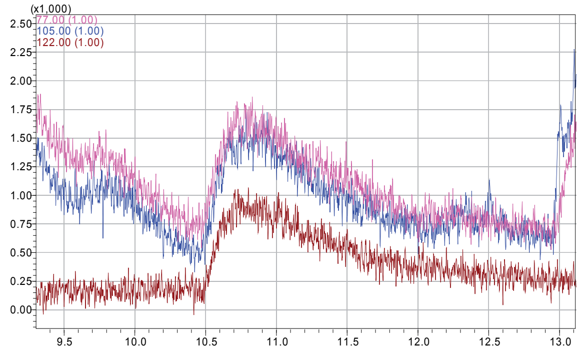

Fig. 6. EIC of filter cake P_PES-St_10

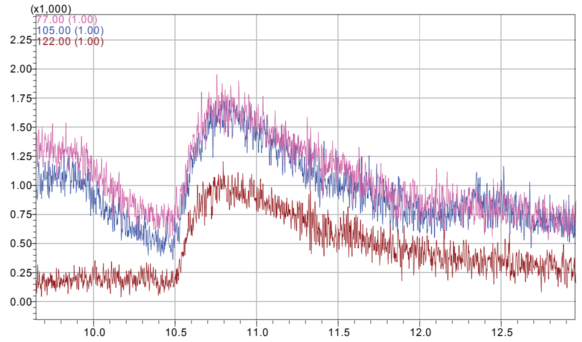

Fig. 7. EIC of filter cake P_PES-In_10

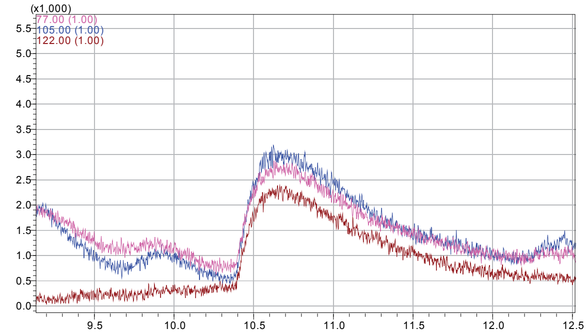

Fig. 8. EIC of filter cake P_PES-AH-St_10

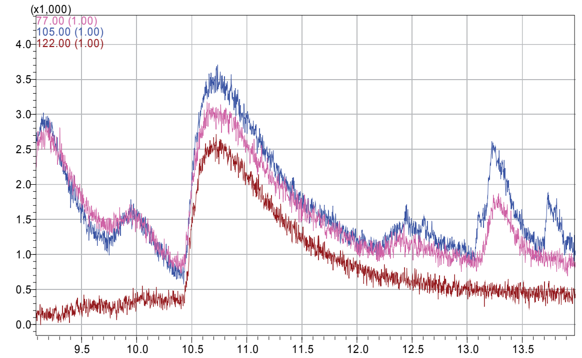

Fig. 9. EIC of filter cake P_PES-AH-In_10

All of these results might point to polyester as the source of the benzoic acid intensities that were seen in the filter cake samples (Figs. 6–9).

4. Conclusions

The surface of pristine PES knitted fabric has been affected by alkaline hydrolysis, as evidenced by the zeta potential curves.

In comparison to the innovative one, washing did not significantly modify the surface of the PES knitted fabric, resulting in a smaller negative surface charge.

The value of the zeta potential does not indicate the difference between the standard and innovative washing processes of alkaline hydrolysed PES knitted fabrics.

Grade 5 for all knitted fabrics showed no tendency to surface fuzz and pilling before and after 10 washing cycles.

The average particle diameter in the effluents from washing of alkali-hydrolysed polyester knitted fabric is smaller than in the effluents from washing of pristine polyester knitted fabric by standard and innovative washing process.

The intensities of benzoic acid originating from the polyester may be seen in the obtained programs of all filter cakes.

The results of the research carried out using the selected methods connect the properties of pristine and alkaline hydrolysis modified knitted fabrics with the composition of the effluent and the filter cake.

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Genetically engineered/modified fibres for the 21st century textiles and fashion

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Abstract

When a journalist asked Steve Jobs what the greatest innovation of the 21st century would be?, he said, "I think the greatest innovation of the 21st century will be the combination of biology and technology" [1]. At that time, almost no one thought that this applied to the textile and fashion industry as well, although genetically engineered (GE) textiles and genetically modified (GM) textile fibres were becoming more and more present in our wardrobes and everyday life. From the early nineties of the last century, when the first ex vitro cultivation of GM cotton was realised, until today, when fibres can be biologically designed, i.e. genetically engineered (GE) according to our needs, GM fibres of plant and animal origin, as well as GE textile fibres of various origins, have become increasingly present as raw materials in the textile and fashion industries, despite numerous controversies surrounding their use. The paper presents an overview of the achievements made in the field of genetically engineered/modified fibres, so far, as well as a review of their use in the context of contemporary thinking on sustainable textiles and fashion.

Keywords: *genetically modified fibres, genetically engineered textiles, fashion industry, sustainable textiles.*

1. Introduction

The twentieth century brought about revolutionary changes in science and technology, which had far-reaching consequences for our lives. [2]. Computers and the Internet were invented, a wide range of synthetic fibres were offered to the market, and the human genome was found and sequenced, all of which allowed biotechnology to become the most popular and promising scientific discipline. Although some forms of biotechnology have been present since the beginning of civilization, through the domestication of plants and animals and the discovery of fermentation, public awareness of the possibilities of biotechnology and genetic engineering is a relatively recent phenomenon, a phenomenon of the 21st century [3]. Biotechnology and genetic engineering are often used interchangeably, although biotechnology represent much broader term that implies the usage of biology in the development of new products, methods and organisms intended to improve human health and society. It can be defined as any technique that uses living organisms to make or modify a product to improve plants or animals or to develop microorganisms for specific use [3-7].

Genetic engineering, also regularly called genetic modification, is just one part of modern biotechnology (along with tissue culture and cloning). It is a collection of current biological techniques used to change an organism’s genetic features, such as introducing, modifying or eliminating certain genes, while also allowing for gene transfer across unrelated species. As a result, a genetically modified organism (GMO) contains additional or modified characteristics encoded by the introduced gene(s) [8]. Through genetic engineering, organisms can be given targeted combinations of new genes, and thus new combinations of properties that do not appear in nature (**Fig. 1.**), i.e. cannot be developed naturally or through long-term and repeated selection of several generations.

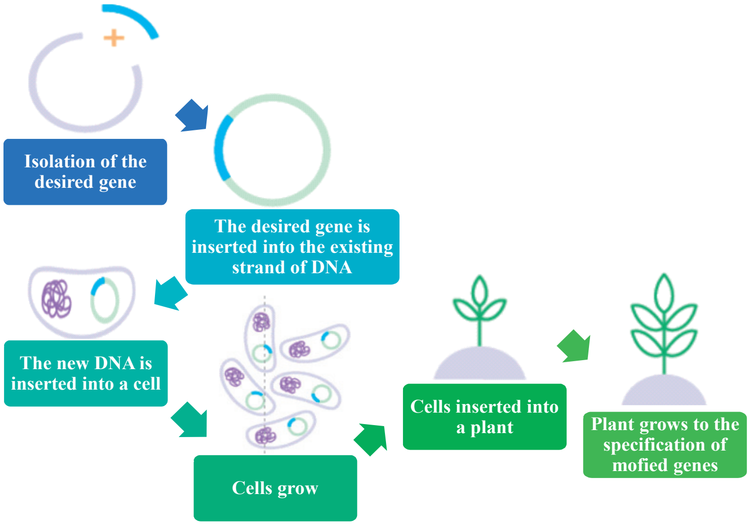


Fig. 1. GMOs depend on genetic engineering for their creation and can be modified in any way a scientist chooses [9]

The possibilities of such genetic manipulation, as well as its benefits, are the main reasons why genetics is today seen as a promising science, and biotechnology as one of the leading industries.

Although, among wider population the textile industry is not perceived as a modern and inventive industry, but as an industry that uses non-renewable raw materials to a greater extent and contributes significantly to environmental pollution, it is worth pointing out that the textile and fashion industries were the first to recognize and use the potential of biotechnology several decades ago. Namely, enzymes are routinely used (**Fig. 2.**) for washing and bleaching textiles, giving the desired fashionable effect of a worn look to denim clothes, preventing shrinkage of wool, etc. [10].

According to some predictions, the new wave of biotechnology and the imperative of sustainable development could lead to the situation where our clothes and functionalised textiles would be made and dyed by living organisms, microbes or bacteria. In this scenario, many of the chemical treatments and manufacturing processes, which are currently most accountable for the negative perception of the textile and fashion industry, could be avoided [11].

Since fibres are the basic building block of every textile product (**Fig. 3.**), it is not surprising that they were the starting point for the implementation of biotechnology and genetic engineering in the textile and fashion industries.

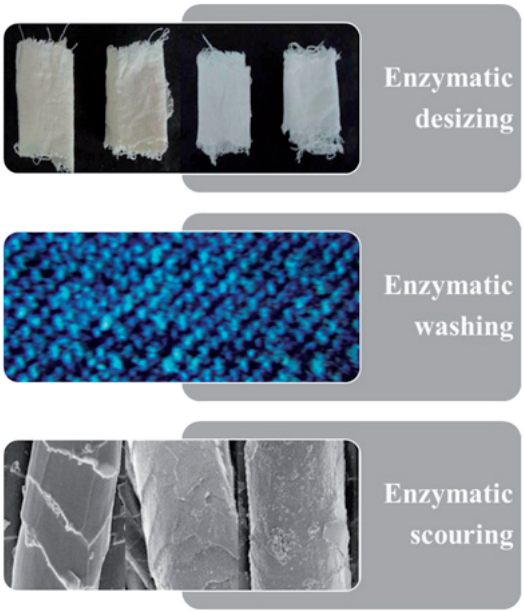


Fig. 2. Some examples of old well-established biotechnology usage in textile industry

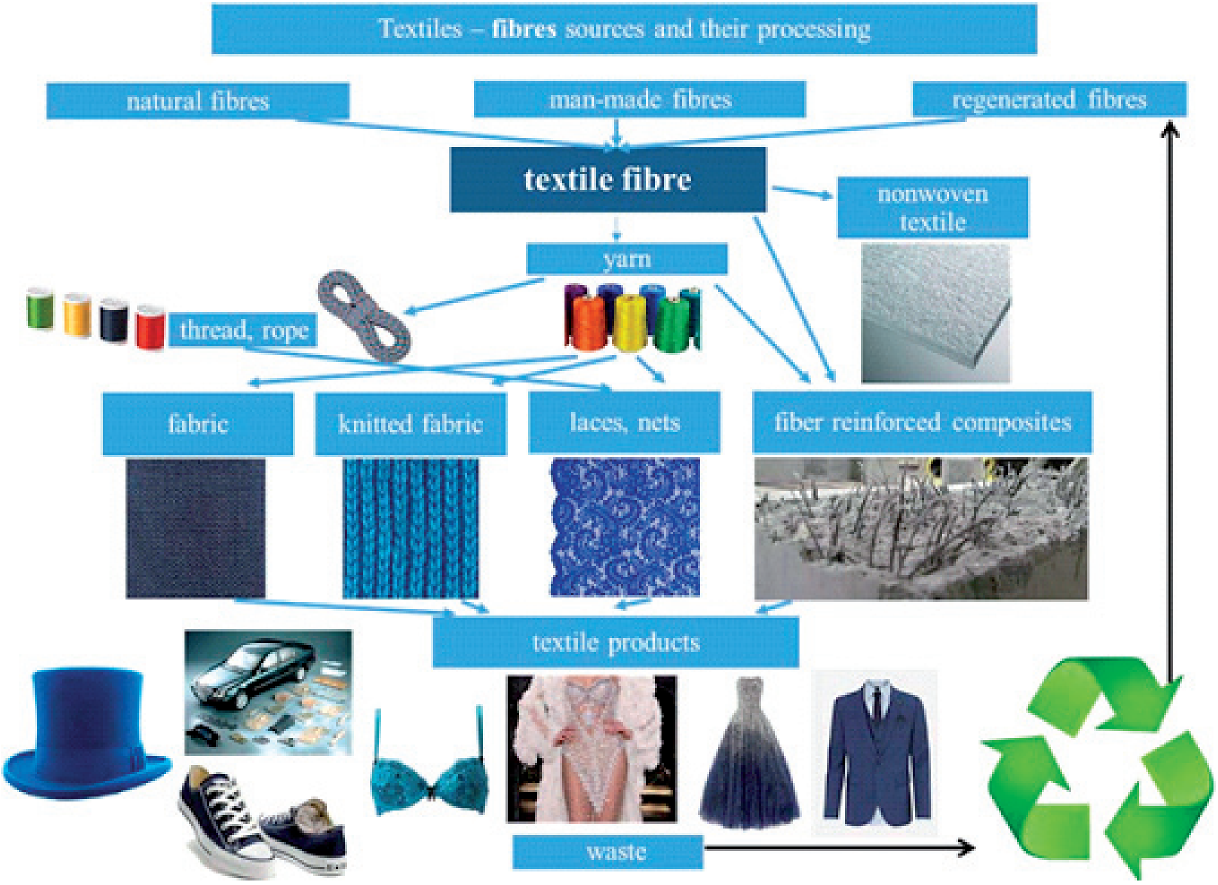


Fig. 3. From fibre to final textile product

2. Genetically engineered/modified natural fibres

Nature has created different types of fibreforming plants and animals, with fibres exhibiting unique properties, resulting from their genetic structure [12]. The genetic traits of naturally occurring fibrous materials are based on the structure and function of DNA (deoxyribonucleic acid), which is the basic carrier of genetic information in all living organisms. DNA is composed of four nucleotides: adenine, thymine, cytosine and guanine, which are covalently linked in a specific order, forming a gene, the functional unit of inheritance [13]. Genes determine the production of specific proteins, which make up the structure and function of all biological systems, including natural fibres. After the discovery of the structure of DNA, in 1953 by Watson and Crick, genetic modification enabled the development of genetic engineering techniques that also found their application in the textile industry [4]. Genetic engineering provides scientists with the possibility of direct intervention in genetic organisms. It allows them to add, remove or modify the existing gene in order to modify certain characteristics of natural fibres [14]. This process uses a variety of methods, including recombinant DNA technology, a process that combines DNA sequences from different sources into a single molecule, CRISPR-Cas9 gene editing, a method for precise gene editing that uses a molecular tool to “cut” and “paste” DNA at desired locations in the genome and transgenesis, the procedure by which the genes of one species are transferred and integrated into the genome of another species, creating so-called transgenic organisms [15-18]. Genetic modification of fibres (namely plants and animals) for the needs of the textile and fashion industries takes place in several directions: increasing fibre yield with less environmental impact, improving the properties of natural fibres and creating new, high-tech fibres, to be used for the production of additionally resistant, durable, more fashionable and environmentally friendly textiles [19-23].

2.1. Genetically engineered/modified plant fibres

Cotton is the seed fibre of the plant of the same name belonging to the *Gossypium* genus of the *Malvaceae* family, and which can be said to be the most important conventional textile fibre despite the increased consumption of different artificial fibres. Thanks to its fineness, strength, shapeability and ability to absorb moisture, cotton is one of the most commonly used materials in the production of clothing [19, 24]. Despite its numerous advantages, conventional cotton also has significant shortcomings, including the need for a significantly high use of water and pesticides during cultivation, and susceptibility to diseases and pests [20, 22]. Only four of the 39 cotton plant species are grown for fibre production: *G. hirsutum*, G. *barbadense*, *G. herbaceum* and *G. arboreum* [25]. Genetic diversity, the increase in crop profit and utilization, and the geographical and market spread of cotton inspired the interest of biotechnologists and geneticists in cotton at the end of the 20th century already. Cotton genetic modification aimed to achieve two primary objectives: to create resistance to pests and tolerance to glyphosate, an herbicide that is often used in agriculture and intensive cotton cultivation [19, 20, 22]. To reduce crop failures, scientists modified cotton genetically by inserting a gene known as Cry2AX1 into the cotton genome. This gene comes from the bacterium *Bacillus thuringiensis* (Bt), which produces a toxin that is harmful to certain species of cotton pests [26]. Successful incorporation of this gene into the cotton DNA creates plants that produce their own pesticide (**Fig. 4.**), resulting in a reduced need to spray chemicals on them. This approach has been used for many years and has proved to provide effective protection against certain types of pests, such as *Helicoverpa armigera*, one of the most dangerous cotton pests [20, 27, 28]. Bt cotton was introduced into commercial use in 1996 [29] and since then has significantly reduced the use of pesticides in many regions of the world, which has had a positive impact not only on the environment, but on human health as well [20, 27, 28].

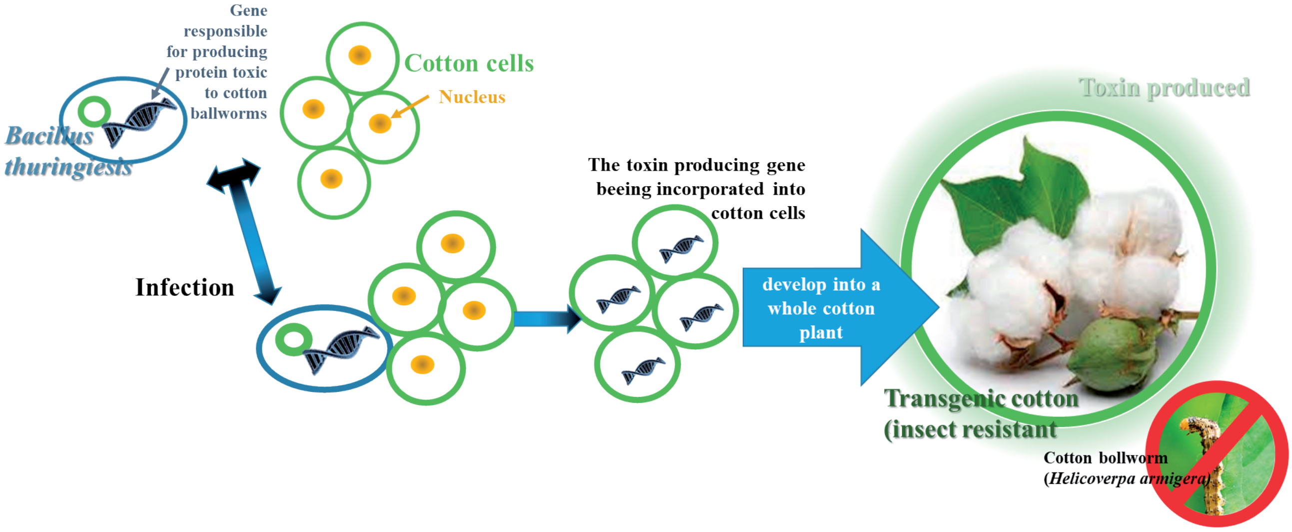


Fig. 4. Production of Bt Cotton

After the successful introduction of Bt cotton, researchers continued to develop new varieties of genetically modified cotton with additional improvements. Namely, cotton (in intensive and extensive cultivation) is sensitive to glyphosate, i.e. one of the most commonly used herbicides in agriculture, which limits its use in cotton fields. To solve this problem, scientists have genetically modified cotton to develop varieties that are resistant to glyphosate [6, 30]. Glyphosate is a broad-spectrum herbicide that in turn inhibits an enzyme known as 5-enolpyruvylshikimate-3-phosphate synthase (EPSPS). This enzyme is crucial for the biosynthesis of essential amino acids, making it necessary for plant growth and development. By modifying the cotton genome, cotton plants can survive glyphosate treatment, which enables effective weed control without crop damage [20, 22, 23]. In order to achieve genetic modification various techniques might be used, including for example the transformation by the bacterium *Agrobacterium tumefaciens*. This method exploits the natural ability of the bacterium for genetic engineering and the ability to transfer new genes into plants [22]. Usually, a modified CP4-EPSPS gene from the bacterial strain *Agrobacterium sp*. is used for this purpose since CP4 strain is naturally resistant to glyphosate [23].

Naturally coloured cotton has grown in popularity in recent years as a result of the ever-present intersection of ecology and fashion, as well as the growing public interest in environmental concerns and environmentally responsible production processes. Coloured cotton, as a type of genetically modified fibre, has become popular in the textile industry due to its environmental friendliness and potential to reduce the costs of textile dyeing [31]. However, there are certain disadvantages of this fibre that reduce consumer enthusiasm for choosing naturally dyed cotton fabrics. The fibres are too short and weak to be used for finer pieces of fabric, and there is a limited selection of desired shades and colours [32]. Colours come in shades of brown, green and purple [31-34]. However, significant progress has recently been made in this area as growers in Australia have genetically modified cotton to produce dyed cotton in black and other dark colours that are durable [35]. Since the introduction of the first genetically modified cotton, biotechnology has progressed, and new generations of GM cotton have been developed with the aim of improving fibre properties (length, strength, colour, resistance to burning, etc.), resistance to diseases and pests, and adaptation to different climatic conditions. Possibilities for reducing the consumption of water and nutrients during cultivation, and thus reducing the negative impact on the environment [36, 37], are also being explored.

Although genetically modified cotton offers many advantages, it also carries certain challenges and controversies. Some of these challenges include potential health and environmental risks associated with genetic modification, as well as ethical issues and regulations that limit its use and distribution [28, 38], such as GOTS (Global Organic Textile Standards) norms and EU directives [39, 40].

Flax, a genus of annual plants from the *Linaceae* family, with more than 200 once yearly and perennial species. The flax fibre is mainly obtained from the stem of the blue flax plant (*Linum usitatissimum* L.), and in addition to being one of the oldest textile fibres, it should be noted that by the end of the 18th century, along with wool and hemp, was the most important textile raw material [36, 41]. Today, when the use of renewable textile raw materials is imperative, flax is gaining in importance once again [42-44]. As a natural fibre, flax is extremely strong, absorbs moisture well and shows resistance to bacteria and fungi [36]. Despite these advantages, conventional flax production faces numerous challenges, including high production costs and limitations in terms of fibre quality, as well as unsustainable maceration processes [45]. Genetic modifications of flax were started at the end of the last century, primarily with the aim of increasing crop yields and flax resistance to herbicides and their residues in the soil [46]. The first flax transgenic plants/fibres on the market were engineered for glyphosate (Roundup®) [30] tolerance. Further study focused on genetic modifications of flax with the goal of improving fibre properties. One example is the introduction of transgenes that encode enzymes involved in lignin synthesis, which, according to some studies, facilitates the extraction of fibres from the stem without impairing their quality [46, 47].

The following step in engineering flax fibre [48] was aimed at generating transgenic flax plants that could be retted more efficiently. The constitutive expression of *Aspergillus aculeatus* genes resulted in a significant reduction in the pectin content in tissue-cultured and field-grown plants. This pectin content reduction was accompanied by a significantly higher retting efficiency of the transgenic flax fibres. Despite the good indicators, there are no genetically modified flax currently grown in the EU, primarily because of the EU regulations and a fact that European importers refused to buy it due to the health and environmental safety issues.

The study of genetic cotton and flax modification has prompted the genetic modification of other plants that generate textile fibres, such as nettle, hemp, and jute. The aim of these researches is to improve fibre properties, including strength, formability and the resistance to diseases and pests, as well as improving the adaptability of plants to different climatic conditions in order to ensure sufficient yields of textile raw materials of plant origin, due to the increased interest in the production of biocomposites [49-54].

Advances in the field of biotechnology and its use for the needs of the textile and fashion industries have recently led to the innovative and creative cultivation of cotton fibres from stem cells in laboratory conditions (**Fig. 5.**) [55]. Various cultivars of cotton plants are grown in greenhouses in order to isolate the stem cells from them in the laboratory. The stem cells, which have the potential to transform into any part of the plant, are placed in enormous bioreactors and their development is aided by the supply of nutrients. Once the cells begin to multiply, they are transferred to a bioreactor where they are differentiated into fibres. Instead of growing the whole plant, this approach merely produces cotton fibres. The process itself is significantly faster than traditional cotton growing - it only takes 18 days compared to the 180 days needed

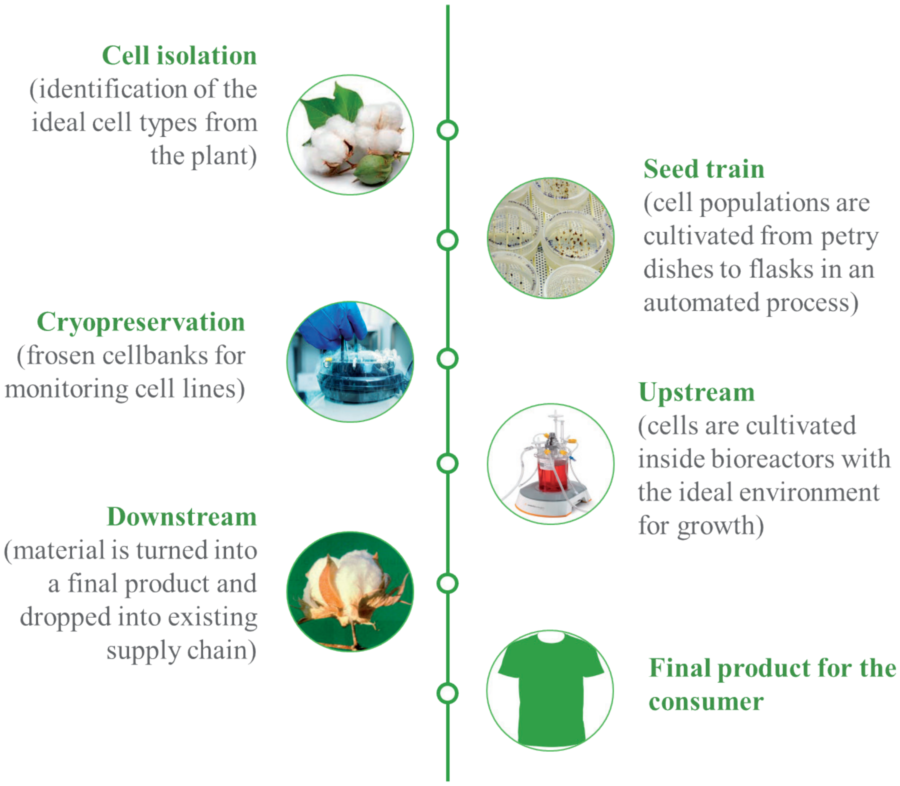


Fig. 5. Production of Bt cotton

for traditional growing. Additionally, this method uses less than 80% of water and land compared to traditional cotton cultivation. However, despite all these advantages, the quality of the fibres themselves is questionable and still unknown. Currently, the project is still in the research and development phase. The plan is to produce cotton, under reasonable price on the market, for ecologically and ethically conscious fashion brands [55, 56].

2.2. Genetically engineered/modified animal fibres

Animal or natural protein fibres is a common name for fibres that represent the hair covering of certain animals (so-called keratin fibres, e.g. wool) and fibres that are secretions of certain animals (so-called fibroin fibres, e.g. mulberry silk) [58].

Wool has been an integral part of human life for millennia, and as such, it has been and continues to be the focus of much research. In particular, wool, thanks to its structure and resulting properties, is considered one of the most complicated fibres whose secrets have not yet been completely discovered. Numerous improvements in the fleece covering of today's sheep compared to their ancestors are the result of sheep domestication, long-term efforts to select the best ones and care for their adequate safe and sound breeding, considering the climatic and vegetation conditions in which they are bred. For generations, genetic selection has been used to improve the existing phenotypic traits of sheep through the breeding of sheep most likely carrying the optimum gene combinations [59, 60]. However, while steady gains have been made, the progress is relatively slow, taking multiple generations. In the process, selective breeding relies solely on genes already within the sheep genome, a factor that clearly sets a biological limit for genetic gain. With respect to the main drivers of profitability in the wool industry, selective breeding has made great progress in the achievement of both higher clean fleece weights and lower fibre diameters. However, that was not enough considering the contemporary requirements of the global textile and fashion industries and their end users. Unlike artificial fibres, wool, as a textile raw material, is extremely non-uniform, not only among individual breeds of sheep (e.g. merino wool and domestic pramenka wool), but also within the breed (different sub-breed), due to the climatic and vegetation conditions in which it is grown (e.g. Australian *vs.* New Zealand or Spanish merino; Istrian vs. Lika pramenka). Additionally, the non-uniformity of wool is also contributed through the fact that wool of the same breed and sub-breed differs according to the gender of the sheep (i.e. ram *vs.* ewe), the part of the body from which it originates (e.g. back wool *vs.* belly wool), but also along the fibre itself (e.g. root *vs.* fibre tip) [61, 62]. Careful on-farm management of nutrition and minimization of environmental stresses contributes to the production of fibres with quite uniform protein compositions as well as properties like fineness, crimpness, length, strength, etc. However, such treatments come at a high cost, in terms of energy, used chemicals and human labour. Traditionally, post-farm processing treatments (starting with sorting and classifying, followed by industrial pre-treatments) have been the primary tool for the improvement or uniformity of wool fibre properties, necessary for its processing into a finished product [63, 64].

It is not unexpected that biotechnology and genetic engineering (especially transgenesis) have emerged as potential alternatives to selective breeding for modifying sheep genetics and achieving fibre modification and unification [65]. Complete decoding of keratin intermediate filaments (KIFs) and keratin-associated proteins (KAPs) present in wool follicles (**Fig. 6.**) has still not been accomplished and is under research. It is partially due to the fact that wool fibre represents the most sophisticated biological composite material and partially because wool fibre characteristics change during fibre formation by the activity of the follicles from which it grows [65-71].

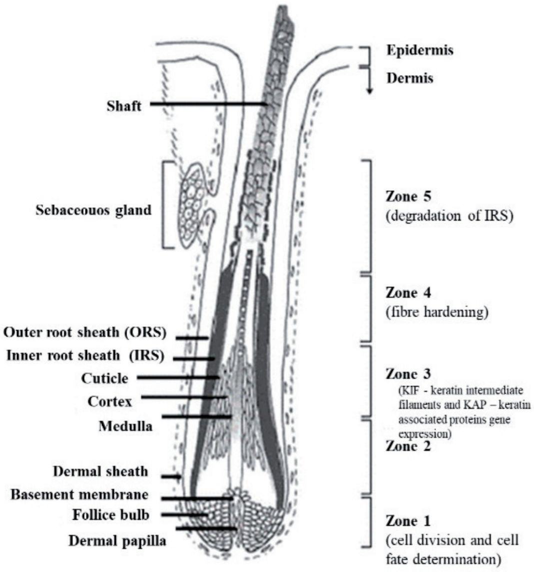


Fig. 6. Sketch of a mature wool follicle and summary of the gene expression that takes place in the wool follicle to produce the KIF and KAP [65, 69]

Numerous attempts have been made to modify wool fibre properties through enzymatically assisted CRISP/Cas9 genetic modification technology. New transgene DNA fragment for adding to sheep has been prepared through this work. In the case of the transgenic sheep made via the cortical-specific K2.10 transgene base vector, sometimes it is possible to see visible difference in the appearance of sheep fleeces depending upon the level of transgene expression, (**Fig. 7.**) [65]. The wool fibres of the high expressor have no crimp but rather a moderate wave, and high lustre. Since waviness is a consequence of cortex bilateral structure, it is obvious that such genetic modification can impact the fibre ultrastructure as well as properties like density, strength and stretchability, dye uptake and moisture sorption [72].

Such genetic sheep modification can offer chances for sustainable, faster and cheaper production of more uniform fibres or functionalized wool of different qualities [73, 74], but it also opens up numerous ethical and safety dilemmas [74], which is illustrated by the news that sheep have already been bred in laboratories with different fleece colours [75].

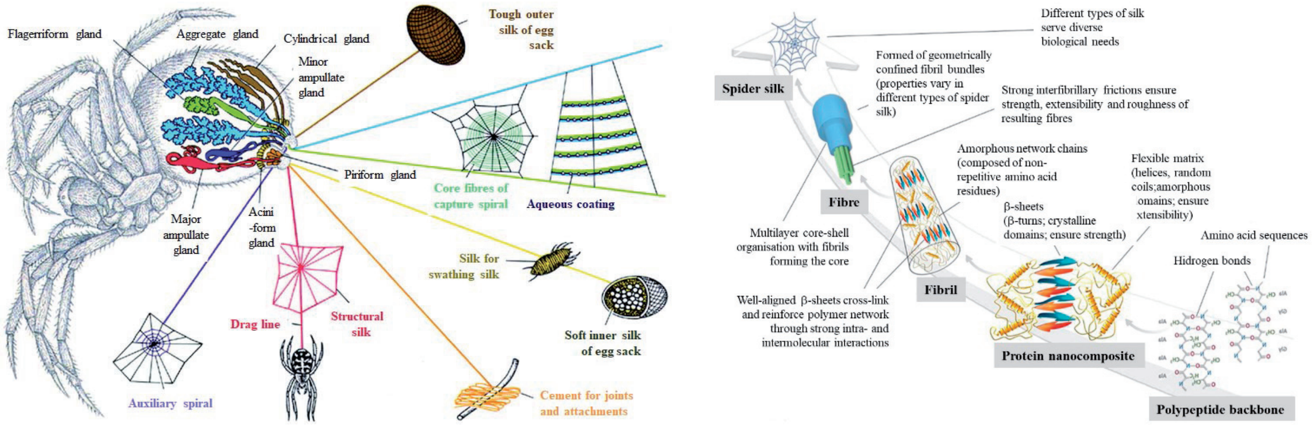
Silk is the common name for all-natural protein fibres that are created by the secretion of silkworms, spiders and/or shellfish [76, 77]. Due to its exclusivity and exceptional properties (fineness, strength, softness, lustre, biocompatibility, etc.), silk is often called the queen of fibres. Still, most commonly, this name is associated with mulberry silk that is obtained by pulling threads from cocoons made by silk moth larvae (silkworms). Historically, it has been cultivated and used not only for making luxury clothes and fashion accessories, but also for making tapestries, wall coverings, floor coverings, parachutes, surgical suture, etc. [78-80]. Advances in the field of biotechnology have opened up possibilities

Fig. 7. Differences between the fibres / fleece depending on the level of expression of the added transgene [65]

for genetic engineering of silk in order to modify and/or improve its original properties and expand its application in e.g. the field of fibre-reinforced composites, the pharmaceutical and cosmetic industries or, to increase its use in the textile and fashion industries through the creation of smart as well as sustainable textiles [78, 80-85]. Genetic modification involves altering the silkworm genome to create improved or entirely new types of silk [83, 86]. After the world's first genetic modification of silkworms in Japan at the National Institute of Agrobiological Sciences (NIAS) in 2002, they managed to develop three lines of transgenic silkworms in 2009 [87]. The first line produced silk threads that emit green, red, or orange fluorescent light. These threads were created by introducing genes into silkworm eggs that promote the generation of fluorescent proteins. Green fluorescence was achieved using genes extracted from jellyfish, while red and orange fluorescence was achieved using genes extracted from corals [88, 89]. The cocoons were smaller and the quality of such genetically modified silk was slightly lower compared to traditional silk. However, fluorescent silk threads have attracted the attention of high-end apparel producers and fashion designers (**Fig. 8.**) [90, 91].The second line of transgenic silkworm yielded an ultra-fine silk thread. By introducing genes for producing especially fine thread into "Haugen silkworms" (a stable crossbreeds for fine silk creation), NARO (Japan National Agriculture and Food Research Organization) [91] developed a silkworm line that produced a thread even finer than that of ordinary "Haugen silkworms" [91-93]. The unique look and feel of fabric made from this type of ultra-fine silk makes it highly attractive to designers and producers of Haute Couture clothing and other fashion products. The third line of transgenic silkworm was realised by introducing genes that bolster cell adhesion and resulted in silk that exhibited high level of cell adhesion. The thread produced by these silkworms is expected to have applications in the field of medicine, e.g. for artificial blood vessels or as a cell culture substrate in the production of artificial cornea and artificial cartilage [83, 93, 94].

Wedding dress produced of the coloured fluorescent silks [90]   Twelve-layered dance costume designed using fluorescent silk [91]

Fig. 8. Clothes from fluorescent silk



Different types of spider` silk [103]   Structural hierarchy in spider silk [98]

Fig. 9. Spider silk

After many years of effort, the silkworm *Bombyx Mori* has been developed into insect systems for which the most advanced genetic technologies are available [83]. The application of these technologies has enabled the sophisticated genetic modification of silkworms to improve their commercial value. One significant step in this direction has been the recombinant production of protein spider silk [95-98].

Spider silk fibres are one of outstanding fibrous biomaterials [99-103]. Due to their remarkable protein sequence and ultramolecular structure (**Fig. 9**. [102]) they possess nature's most exceptional mechanical properties, along with biocompatibility and biodegradability [103]

Spider silk fibres have tensile strength comparable to steel and some silks are nearly as elastic as rubber on a weight-to-weight basis. Through the combination of these properties, silks reveals toughness that is several times higher than that of synthetic fibres like Nylon® or Kevlar® [10, 25, 58, 104, 105]. With such unrivalled combination of strength and toughness, spider silk is the model for creating high performance materials in the area of technical textiles, as well as in the area of sustainable fashion [104, 106-108]. Unfortunately, spiders cannot be farmed at a large scale to meet the commercial demand for spider silks, because of their territorial and cannibalistic behaviours [101, 109, 110]. In order to produce the purified spider silk proteins a variety of heterologous hosts have been explored like e.g. *Escherichia coli*, yeast, alfalfa, potatoes, goats etc. [111]. The purified spider silk proteins can artificially be spun into fibres, but the process is too complex, unpractical, and not so sustainable and, above all, such artificial spider silk fibres can not reach similar mechanical properties as natural spider silk fibres [110-112]. The questions – Why? and What is wrong? arises within scientific community. The answer appears to be in particular conditions of silkworm spinning that, in the case of natural silk, is responsible for finalising ultrastructure of fibres and corresponding properties [113-115]. CRISPR/Cas9 initiated fixed-point strategy used to successfully incorporate spider silk protein genes into the *Bombyx Mori* genome [111]. Using transgenic silkworms with their natural spinning apparatus has proven to be a promising way to spin spider silk-like fibres. The resulting fibres were 100% spider silk, as strong as native spider silks (1.2 GPa tensile strength), could withstand a stretching force of 1299 MPa without breaking, making them several times stronger than nylon. The energy that these fibers could absorb from an impact (toughness) was 319 MJ/m^3, which is six times stronger than Kevlar® [106].This strategy shows the feasibility of using silkworms as a natural spider silk spinner for industrial production of high-performance fibres, the more so transgenic silkworms. If realised in this way, they exhibit normal inheritance of the transgenes.

3. Fibres obtained by genetically modifying microorganisms

BioSteel fibres are genetically modified fibres that imitate the properties of spider silk. They are created using genetically modified microorganisms [117]. Using recombinant DNA methods, researchers managed to produce spider silk proteins through genetic modification of bacteria and yeast, thereby creating the possibility for the production of a large amount of this valuable material [14, 95, 96, 118]. *Escherichia coli*, mostly known as *E. coli*, is widely used bacterium in biotechnology since it exhibits fast growth and a large capacity for the production of foreign proteins. Modified *E. coli* can produce silk proteins that can be used for silk production [14, 119]. The researchers cloned the relevant genes from the spider *Nephila clavipes* and introduced them into the bacteria. Using small and large-scale bioreactors, they were able to produce synthetic spider silk proteins (spidrones). The final product is a silk fibre similar to natural silk [15, 119], and can be used in the textile and fashion industry [96, 98,120] or, more broadly, in the field of biomedicine [82, 119, 121]. The advantage of silk production through *E. coli* is that it can be carried out in laboratory conditions, without the necessity for large amounts of space and resources [14, 119], which contributes to global sustainable development [117, 122].

The term "Biosteel" was first used to describe a recombinant protein material similar to spider silk that was obtained from the milk of transgenic goats and produced by Nexia Biotechnologies [123]. Since the company ceased to exist in 2009, the name was taken over and registered by the company AMsilk, which produced fibres obtained from bacteria through an industrially proven fermentation process [124, 125]. In cooperation with Adidas, AMSilk developed the world's first sports shoes (**Fig. 10.**) made of Biosteel® fibres [125-127]. The Adidas Futurecraft Biofabric prototype shoe features an upper made of 100% Biosteel® fibres. The material offers a unique combination of properties, such as being 15% lighter than conventional synthetic fibres, and has the potential to be the strongest all-natural material. In addition, Biosteel® fibres are 100% biodegradable in a completely natural process [124].

Bacterial cellulose (BC) represents a significant part of the research in the context of genetically engineered/modified fibres for the textile and fashion industries. It is recognized as a new material suitable for a wide range of applications, due to its unique structure, high purity, outstanding mechanical properties and biocompatibility [128]. Various types of bacteria, including the genera *Acetobacter*, *Gluconobacter*, *Komagataeibacter*, *Rhizobium*, *Agrobacterium*, and *Sarcina* [129, 130], can synthesize BC from a wide variety of substrate through fermentation [117, 130]. During this process, bacteria produce and secrete extracellular polymers, which form a cellulose structure with unique properties [130]. The structure and properties of BC can be controlled by varying fermentation conditions, including substrate concentration, fermentation time, temperature, and pH [131] (**Fig. 11.**).

Bacterial cellulose has a wide range of potential applications. Biocompatibility and ability to be formed into different shapes and sizes makes BC an ideal material to be used in the sustainable fashion production [128, 130]. Bacterial cellulose is biodegradable, which further encourages its use as an environmentally friendly material for the textile and fashion industries. Due to its outstanding ability to absorb water, it can also be used in the production of highly absorbent products, such as diapers [131].

Fig. 10. Adidas shoe from Biosteel® fibre [124-127]

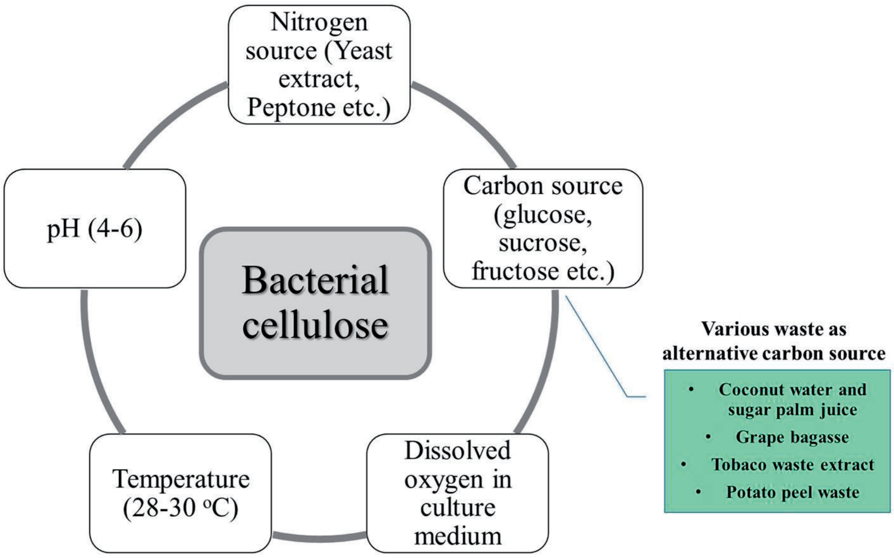


Fig. 11. Factors influencing the production of bacterial cellulose [131]

In addition, properties such as high strength and flexibility, together with excellent wear resistance, make it suitable for the use in the production of various types of textiles and composite materials. Bacterial cellulose also plays a significant role in biomedical applications. Due to its biocompatibility, BC can be used in the production of medical implants, tissue engineering, wound and skin dressings, as well as in drug delivery control [128].

4. Benefits and risks of genetically engineered/modified textiles: Current controversies and perspective

Today, there are already numerous innovative companies and projects [117, 120, 122, 131-137] that have dedicated themselves to the development and commercialization of genetically engineered/modified textiles in advanced biotechnological procedures (**Fig, 12.**) [117, 132].

Such projects offer an alternative to traditional, often resource-intensive, fiber manufacturing processes, promoting the shift to greener and more sustainable textile and fashion industries. Over time, more and more of these pioneering projects have appeared on the market (**Fig. 13.**), bringing with them new materials and technologies that have the potential to radically reshape the textile industry as we know it today [133-136]. At the same time, they not only open up new possibilities in the production of functionalized textiles (designed for use), but also contribute to sustainability and environmental preservation. However, every progress in science and technology, as history has shown, is accompanied by controversies and shortcomings that we unfortunately become aware of after the initial euphoria. Some of the advantages and disadvantages of genetic textiles are summarised in Table 1.

Genetically modified textile fibres and their applications will continue to evolve as technology advances. Collaboration between researchers, industry and regulatory authorities will be the key to successful dissemination of these innovations and their application across various sectors.

Fig. 12. Bio-couture clothes (The first research on bacterial cellulose inside the fashion domain was performed by the British fashion designer Suzanne Lee, for her research project “Biocouture, an initiative on sustainable material”) [117, 132]

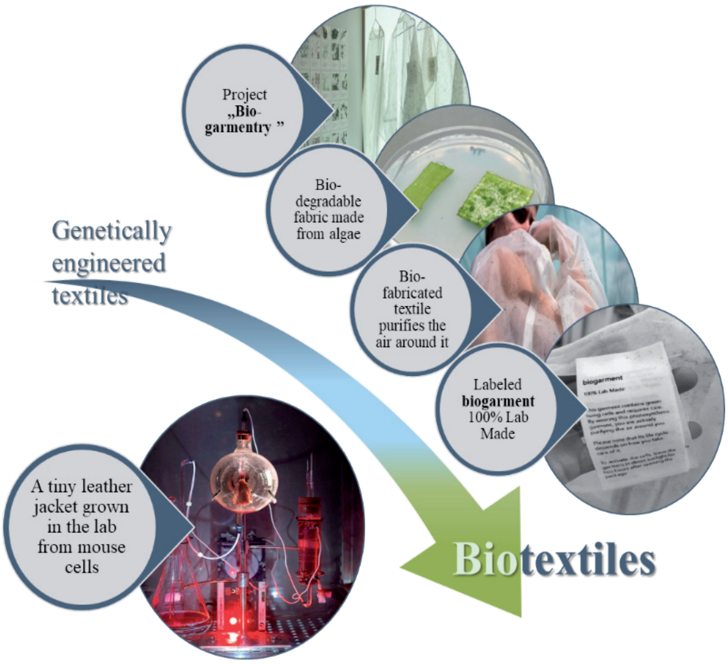


Fig. 13. Textiles for the future

Table 1. Genetically engineered/modified textiles - *pro at contra*

Advantages (*pro*)	Disadvantages (*contra*)
• Increasing fibre yield - genetic modification (GM) of fibre-forming plants can increase fibre yield per unit area of cultivation resulting in: reduction of resources/lands needed for fibre production, reduction of the need to expand agricultural areas, which helps preserve natural habitats and biodiversity. • Improving fibre quality - GM can improve fibre quality, including strength, elasticity, softness, air and water permeability and resistance to abrasion.	• Danger to human health - GM fibres can affect human health, especially if they are used to make clothing and textile products that come into contact, with the skin. • Environmental risks - GM plants can have environmental impacts on non-target organisms such as fish, worms, bees and insects, loss of biodiversity and gene instability. The release of such products and their possible impacts on the environment asks for high environmental biosecurity surveillance in order to reduce or completely eradicate the risk they cause.

Advantages (*pro*)	Disadvantages (*contra*)
• Resistance to diseases and pests - GM fibres can be more resistant to diseases and pests, which reduces the need for pesticides and other chemical means to protect plants. • Resistance to abiotic stress - GM can improve the resistance of plants to stressful conditions, such as drought or high salt concentrations in the soil, thus enabling the production of fibres in different climatic and geographical areas, reducing the need for irrigation and helping to conserve water resources, thus reducing emission of greenhouse gases. • Potential functionalization of fibres - GM fibres can be engineered to incorporate new properties such as resistance to moisture, fire, bacteria or UV radiation. • Potential increase in durability - GM can be used to develop fibres that are even more sustainable than now, thus reducing resource consumption, waste and negative impact on the environment. • Possibly personalized textile products - GM materials can be used to create personalized textile products, such as clothing that adapts to body temperature or the environment.	• Legislation - different laws and regulations in countries around the world regulate the way how genetically modified fibres can be used and traded. There is controversy over how products containing genetically modified fibres should be regulated and labelled. • Monopolization - several large companies hold patents on GMO technology, which can lead to monopolization in the industry and potentially disadvantage small farmers. • Gene flow - The most serious problem associated with gene flow is the loss of biodiversity, and it is often cited as a potential risk. Chances of accidental cross-pollination between GM crops and their wild relatives are very high, making them super-weeds that could be resistant to various herbicides and become difficult to control. There are several examples of gene flow from crops to related weeds, such as *Beta vulgaris, Avena strigose, Brassica napus*, etc. • Increased resistance to antibiotics - GM products might enter the human body through food, vaccines, bacteria or viruses. There is a concern that GM plants with bacterial resistance genes in their genome could act as a source of drug resistance genes for clinically important bacteria. • Allergies - introducing new genes into plants can cause allergies by creating unexpected products (proteins and metabolites) in plants. For example, Bt bacteria can effectively control insects that attack crops. However, the probability of consuming Bt toxins and reacting to mammals that cause allergies is equally high. • Starvation - insects, birds and other animals that feed on certain crops may not consume genetically modified crops due to allergic reactions or toxic products. As a result, large numbers of fauna may face starvation, affecting entire food chains and causing serious threats to ecosystems.

Public opinion and perception are significant barriers to the widespread use of genetically engineered textile fibers. The negative perception of GMOs can in some cases lead to opposition to their use, despite potential benefits. Educating and communicating with the public about the benefits, risks and safety of genetically modified textile fibres can help overcome this challenge.

Complex regulatory and legal frameworks can also be an obstacle to the spread of genetically modified textile fibres. It is necessary to establish clear and comprehensive regulations that enable the research, development and commercialization of genetically modified fibres, while at the same time ensuring the safety and protection of people and the environment.

Ethical concerns about genetic alteration may potentially influence the acceptability of genetically modified textile fibers. The ethical implications of genetic modification need to be carefully considered, including issues of genetic material ownership, potential risks to biodiversity and the environment, and potential social and economic impacts.

Future research and development in the field of genetically modified textile fibres should include an interdisciplinary approach that must combine knowledge and skills from different disciplines, such as biotechnology, genetics, textile technology and engineering, ecology, ethics and sociology.

5. Some final remarks

When studying genetically engineered/modified textiles, it becomes evident that it is still an insufficiently known area of progress, novelties, patents, innovations and creative designs, striving to overcome the deficiency of raw materials, improve the functionality or multifunctionality of textiles and clothing through rapidly renewable raw materials, eco - design and production, with a significant reduction of environmental load and impact on climate change. At the same time, it should be emphasized that along with rapid and often legally and regulatory unsupported progress, there are also controversies and problems that we will become aware later, such as the problem of fast fashion and the desire to fulfil the wishes of customers through a profitable and exclusive product that exist now. Having perceived the advantages and disadvantages of genetic textiles and the positive and negative implications on our life and the environment, instead of a conclusion, it is left to each and every reader to make his/her own judgment and decide, whether he/she will in the future support well-promoted marketing brands and designers that use genetically modified clothing or, perhaps ask himself/herself - What am I wearing? (**Fig. 14.**) or Have we crossed the line?

In ethical reflection, crossing the border implies the ability to deal with the content beyond the boundary. The technological power of today is greater than man's

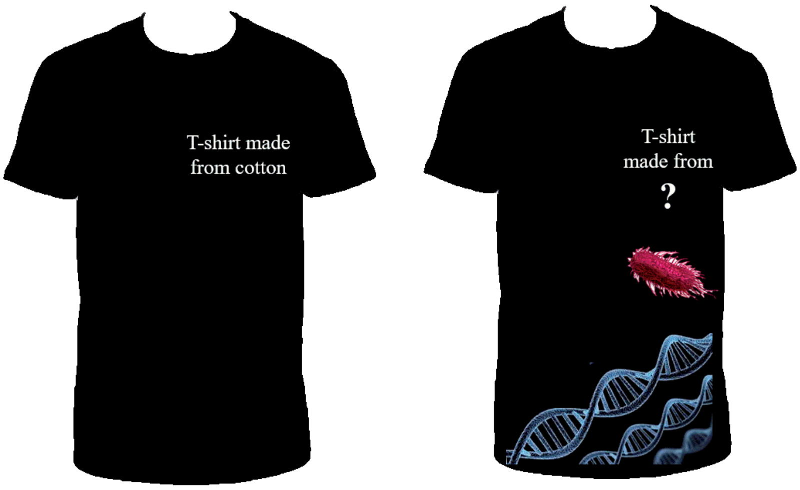


Fig. 14. Simple T-shirt heretofore and these days

reflective power [152], which means that we have to take into account - bioethics. Bioethical situation can be reconstructed and followed in its changes within various forms of social and cultural life as well as at different levels of collective existence. At the same time, it should be kept in mind that all formats of bioethical situation are resulting not only in awareness, but could also historically determine the change in the world-historical situation, which we call an epoch [153]. In other words, genetically engineered/modified textiles become a key point and a sign in which a new epoch appears - the epoch of biotextiles, which will be achieved through synthetic biology in addition to genetic design.

Funding

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The European Community of Textile Research and Innovation Professionals

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Abstract

The European Technology Platform for the Future of Textiles and Clothing (Textile ETP) is the largest European network dedicated to textile research and innovation. Its main objectives are to ensure the long-term competitiveness of the EU textile and clothing industry through collaborative research across national borders in Europe and a rapid translation of research results into industrial innovation.

Keywords: *textiles, innovation, networks, sustainability, cooperation*

1. Introduction

The European Technology Platform for the Future of Textiles and Clothing (Textile ETP) was created in 2004 to match research and innovation needs and priorities of the European industry (represented by companies, associations and clusters) with the knowledge, scientific and technological capacities of universities, research organisation and technology developers. **The aim is to raise the textile competitiveness through intensified collaborative research and innovation.**

Since 2013 the Textile ETP has been established as an international non-profit association under Belgian law with its permanent premises based in Brussels.

Textile ETP counts 300+ member organisations from industry, research and higher education with a **total of over 1200 registered individual experts.**

According to the current strategy, adopted in 2018, the Textile ETP acts as Strategic Connector, Think Tank and Advocate, and Intelligence and Funding Access Provider to all our members across Europe. Therefore, the main activities of the Textile ETP revolve around Networking, Learning and EU Funding.

Fig. 1. Dr. Marina Crnoja-Ćosić –the newly appointed President of Textile ETP und Lutz Walter – ETP Secretary General

Networking

The Textile ETP is known as **the biggest networking organisation for textile research and innovation in Europe**. Through online member platform, and regular networking events, conferences and webinars, textile inovation experts from across Europe get to build connections and start collaborations for mutual benefit (Fig. 2.)

Fig. 2. Textile ETP President Marina Crnoja-Ćosic delivered a keynote address at the official kick-off event of 'New Textile Ecosystems' (NEWTEXECO), September 21st 2023, Arnhem, the Netherlands

All events and communication channels are also at the disposal of our members to disseminate the results of their collaborative research or innovation projects.

EU Funding

The Textile ETP provides its members with timely and focussed access to information on EU funding opportunities. The **TEPPIES programme** is the members-only brokerage system to facilitate access to EU funding by supporting members in forming strong project consortia for successful funding applications.

Together with full members **EURATEX, TEXTRANET, NETFAS,** and **EU-TEXTILE2030**, we also engage with European policymakers to inform them about textile innovation potential and research needs to have them reflected in EU policies and funding programmes.

Learning

The Textile ETP organises year-long **Masterclasse**s and **Expert Communities** on various topics.

- Monthly **Masterclass** webinars provide in-depth knowledge and understanding of research and **innovation** trends related to specific textile subjects.
- Expert **Communitie**s offer an excellent opportunity to network with companies, researchers and technology developers, and to help develop new partnerships. They also provide opportunities to participate in key thematic webinars and in-person meet-ups.

Strategic roadmap - Ready to Transform

The Textile ETP is based on three pillars, which at the same time represent the three crucial elements of the long-term vision that will exploit these strengths while at the same time benefiting from general societal and economic trends:

- A move from commodities towards specialty products from high-tech processes along the entire value chain from fibres to final products with highly functional, purpose-targeted properties
- The establishment and expansion of textiles as a material of choice in many sectors and application fields
- A move toward a new industrial era characteristised by customisation, personalisation as well as flexible, on-demand production.

The Textile ETP collaborates with its members to generate strategic roadmaps or themed opinion and position papers that address the primary goals of the “European Textile Strategy.”

The latest comprehensive roadmap entitled “**Ready to Transform**” was adopted in 2022.

Four strategic Innovation Themes have been singled out as particularly impactful for the successful and rapid transformation of the European textile ecosystem.

- Smart, high-performance materials for new growth markets
- Digitised textile materials, products, manufacturing, supply chains and business models
- Durable, circular and biobased materials and processes
- Safe, low footprint products, processes and responsible supply chains.

For each theme, several research and innovation topics and subtopics are defined followed by a listening of concrete research and innovation targets and promising technology approaches.

The implementation of the **EU Strategy for Sustainable and Circular Textiles** must be accompanied by a policy framework that fosters innovation and skills development at all levels. An investment program is required to bring about the expected massive systematic change. On this path of transformation the textile and clothing, industry in Europe is facing great challenges and opportunities,

Textile ETP acts as a reminder of the collaborative effort required to transform the industry and have a worldwide influence.

It supports national initiatives, e.g. French Re_fashion with a mission “For a 100% circular textile industry” (Fig. 3.).

Fig. 3. Textile ETP attended the event Re_fashion Day in Paris, October 2023

Sustainability, innovation and cooperation are essential parts of these developments. The ETP continuously offers a platform on which research and development in the textile sector in Europe are optimally networked. This will make it possible to bring innovations to the market faster and more efficiently and to implement the EU textile strategy. By doing this, the European textile sector will remain competitive.

Sources: ETP internal documents, ETP Roadmap “Ready to transform”

Interested in learning more? Contact us!

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ETP
Fibres
Textiles
Clothing
EUROPEAN TECHNOLOGY PLATFORM

30th anniversary of the Croatian Academy of Engineering

The Croatian Academy of Engineering (HATZ) held its 47th regular annual assembly on June 30, 2023, at the University of Zagreb Faculty of Electrical Engineering and Computer Science. The regular annual assembly also served as the focal point for HATZ's 30th anniversary celebrations.

The Croatian Academy of Engineering was founded on January 19, 1993 and registered with the Ministry of Justice and Administration of the Republic of Croatia on January 25, 1993. In 1997, Hrvatska akademija tehničkih znanosti changed its name to the Akademija tehničkih znanosti Hrvatske. This was done on the basis of the Act on CASA and the decision of the Parliament of the Republic of Croatia on June 27, 1997. In 2009, the Academy was granted the status of a scientific organization. It was founded as a non-governmental, independent, non-partisan and non-profit association of outstanding, experienced and proven scientists and engineers from technical and biotechnical professions. Since then, the Academy has been led by six presidents: Academician Josip Božičević (1993–1997), Prof. Juraj Božičević, Ph.D. (1997–2003), Prof. Emeritus Zlatko Kniewald, Ph.D. (2003–2009), Prof. Stanko Tonković, Ph.D. (2009–2013), Prof. Vladimir Andročec, Ph.D. (2013–2022) and Prof. Vedran Mornar, Ph.D. (2022-2026).

Since October 2000, the Academy has been a member of the International Council of Academies of Technical Sciences based in Washington, USA (CAETS - International Council of Academies of Engineering and Technological Sciences), and since January 2005 it has been an associate member of the European Council of Applied and Technical Sciences based in Paris, France (Euro-CASE – European Council of Applied Sciences and Engineering). The Academy has been a permanent member of Euro-CASE since May 2009.

In the 30 years of its existence, the Academy has had a significant impact on the development of Croatian technical and biotechnical sciences through the organization of numerous scientific and professional meetings, round tables, forums and public lectures, through the work of its members published in the Academy's Engineering Power newsletters and the Annual of the Croatian Academy of Engineering, through the realization of scientific and professional projects and cooperation with the Croatian business community.

As a contribution to the activities of the Academy and at the same time as an introductory event to the activities related to the regular annual assembly and the celebrations of the 30th anniversary of the Academy, a scientific and professional conference "Croatian Academy of Engineering - cohesive factor of technical and biotechnical sciences and the Croatian economy" was held. The conference was attended by the distinguished members of the Academy. Prof. Franjo Jović, Ph.D., Prof. Saša Zelenika, Ph.D., Prof. Edouard Ivanjko, Prof. Božidar Šantek, Ph.D., Prof. Meho Saša Kovačević, Ph.D. and Prof. Neven Duić, Ph.D. gave lectures on a number of topics in the field of technical and biotechnical sciences, the main aim of which was to show the connection between the scientific and professional activities of the Academy members and the Croatian economy.

Prijedlog Dnevnog reda (1)

- **Obavijest o preminulim članovima Akademije (svibanj 2019. – lipanj 2022.)**

1. Prihvaćanje Zapisnika 46. (elektroničke) skupštine HATZ-a i rezultati glasanja o izborima u Akademiji (12.-19.6.2023.)
(Izvjestitelj: prof. dr. sc. V. Mornar)

2. Primanje na znanje zapisnika 3. sjednice Predsjedništva HATZ-a održane između 45. i 47. skupštine HATZ-a:
(Izvjestitelj: prof. dr. sc. V. Mornar)

3. Izvještaj o financijskom stanju HATZ-a
- *Izvještaj o financijskom stanju na dan 23.6.2023.*
- *Individualne članarine – stanje na dan 23.6.2023.*
- *Članarine podupirućih članova – stanje na dan 23.6.2023.*

(Izvjestitelj: prof. dr. sc. V. Mornar)

47th regular annual assembly of the Croatian Academy of Engineering

In the opening speech at the opening of the regular annual assembly, the President of the Academy, Prof. Vedran Mornar, Ph.D., emphasized the role and importance of the Academy in the national context.

In addition to the topics of the Academy's regular activities, which are dealt with in the working part, the jubilee Annual of the Croatian Academy of Engineering - Jubilee Annual 2022-2023, which is dedicated to the 30th anniversary of the Academy, was also presented. In addition to presenting the historical development, organization and activities of the Academy, the Annual contains the CAETS Energy Report for 2022 and a list of all full and associate members of the Academy.

During the ceremonial part of the regular annual assembly, diplomas were presented to the new associate members, emeritus members, international members and supporting members of the Academy, as well as diplomas to the winners of the Academy Awards for 2022, who had previously been confirmed by the Academy's Presidency and the Assembly of the Academy. Thus, the status of a new associate member of the HATZ was obtained by:

- Prof. Borislav Miličević, Ph.D. in the Department of Bioprocess Engineering
- Assoc. Prof. Josip Lörincz, Ph.D., Prof. Tomislav Matić, Ph.D., Prof. Stjepan Stipetić, Ph.D. and Assoc. Prof. Petar Šolić, Ph.D. in the Department of Electrical Engineering and Electronics
- Assoc. Prof. Goran Krajačić, Ph.D., Prof. Sandro Nižetić, Ph.D. and Prof. Hrvoje Pandžić, Ph.D. in the Department of Energy Systems
- Assoc. Prof. Mario Bačić, Ph.D., Prof. Željana Nikolić, Ph.D., and Prof. Damir Varevac, Ph.D. in the Department of Civil Engineering and Geodesy
- Assoc. Prof. Daniel Hofman, Ph.D. in the Department of Information Systems
- Prof. Domagoj Vrsaljko, Ph.D. in the Department of Chemical Engineering
- Prof. Borna Abramović, Ph.D. and Assoc. Prof. Robert Maršanić, Ph.D. in the Department of Transport
- Prof. Nastia Degiuli, Ph.D. in the Department of Mechanical Engineering and Naval Architecture
- Prof. Nikola Mišković, Ph.D. in the Department of Systems and Cybernetics
- Assoc. Prof. Sandra Flinčec Grgac, Ph.D. in the Department of Textile Technology

Prof. Vedran Mornar, Ph.D. and Prof. Tanja Pušić, Ph.D. at the awarding ceremony

The following were appointed as new emeritus members of the Academy:

- Prof. Predrag Horvat, Ph.D. in the Department of Bioprocess Engineering
- Prof. Emeritus Ante Mihanović, Ph.D. in the Department of Civil Engineering and Geodesy
- Prof. Mladen Šercer, Ph.D. in the Department of Mechanical Engineering and Naval Architecture.

The status of a new international member has been granted to:

- Prof. Peter Virtič, Ph.D. in the Department of Electrical Engineering and Electronics
- Marina Crnoja-Ćosić, Ph.D. in the Department of Textile Technology.

The following companies have acquired the status of a new supporting member:

- Aircash d.o.o.
- Jadrolinija, Rijeka

The prizes of the Croatian Academy of Engineering for the year 2022 were awarded at the assembly as follows:

- The annual "Rikard Podhorsky" prize was awarded to: Prof. Alen Jugović, Ph.D., Assoc. Prof. Stjepan Picek, Ph.D. and Prof. Tanja Pušić, Ph.D.
- The prize for young scientists "Vera Johanides" (for science) was awarded to Assist. Prof. Mirna Gržanić, Ph.D., Nino Horvat, Ph.D., Katarina Mužina Ph.D. and Luka Pravica, Ph.D.

The funds for the HATZ prizes were provided by the following companies: Xellia d.o.o. Zagreb and Centar za vozila Hrvatske d.d., Zagreb

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