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# Application of Taguchi method to investigate the effect of temperature, heating time, concentration and particle size on improved gel spinning process of UHMWPE

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## REZUMAT – ABSTRACT

### Aplicarea metodei Taguchi pentru a investiga efectul temperaturii, al timpului de încălzire, concentrației și dimensiunii particulelor asupra procesului de filare îmbunătățită cu gel a UHMWPE

În cadrul acestui studiu, filamentele UHMWPE au fost extrudate utilizând procedeul de filare cu gel pe bază de terpen nou dezvoltat. Modelul experimental factorial fracțional Taguchi a fost conceput pentru a studia impactul diferiților factori asupra tenacității fibrelor. Filamentele extrudate au fost caracterizate prin luarea în considerare a tenacității filamentelor ca răspuns. Extrudarea a fost efectuată folosind un procedeu de filare cu gel pe bază de terpen dezvoltat de autor, care utilizează terpen ecologic (ulei de portocală) în locul substanțelor petrochimice utilizate în extrudarea convențională a UHMWPE. Au fost utilizați patru parametri de procesare selectați, iar efectul lor asupra tenacității filamentelor rezultate a fost evaluat utilizând metode statistice standard. S-a constatat că timpul de încălzire și concentrația exercită un efect semnificativ asupra tenacității filamentelor. În plus, interacțiunea dintre concentrație și dimensiunea particulelor, temperatură și concentrație, timpul de încălzire și concentrație au indicat un efect major asupra răspunsului.

Cuvinte-cheie: UHMWPE, terpen, ecologic, tenacitate, filare cu gel

### Application of Taguchi method to investigate the effect of temperature, heating time, concentration and particle size on improved gel spinning process of UHMWPE

In this research UHMWPE filaments were extruded utilising newly developed terpene based gel spinning process. Taguchi's fractional factorial experimental design was implemented to study the impact of different factors on the tenacity of the fibres. Extruded filaments were characterized by taking filament tenacity as response. The extrusion was carried out utilising terpene base gel spinning process developed by the author reported previously, which uses environmentally friendly terpene (orange oil) instead of petrochemicals used in the conventional extrusion of UHMWPE. Four selected processing parameters were used and their effect on the tenacity of resultant filaments was assessed using standard statistical methods. It was found that the concentration and heating time exerts significant effect on the tenacity of filaments. In addition, interaction between concentration and particle size, temperature and concentration, heating time and concentration indicated major effect on the response.

Keywords: UHMWPE, terpene, environmentally friendly, tenacity, gel spinning

## INTRODUCTION

Ultra high molecular weight polyethylene (UHMWPE) has very high melt viscosity; conventional methods of extrusion cannot be applied for the production of fibres. One of the earliest methods used to produce the UHMWPE fibres was surface growth method. Contrary to other manufacturing methods of UHMWPE which consists of at least two or more stages [1]. In surface growth method fibres are produced through couette flow of a dilute solution. Fibrous crystals grow on the surface of the rotating internal cylinder. Fibres are produced by pulling out the growing fibrous crystal [2–6]. There are number of techniques to achieve the surface growth; Zwijnenburg developed one of the simplest method [3]. The technique proposed by Zwijnenburg composed of Teflon rotor and beaker. The Teflon rotor is placed in the middle of the beaker. A tube is connected to the beaker through which seed yarn is passed on to the cylinder. The seed yarn is pulled by the take up roller. Tension measuring

device is installed to measure the tension of the yarn between tube and take up roller. UHMWPE is dissolved in the p-xylene solution at 130°C. The rotor is set in motion. The seed yarn is introduced into the system through the tube. The rotation of the rotor catches the seed yarn. The rotation brings the yarn in contact with the rotor which initiates the surface growth. The seed yarn is pulled out and wound on the take up roller. Winding speed of take up roller is adjusted according the rate of fibrous crystal growth. The process continues for days [1]. The same design was used by Braham and Keller they managed to achieve high modulus [2]. Kavesh and his co-workers developed a slightly different surface growth method [4]. Prevorsek made comparison between surface growth and other methods [7]. He argued that the fibres produced by the surface growth apparatus have exceptionally high strength and modulus [8], which is due to the reduced number of chain folding during this process compared to melt spinning process.

However this process is only suitable for producing monofilament for fishing lines, dental floss and surgical sutures. The process is not capable of producing fine cross section multifilament [7]. Additionally the control of fibre diameter is also a challenge in surface growth method.

An alternate to the surface growth is gel spinning process. Multifilament with predetermined thickness can be produced by gel spinning in contrast to surface growth. The control of fibre thickness is easier to control in gel spinning. Compared to UHMWPE normal polyethylene contains less oriented molecules which results in weaker fibres. To impart strength in fibre the molecule chains are stretched to orientate and crystallise the chain along the axis of the fibres. Stronger fibres can only be achieved with long enough polymer chains to give rise to chain interactions. Hence to achieve very strong fibres, ultra high molecular weight PE is used as starting material. UHMWPE contains very long polymer chains; however the longer polymer chains give rise to chain entanglements. These chain entanglements affect the melt flow index of the UHMWPE making it impossible to be extruded by conventional extrusion methods. Furthermore, the chain entanglements limit the ability of the fibres to draw. Drawing plays a vital role in achieving the high strength fibres by orientating the polymer chains along the axis of the fibre. Gel spinning process is used to overcome these problems. In the first of step of the gel spinning process polymer is dissolved in a solvent to form slurry. This results in the disentanglement of polymer chains, hence making extrusion possible. In the second step of the process the slurry is extruded through a spinneret to produce gel like monofilament or multifilament depending on the spinneret. The disengagement of chains during the extrusion process enables the extruded filaments to be drawn to very high draw ratios. Higher draw ratio make polymer chains highly oriented which results in the fibre with higher strength and modulus. Gel spun fibres can attain an orientation of more than 95% and up to 85% crystallinity which give UHMWPE fibres superior properties [9].

In conventional gel spinning process petrochemicals are used to dissolve polymer followed by extrusion of polymer solution [10]. Extrusion of polymer solution produces gel like fibres which contains both polymer and petrochemical solvent. In the second stage gel like fibres are treated with extraction solvent to remove petrochemical solvent. In present work gel spinning was carried out utilizing terpene based gel spinning process developed by author [11] instead of petrochemicals used in conventional gel spinning of UHMWPE. The schematic diagram of newly developed process is shown in figure 1.

There are many factors that influence the gel spinning process. In this research an experimental design was implemented to find out the effect of the concentration, temperature, heating time, particle size and their interactions. Taguchi's method of statistical experimental design was used to identify the effect of processing factors and interaction between various

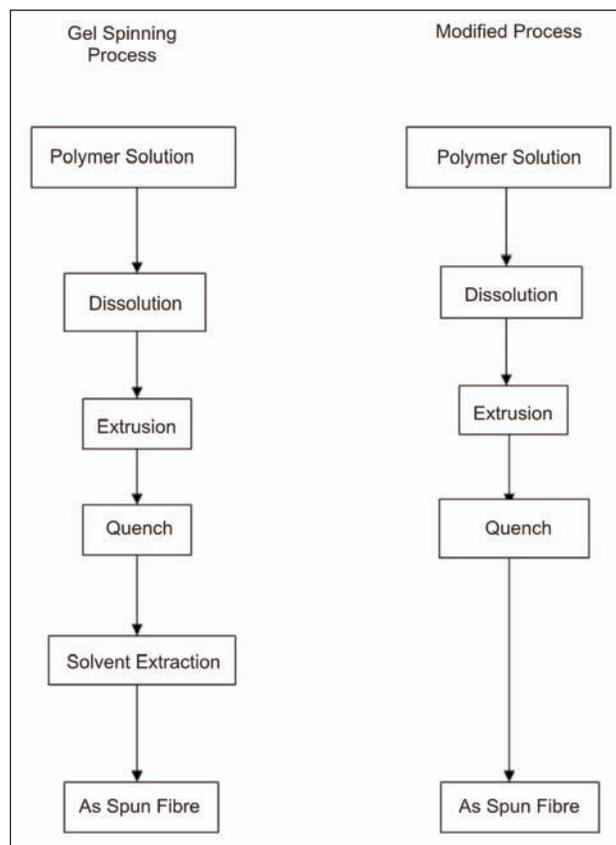


Fig. 1. Comparison between conventional and modified gel spinning process

factors. Taguchi's method of quality control is based on orthogonal array experiments that help in optimizing the process.

## EXPERIMENTAL WORK

### Materials and methods

Ultra high molecular weight polyethylene Gur 4120 with average molecular weight of  $5.0 \times 10^6$  supplied by the Ticona UK Ltd was used in this work. The polymer density was  $0.93 \text{ g cm}^{-3}$ , with melting point in the range of  $130^\circ\text{C} - 135^\circ\text{C}$ . Orange oil (terpene) was sourced from Sigma-Aldrich. The boiling point of terpene was  $176^\circ\text{C}$ . 2,6-Di-tetra-butyl-4-methylphenol antioxidant was also supplied by Sigma-Aldrich.

Samples were prepared as reported previously by the author [11]. Tensile tests were carried out on Instron 3345. Samples were conditioned at  $20 \pm 2^\circ\text{C}$  and  $65 \pm 2\%$  relative humidity for 24 hours before testing. ASTM standard D 3822 was followed to conduct testing.

### Experimental design

A two level four variables fraction factorial experimental design was used for the evaluation of variables effect on the as spun fibre strength. Polymer particle size, temperature, heating time and concentration were chosen as variables. Instron tensile tester was used for the testing of as spun fibre strength. The strength of the as spun fibres was used as a response in the experimental design.

Initial experiments were carried out to set the levels of the experimental design. Due to the novelty of the process no data was available in literature. UHMWPE polymer powder SEM images showed the particle size of the polymer differ greatly. Particles of having size of 150 micron and greater but less than 250 microns were set as level one and particles with greater than 250 microns were set as level two.

Experiments were carried out to find the gelation temperature of polymer in terpene solvent. The gelation starts at 120°C therefore level one for temperature was set at 130°C. Wide range of temperatures had been reported previously for the preparation of solution in different solvents. Since terpene was used in this work it was decided to set the level two at 150°C to avoid the excessive evaporation of the terpene during solution preparation process since terpene boils at 176°C. For setting the heating time there were significant differences in the available literature. Solvent made from decalin were reported to be heated for 40 min while solvent made from paraffin solvent had been reported to be heated for 48 hours. The level one for heating time was set at 1 hr and level two at 3 hr. Polymer concentration plays vital role in the final strength of the fibres as reported by several researchers. Experiments conducted by researcher with decalin reported fibre preparation with as low polymer concentration as 0.5% [12]. However solution made with 0.5% polymer concentration in paraffin was not extrude able [13]. Experiments were carried out by preparing 0.5%, 1%, 2% and 3% solution in terpene and extruded on the

ram extruder to find out the lowest limit of concentration for the preparation of fibre. It was observed the solution having concentration of 0.5% and 1% were too thin to be extruded on the ram extruder. Gel fibres were successfully prepared with 2% concentration and collected on the bobbins but after the removal of the solvent by air drying fibres lost their shape and stuck together. Fibres extruded from 3% solution kept their fibre form. Hence level for the polymer concentration was set at 3% and level at 5%. The experimental design and levels are shown in table 1 and table 2.

## RESULTS AND DISCUSSION

Polymer concentration of the solution affected the strength of the as spun fibres significantly as shown in the pareto chart (figure 2). Combination of polymer concentration and particle size showed inverse relation to fibre strength. Combination of concentration and heating time improves as spun fibre strength considerably followed by heating time and combination of temperature and concentration. Particle size and temperature showed negligible effect.

Table 1

Factors	Low level	High level
Polymer particle size (A)	150 < X < 250 micron	Y < 250 micron
Temperature (B)	130°C	150°C
Heating time (C)	1 hr	3 hr
Concentration (D)	3%	5%

X represents the size of the particle which are greater than 150 micron but smaller than 250 microns.

Y represents the particles size greater than 250 micron.

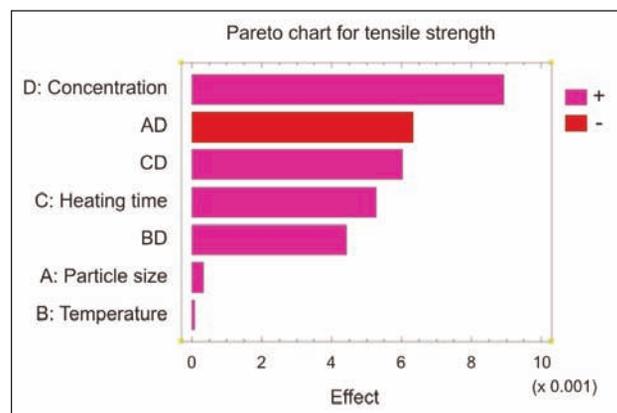


Fig. 2. Effects of different factors on the tensile strength of fibre

Effect of main factors on the response (tensile strength) is shown in figure 3. Particle size showed no significant effect on the strength of the as spun fibres. Changing of particle size levels brought no

Table 2

E.No.	R.O	Polymer particle size [µm]	Temperature [°C]	Heating time [hr]	UHMWPE concentration [%]
1	3	X	130	1	3
2	8	X	130	3	5
3	4	X	150	1	5
4	7	X	150	3	3
5	2	Y	130	1	5
6	5	Y	130	3	3
7	1	Y	150	1	3
8	6	Y	150	3	5

R.O. – Random order in which the experiment was conducted.

E.No. – Experiment number.

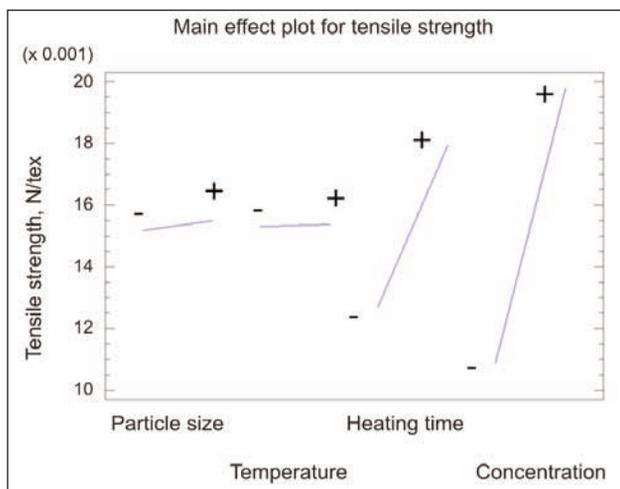


Fig. 3. Effects of main factors level on tensile strength (-ve represents low level, +ve represents high level)

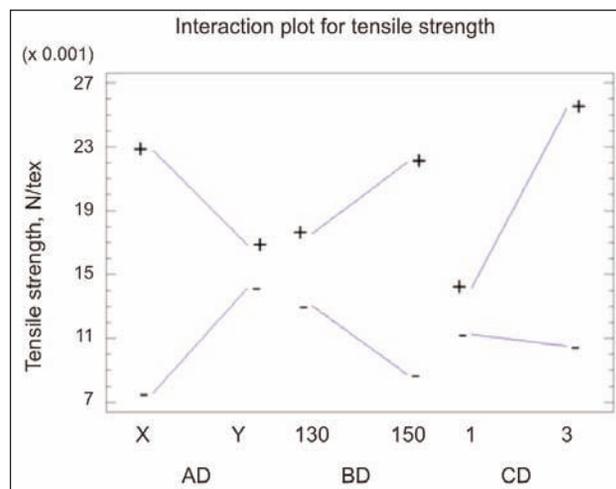


Fig. 4. Interaction Plot for tensile strength (-ve indicates low level of D and +ve indicates high level of D)

significant change in response. This could be because the irrespective of the size of particle it gets dissolved into the solution and forms homogenous solution. Furthermore the solution was observed to have no undissolved particle indication homogenous mixing of the particles in the solvent. Both level of temperature indicated no change on the response. This could be due to the lower melting of the UHMWPE i.e. 130°C to 135°C at both level of temperature UHMWPE got completely dissolved into the solvent. However changes in the level of heating time significantly affects the response. Longer heating time resulted in stronger fibres in contrast to the weaker fibres achieved by reduced heating time.

Concentration of the polymer in the solution significantly affected the strength of the fibre indicated by the vertical line in figure 3.

The interaction plot is shown in figure 4. It indicates interaction between concentration (D) and Particle size (A). Lower level of D and A results in weaker fibres. High level of A along with lower level of D results in stronger fibres compared to previous combination, however high level of D and low level of A

results in the maximum response values. Positive interaction was indicated by Temperature (B) and D. Lower levels of D and B yield lower response while high levels of both resulted in higher response. Heating time (C) and D showed very high positive interaction. Higher values of response were achieved by with higher levels of C and D.

## CONCLUSION

The results represented that concentration has significant effect on the strength of the fibres along with combination of heating time and particle size. Results also showed strong interactions between polymer concentration and particle size also between temperature and concentration. Optimal strength can be achieved by keeping concentration and heating time at high levels. However further investigation would be needed to find out the limitation of these factors. Since higher concentration of polymer will result in the polymer chain entanglement hence will result in increased viscosity of the solution. Higher viscosity makes extrusion difficult.

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# Effect of process variables on the properties of dual-core yarns containing wool/elastane

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## REZUMAT – ABSTRACT

### Efectul variabilelor de proces asupra proprietăților firelor cu miez dublu care conțin lână/elastan

Denimul, cu un număr mare de utilizatori, indiferent de vârstă, sex și statut social, a fost unul dintre cele mai importante produse pentru sectorul îmbrăcăminte. Cererea de țesătură denim s-a diversificat odată cu schimbarea stilului de viață al consumatorului. Producătorii de denim dezvoltă tehnici și materiale de producție alternative prin aplicarea de noi cercetări pentru a se adapta cerințelor consumatorilor. Unul dintre materialele alternative utilizate în structura țesăturilor denim este firul cu miez dublu. Firul cu miez dublu este fabricat cu mașina de filat cu inele modificată, pentru a beneficia de proprietățile miezului dublu. În acest studiu este investigată influența unor parametri de producție, cum ar fi: nivelul de torsiune, etirarea lanai și etirarea elastanului asupra proprietăților firelor cu miez dublu care conțin lână/elastan. Rezultatele au arătat că nivelul de torsiune este un parametru important pentru valorile neuniformității, pilozității, tenacității și alungirii firelor cu miez dublu. În plus, etirarea lanai este un parametru semnificativ pentru valorile pilozității și alungirii la rupere. De asemenea, s-a observat că variația nivelului de etirare al elastanului afectează valorile tenacității și alungirii firelor cu miez dublu.

Cuvinte-cheie: filat cu miez, fir cu miez dublu, fir de lână, elastan, țesătură denim

### Effect of process variables on the properties of dual-core yarns containing wool/elastane

The denim, having a large customer base irrelevant of age, gender and social status limitation, has been one of the most important products for the garment sector. Denim fabric demand has diversified with the changing consumer's sense of life day by day. The denim manufacturers develop alternative production techniques and materials by turning towards new researches in order to adapt to consumer demands. One of the alternative materials, which are used in denim fabric structure, is the dual-core yarns. The dual-core yarn is manufactured through the modified ring-spinning machine in order to benefit at the same time from the properties of two core components. In this study the influence of some production parameters such as twist level, wool draft and elastane draft on the properties of dual-core yarns containing wool/elastane is investigated. The results indicated that the twist level is significantly effective parameter for the unevenness, hairiness, tenacity and elongation values of dual-core yarns. In addition, wool draft is significantly effective parameter for hairiness and breaking elongation values. It was also observed that variation of elastane draft level affects tenacity and elongation values of dual-core yarns.

Keywords: core-spun, dual-core yarn, wool yarn, elastane, denim fabric

## INTRODUCTION

Denim fabric has become a crucial part of fabric production sector since it has been used extensively by people of all ages, classes and genders. Moreover, customers' requirement for the aesthetic and functional performance of denim is increasing with each passing day, which has led to the use of different types of materials and finishing treatments.

Stretch denims are products used for function and at the same time fashion as well. The stretch property is gained with core spun wefts which contain elastane filament in denim fabric structure. Core-spun yarn spinning is a process defined as the twisting the staple fibres around the core yarn, which is either filament or staple spun yarn [1]. The produced yarn has the sheath-core structure. Elastane filament is a manufactured filament in which the filament-forming substance is along chain synthetic polymer comprised of at least 85% by weight of segmented polyurethane [2].

Core spun yarns containing elastane which has low modules, gain easy stretch properties to the denim fabrics. However denim fabric consumers also demand the high recovery power and low fabric growth besides easy stretch properties. In order to meet the consumer demand another core yarn which has high tension modules compare to the elastane, is required. Hereby, PET, PA, T400, PBT etc. and elastane are usually used together as the core part in order to benefit from the properties of two different core components at the same time. For the production of this kind of multi-component core-spun yarn, PET, PA, T400, PBT etc. (1st core) and elastane (2nd core) are fed separately to the drafting unit of ring spinning machine and this system is called dual-core method [3].

There are limited studies about dual-core yarns in the literature. Material content and production parameters are two important factors which affect the performances of dual-core spun yarns [4–8]. In order to give different performance characteristics to denim

fabrics, various core spun yarns can be used in denim fabric structure. In this study, dual-core spun yarns which consist of wool yarn as the first core and elastane filament as the second core were produced to benefit tactile and thermal effects of wool fibers and stretch effects of elastane filament at the same time, with a novel approach. The purpose of this study was to examine the influence of production parameters such as twist level, wool draft and elastane draft on the various properties of this novel dual-core spun yarns.

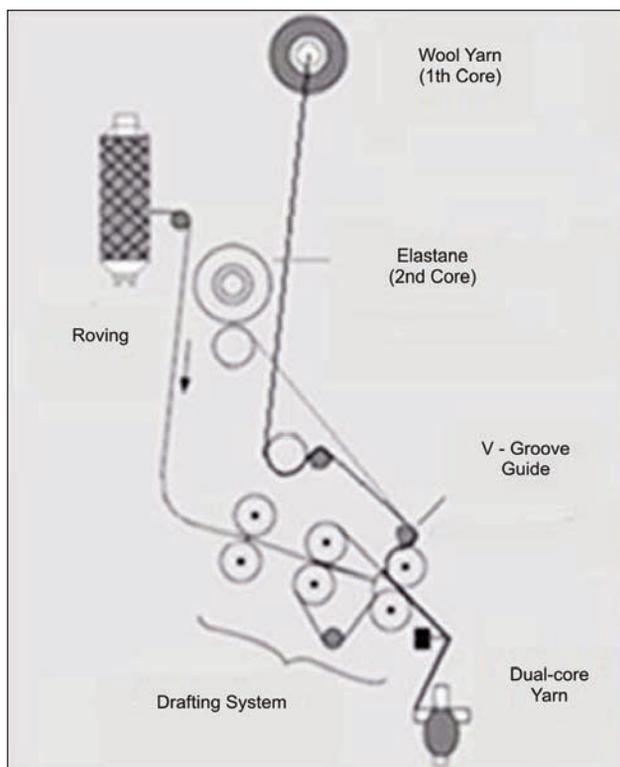
## MATERIALS AND METHODS

27 different types of dual-core spun yarns were produced in modified ring frame machine by passing a Nm 80/1 wool yarn (S-twist with 800 T/m) and a

It was investigated the effects of production parameters on the yarn unevenness, hairiness, tenacity, and elongation values of dual-core yarn samples. Yarn unevenness and hairiness were measured on Uster Tester 5 with the testing speed of 400 m/min throughout 1 minute. Yarn tenacity and breaking elongation were determined on UsterTensorapid 4 Tester. For each yarn sample, five tests were performed and the averages were reported. The tests, samples were conditioned at least for 24 hours in an atmosphere of  $20 \pm 2^\circ\text{C}$  and  $65 \pm 2\%$  relative humidity in order to adjust humidity balance.

## RESULTS AND DISCUSSIONS

The obtained results of dual-core yarns were evaluated statistically for significance in differences using



a



b

Fig. 1. (a) Schematic diagram of dual-core yarn production method and (b) Drafting system-V groove guide

78 dtex elastane filament through the front rollers which form the core and cotton fibers through normal roller drafting system which form the outer cover of yarn known as sheath as seen in figure 1.

Scanning electron microscope (SEM) images were taken in order to better visualize the morphological structure of the dual core yarns. The surface morphology of the dual core yarn was studied employing a ZEISS EVO scanning electron microscope (SEM) in VP mode operating with an accelerating voltage of 25 keV. The core (wool yarn and elastane filament) and cover (cotton fibers) parts of dual-core yarn can be seen in figure 2. Codes and production parameters of the dual-core yarn samples were summarized in table 1.

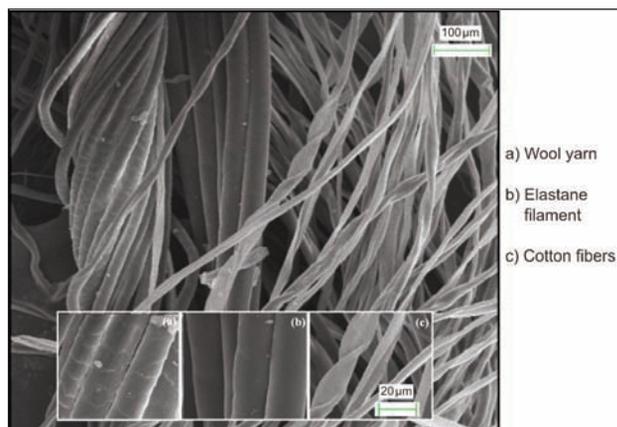


Fig. 2. SEM image of the dual-core spun yarn

Table 1

Yarn code	Twist level [T/m]	Wool draft	Elastane draft
1	585	1.01	3.3
2	585	1.01	3.5
3	585	1.01	3.8
4	585	1.03	3.3
5	585	1.03	3.5
6	585	1.03	3.8
7	585	1.05	3.3
8	585	1.05	3.5
9	585	1.05	3.8
10	670	1.01	3.3
11	670	1.01	3.5
12	670	1.01	3.8
13	670	1.03	3.3
14	670	1.03	3.5
15	670	1.03	3.8
16	670	1.05	3.3
17	670	1.05	3.5
18	670	1.05	3.8
19	750	1.01	3.3
20	750	1.01	3.5
21	750	1.01	3.8
22	750	1.03	3.3
23	750	1.03	3.5
24	750	1.03	3.8
25	750	1.05	3.3
26	750	1.05	3.5
27	750	1.05	3.8

three-way replicated analysis of variance (ANOVA) and the means were compared by conducting Student Newman-Keuls (SNK) tests at a level of 0.05 using SPSS statistical package. Table 2 shows the SNK test results for unevenness hairiness and tensile

Table 2

	CVm [%]	Hairiness [H]	Tenacity [cN/tex]	Elongation [%]
<b>Twist level</b>				
585 T/m	14.87a	8.26a	7.65a	10.35a
670 T/m	14.56ab	7.61b	7.88b	10.48ab
750 T/m	14.16b	6.62c	8.05b	10.73b
<b>Wool draft</b>				
1.01	14.72a	7.61a	7.75a	10.14a
1.03	14.55a	7.50a	7.90a	10.46b
1.05	14.31a	7.38b	7.93a	10.95c
<b>Elastane draft</b>				
3.3	14.46a	7.46a	7.64a	10.03a
3.5	14.46a	7.50a	7.82a	10.64b
3.8	14.66a	7.53a	8.12b	10.89b

properties of dual-core yarn samples. In the interpretation of SNK results, abbreviations a, b, c, d, and e represent factor level; factor levels that have the same letters are not different from each other at a significance level of 0.05 (table 2).

Figure 3 shows the mean values yarn unevenness values of dual-core yarns produced with different twist level, wool draft and elastane draft. According to ANOVA test results, only the twist level ( $P_T=0,023$ ) was significant factor for yarn unevenness.

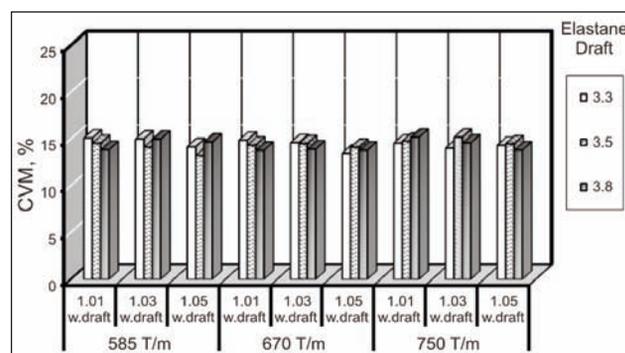


Fig. 3. Yarn unevenness versus twist level for comparable 1.01, 1.03, and 1.05 wool draft with 3.3, 3.5, and 3.8 elastane draft

From the SNK test results the differences between yarn unevenness values for 585 and 750 T/m twist level were found to be statistically significant. It was observed that there is decreasing trend in the unevenness values of yarn samples as twist level increases. This can be explained by the fact that the staple fiber may not accurately cover the filament due to low twist level as explained in earlier studies [9,10]. Figure 4 shows the average UsterHairiness (H) values of dual-core yarns produced with different twist level, wool draft and elastane draft. According to ANOVA test result, the twist level ( $P_T=0.000$ ) and wool draft ( $P_W=0.001$ ) were found to be statistically significant for the UsterHairiness (H) values of yarn samples. In addition the intersections of twist level/wool draft ( $P_{T*W}=0.000$ ), wool draft/elastane draft ( $P_{W*E}=0.000$ ), twist level/elastane draft ( $P_{T*E}=0.000$ ) and the triple intersection of all factors ( $P_{T*W*E}=0.000$ ) were found to be statistically significant for Uster Hairiness (H).

From the SNK test results, the differences between UsterHairiness (H) values of yarns for all twist level were found to be statistically significant. SNK test results showed that an increase in twist level from 585 T/m to 750 T/m resulted in an improvement in UsterHairiness (H). This is caused by the decreasing amount of free fiber ends and/or fiber loops protruding from a yarnbody with increasing twist level.

In addition, the difference between UsterHairiness (H) values for 1.05 wool draft and the other wool drafts was found to be statistically significant. From the results, it was observed that there is decreasing trend in the hairiness values of yarn samples as wool draft increases.

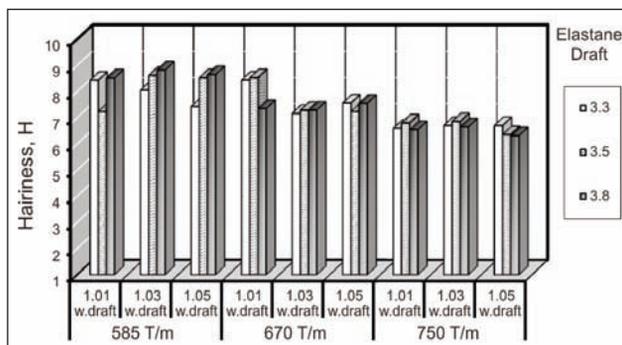


Fig. 4. Hairiness (H) versus twist level for comparable 1.01, 1.03, and 1.05 wool draft with 3.3, 3.5, and 3.8 elastane draft

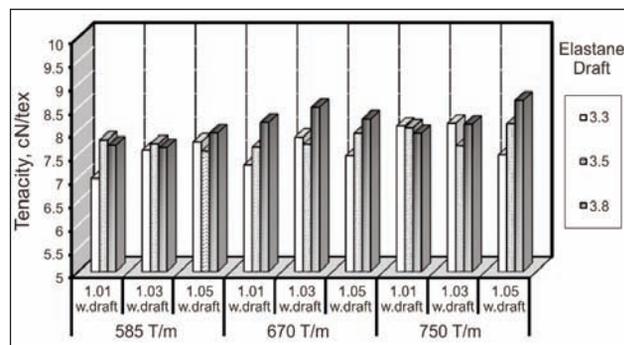


Fig. 5. Tenacity (cN/tex) versus twist level for comparable 1.01, 1.03, and 1.05 wool draft with 3.3, 3.5, and 3.8 elastane draft

Figure 5 shows the average tenacity (cN/tex) values of dual-core yarns produced with different twist level, wool draft and elastane draft. According to ANOVA test results, both the twist level ( $P_T=0.003$ ) and elastane draft ( $P_E=0.000$ ) were significant factors for the tenacity of yarn samples.

From the SNK test results (table 2), the difference between tenacity values for 750 T/m and the other twist levels was found to be statistically significant. It was observed that there is increasing trend in the tenacity values of yarn samples as twist level increases. The reason of this situation is the fact that the cohesion between the cores (wool/elastane) and sheath cotton fibers increases with the increase in twist level. These results are supported by previous studies on core-spun yarns [10, 11].

In addition, the difference between tenacity values for 3.8 elastane draft and the other elastane drafts was found to be statistically significant. From the results, it was observed that there is increasing trend in the tenacity values of dual-core yarn samples as elastane draft increases. This can be explained by the stress-induced crystallisation phenomenon of the elastane filament with increasing draft value. Su et al. have explained this phenomenon by the fact that when elastane with higher draw ratio is fed in production, the originally folded and twisted soft segments in the elastane filament are straightened allowing harder segments to form a crystal lattice by the effect of hydrogen bonding [12]. Moreover, similar to previous studies [13], the increase in the tenacity values with increasing elastane draft can also be associated with the decreasing elastane ratio which also means increasing sheath fibers' percentage in dual-core yarn structure.

Figure 6 shows the average breaking elongation values of dual-core yarns produced with different twist level, wool draft and elastane draft. According to ANOVA test results, the twist level ( $P_T=0.032$ ), elastane draft ( $P_E=0.000$ ) and wool draft ( $P_W=0.000$ ) were significant factors for breaking elongation (%) of yarn samples. The intersections of, wool draft/elastane draft ( $P_{W*E}=0.000$ ), twist/elastane draft ( $P_{T*E}=0.001$ ) and the triple intersection of all factors

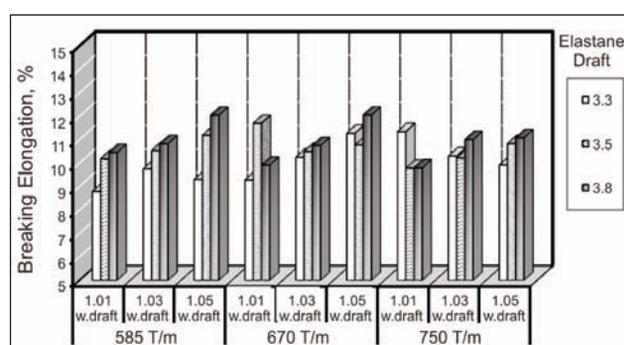


Fig. 6. Elongation (%) versus twist level for comparable 1.01, 1.03, and 1.05 wool draft with 3.3, 3.5 and 3.8 elastane draft

( $P_{T*W*E}=0.000$ ) were also found to be statistically significant for breaking elongation.

From the SNK test results, the difference between breaking elongation values for 585 and 750 T/m twist levels was found to be statistically significant. It was observed that there is increasing trend in the breaking elongation values of yarn samples as twist level increases. The reason of this case is the fact that the sheath fibers are wrapped better around each other with increasing twist level.

In addition, the differences between breaking elongation values of yarns for all wool drafts were found to be statistically significant. This increase in wool draft from 1.01 to 1.05 resulted in an improvement in breaking elongation.

From the SNK test results, the difference between breaking elongation values for 3.3 elastane draft and the other elastane drafts was found to be statistically significant. It was observed that there is increasing trend in breaking elongation values of yarn samples as elastane draft increase. This may be explained by the fact that the chance of fibers slipping in the dual-core yarn increases, as staple fibers in dual-core yarn increase with increasing elastane draft. Wu et al. (2003) and Lin et al. (2011) found similar results in their studies [14, 15].

## CONCLUSION

This study demonstrates that various properties of dual-core yarns are significantly affected by twist

level, wool draft and elastane draft as outlined in the followings:

The unevenness of dual-core yarns is only affected by twist level factor. Experimental results showed that there is decreasing trend in the unevenness values of yarn samples as twist level increase.

The hairiness of dual-core yarns is affected twist level and wool draft. The hairiness values decrease while twist level of dual-core yarns increases. The best hairiness values are obtained from dual-core yarn samples produced with 750 T/m twist level. Furthermore, experimental results show that there is decreasing trend in the hairiness values of yarn samples as wool draft increases.

The tenacity of dual-core yarns is affected by twist level and elastane draft factors. Experimental results

show that there is increasing trend in the tenacity values of yarn samples as twist level and elastane draft increase.

The elongation of dual-core yarns is affected by twist level, wool draft and elastane draft factors. Experimental results show that there is increasing trend in the tenacity values of yarn samples as twist level and elastane draft increase. The elongation values increase while wool draft of dual-core yarns increases. The best elongation values are obtained from dual-core yarn samples produced with 1.05 wool draft.

#### ACKNOWLEDGEMENTS

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# Multifunctional finishing treatments applied on textiles for protection of emergency personnel

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## REZUMAT – ABSTRACT

### Tratamente de finisare multifuncțională aplicate materialelor textile pentru protecția personalului de intervenție în situații de urgență

Lucrarea prezintă rezultatele cercetărilor efectuate pentru obținerea de materiale textile multifuncționale cu efecte multiple, prin tehnici de finisare superioară, utilizând produse chimice funcționale sub formă de dispersii apoase. S-a studiat posibilitatea combinării tratamentului cu dispersii cu efecte fotocatalitice și antibacteriene cu un tratament de hidrofobizare/oleofobizare, care să ofere simultan atât efect fotocatalitic și antibacterian durabil, cât și efect hidrofob/oleofob, în limite satisfăcătoare pentru toate aceste efecte. Rezultatele evaluărilor de laborator, efectuate pe suportul textil țesut din 50% bumbac și 50% poliamidă HT funcționalizat, au demonstrat că tratamentul de hidrofobizare cu dispersii fluoropolimerice poate fi combinat cu un tratament cu dispersii fotocatalitice pe bază de dioxid de titan sau cu dispersii pe bază de clorură de argint și dioxid de titan pentru obținerea de efecte multiple fotocatalitice, antibacteriene și hidrofob/oleofobe fără a diminua efectele de funcționalizare care s-ar fi obținut prin tratamentele realizate individual.

Cuvinte-cheie: tratamente de funcționalizare, efect hidrofob, activitate fotocatalitică, activitate antibacteriană, efecte combinate

### Multifunctional finishing treatments applied on textiles for protection of emergency personnel

The paper presents the results of the researches carried out for obtaining multifunctional textile materials with multiple effects, by means of superior finishing techniques, using functional chemicals in the form of aqueous dispersions. It has been studied the possibility of combining treatment with dispersions with photocatalytic and antibacterial effects with a hydrophobic/oleophobic treatment that simultaneously provides both sustainable photocatalytic and antibacterial effect as well as hydrophobic/oleophobic effect within satisfactory limits for all these effects. The results of the laboratory evaluations performed on 50% cotton and 50% functionalised HT polyamide textile fabrics showed that hydrophobization treatment with fluoropolymer dispersions can be combined with the treatment with titanium dioxide photocatalytic dispersions or silver chloride and titanium dioxide dispersions to obtain multiple photocatalytic, antibacterial and hydrophobic/oleophobic effects without diminishing the functionalization effects that would have been achieved by individual treatments.

Keywords: functionalization treatments, hydrophobic effects, photocatalytic activity, antibacterial activity, combined effects

## INTRODUCTION

Functional clothing can therefore be defined as a generic term that includes all such types of clothing or assemblies that are specifically engineered to deliver a pre-defined performance or functionality to the user, over and above its normal functions. Such clothing would normally be made from a mix of innovative materials, and functionality in this case would imply the added value or function that a garment is expected to perform. Functional clothing is a relatively new and exciting segment of the technical textiles group – one which is receptive to new product developments & technologies and abounding with niche applications. The emergence of performance clothing has been fuelled by recent breakthroughs and advances in technical fibres & fabrics and advances in garment manufacturing technologies. A lot of technologies originally developed for protective clothing applications have also become available in the

public domain and form a major constituent of this field [1].

Textiles are considered to be the interface between the user and the environment, but besides this characteristic, they must also have an active role, adapting to major changes dictated by physiological needs, in line with changes in environmental conditions. The field of functional clothing is wide and diverse with each functionality having its own specifications, material requirements, consequent technologies and processes. End use applications are diverse and often quite complex, ranging from life saving and hostile environment responsive to those improving the quality of life [2].

Except the hydro- and oleophobic effects, multi-barrier properties (protection against heat and flame, heat stress and heat stroke protection, soil-release) and relevant physiological parameters (breathability, thermoregulating/insulating properties) and wearing

comfort without the movement restriction are required for protective clothing. These properties achieved by customized yarn and fabric construction in combination with textile fibre selection, followed by special textile finishing and garment design (cut, multi-layered structures) [3].

Textile finishing plays an essential role in modifying the appearance, texture, touch and performance of all textiles so that the perception of the end user should be appropriate. Therefore, the use of functional finishing is particularly appreciated by textile manufacturers as it involves surface modification techniques that can be achieved in the final stages of the chemical finishing process. Functional finishing technologies allow manufacturers to continue using traditional raw materials, existing classical machinery and technologies while at the same time gaining added value, thereby enabling the potential buyers interested in functional or multifunctional fabrics to be stimulated and captured [4–5].

In order to obtain multifunctional textile materials with multiple photocatalytic, antibacterial and hydrophobic/oleophobic effects, this article presents laboratory technological experiments for the functionalization of traditional fiber textile materials by means of finishing techniques using functional chemical products as aqueous dispersions. In this regard, the possibility of combining treatment with dispersions with photocatalytic and antibacterial effects with a hydrophobic/oleophobic treatment has been studied, which simultaneously provides both a durable photocatalytic and antibacterial effect and a hydrophobic/oleophobic effect within satisfactory limits for all these effects.

## EXPERIMENTAL

### Materials

For technological functionalization experiments, a textile fabric was used which includes both in the warp and weft direction Nm 50/2 yarns made of 50%

cotton/50% polyamide HT. To obtain textile materials with photocatalytic activity, commercial photocatalytic aqueous dispersion based on TiO<sub>2</sub> – AERODISP® W 740 X with 40% content of the active substance (Evonik Degussa, Germany) has been used. The product based on fluorocarbon polymer dispersions (C6) NUVA 2114 (ARCHROMA), has been used for hydrophobic/oleophobic treatment. Sanitized® T 27-22 Silver (Sanitized AG, Switzerland) has been used in order to obtain the antibacterial effect.

### Preliminary preparation of the textile fabrics

In order to ensure a proper hydrophilicity of the textile material, which ensures the proper functioning of the textile backings, they have been subjected to conventional preliminary preparation by hot alkaline treatment, at a pH of medium alkalinity, on a laboratory jigger.

### Dyeing of the textile fabrics

The dyeing of the textile fabrics was performed with the direct dye Sirius Light Turquoise Blau GL (DyStar) and with a dyes mixture: Solophenil direct dye and Nylosan ROT N 2RBL reactive dye.

The parameters of the preliminary preparation and dyeing are presented in table 1.

### Functionalization treatments

The categories of chemicals selected to confer multifunctional effects were applied on the textile fabrics by padding method on the laboratory padder (Roaches, UK). After impregnation, the samples were heat treated for drying/condensation on the specific laboratory equipment for these operations (Roaches, UK). The experimental variants are shown below.

**Functionalization treatments for conferring the combined antibacterial-oleophobic/hydrophobic effect.** To confer the antibacterial-hydrophobic/oleophobic combined effect, the textile fabrics were subjected to treatment in the concomitant phase with the

Table 1

PARAMETERS OF PRELIMINARY TREATMENT AND DYEING FOR THE FABRIC MADE OF 50% COTTON/50% PA			
Composition of the treatment baths	Temperature	Duration of treatment	M:LR
<b>Bath 1: Hot alkaline treatment</b> 2 g/L Kemapon PC 3 g/L Na <sub>2</sub> CO <sub>3</sub> 3 g/L trisodium phosphate	95°C	60 min	1 : 10
<b>Bath 2: Dyeing</b> 2% Light Turquoise Blau GL 20 g/L NaCl	95°C	60 min	1 : 10
<b>Bath 2: Dyeing</b> 3% Solophenil 3% Nylosan ROT N 2RBL 20 g/L NaCl 1 mL/L CH <sub>3</sub> COOH	98°C	60 min	1 : 10
<i>After each technological operation, rinsing was performed under the following conditions: 80°C, 60°C, 40°C for 10 minutes each rinsing and a cold rinse for 10 minutes</i>			

THE CODIFICATION OF EXPERIMENTAL VARIANTS, TECHNOLOGICAL PARAMETERS, COMPOSITION OF THE TREATMENT BATHS		
Code	Composition of the treatment baths	Technological operations/technological parameters
V1	7 g/L Sanitized T 27-22 Silver	1. <b>Padding:</b> 2 bar squeeze pressure 2. <b>Drying:</b> 100°C, 2 minute
V2	1 mL/L acid CH <sub>3</sub> COOH (60%) 50 g/L NUVA N 2114 liq.	1. <b>Padding:</b> 2 bar squeeze pressure 2. <b>Drying:</b> 100°C, 2 minutes 3. <b>Heat-setting:</b> 170°C, 40 sec.
V3	1 mL/L acid CH <sub>3</sub> COOH (60%) 50 g/L NUVA N 2114 liq. 7 g/L Sanitized T 27-22 Silver	1. <b>Padding:</b> 2 bar squeeze pressure 2. <b>Drying:</b> 100°C, 2 minutes 3. <b>Heat-setting:</b> 170°C, 40 sec.
V4	1 mL/L acid CH <sub>3</sub> COOH (60%) 50 g/L Nuva 4211 liq.	1. <b>Padding:</b> 2 bar squeeze pressure 2. <b>Drying:</b> 120°C, 2 minute
	50 mL/L AERODISP W 740 X	3. <b>Padding:</b> 2 bar squeeze pressure 4. <b>Drying:</b> 120°C, 2 minutes
	1 mL/L acetic acid 60% 50 g/L Nuva 2114 liq.	5. <b>Padding:</b> 2 bar squeeze pressure 6. <b>Drying:</b> 100°C, 2 minute 7. <b>Heat-setting:</b> 170°C, 1 minute

Sanitized® T 27-22 Silver and with the hydrophobization product NUVA 2114, the operations of technological flow being the following: preliminary preparation → dyeing → padding in single bath with Sanitized® T 27-22 Silver and NUVA 2114 liq → drying → condensation.

**Functionalization treatments for conferring the combined photocatalytic-hydrophobic/oleophobic effect.** In order to obtain the water/oil repellent effect in combination with the photocatalytic effect it was chosen to treat the textile fabrics in successive phases with the product based on fluorocarbon polymer dispersions (C<sub>6</sub>) with a hydrophobic/oleophobic effect (NUVA 2114) and with commercial photocatalytic dispersion based on TiO<sub>2</sub> (AERODISP® W 740 X). The sequence of the constituent operations of technological flow being the following: preliminary preparation → dyeing → padding with NUVA 2114 → drying → padding with AERODISP® W 740 X → drying → re-treatment by padding with NUVA 2114 → drying → condensation.

The codification of the experimental variants, the operations, technological parameters and composition of the treatment baths for each treatment alternative selected in order to produce multifunctional textiles are shown in table 2.

## Methods

**Evaluation of the photocatalytic activity of functionalized fabrics.** Photocatalytic activity of textile fabrics treated in successive phases with commercial photocatalytic dispersion based on TiO<sub>2</sub> and with the water and oil repellent product base on fluorocarbon dispersion was evaluated by determining the photodegradation efficiency of methylene blue dye (MB), used as aqueous solution of 0.008 g/L. Functionalized textile material were immersed for 30 minutes in MB solution and subsequently has been subjected to UV irradiation for 6 hours using the “dark room” type CN

15 LC (Vilber Lourmat, France). Incorporated lamps (2 x 15 W) were the sources of ultraviolet radiations and emitted radiation of λ<sub>max</sub> (emission) = 365 nm and respectively 254 nm. Evaluation of the photocatalytic activity was performed by measuring the color difference of the irradiated samples compared with non-irradiated samples (reference). Color measurements were performed according to ISO 105 J03: 2001, using the Datacolor™ 650 spectrophotometer (Datacolor, Switzerland) and the light source was the illuminant D65/10. Values obtained for color difference are the average of 5 individual measurements carried out on the treated samples with photocatalytic dispersions and on the standard samples considered, treated only with photocatalytic activity.

**Physical-chemical and physical-mechanical characteristics.** The finished fabrics were also characterized in terms of the main physical-chemical and physical-mechanical characteristics, respectively: mass (SR EN 12127-2003), tensile strength (SR EN ISO 13934-1/2013), tearing strength (SR EN ISO 13937-3: 2002), resistance to water vapor in stationary mode (SR EN 31092/ A1:2013 ISO 11092:1997), air permeability (SR EN ISO 9237: 1999), thermal resistance (SR EN 31092/ A1:2013 ISO 11092:1997). **Evaluation of hydrophobicity of functionalized textiles.** In order to evaluate hydrophobicity, the samples treated in different experimental variants were tested for surface wetting resistance – Spraytest (SR EN ISO 4920: 2013).

**Antimicrobial tests.** The antibacterial activity of the functionalized materials in different variants was qualitatively determined in accordance with ISO 20645: 2004 (E) standard method, by using of cultures in liquid medium replicated at 24 hours of ATCC 6538 *Staphylococcus aureus* (Gram-positive) and *Pseudomonas aeruginosa* (Gram-negative) strains. For determination, the samples were cut in circular shape with a diameter of 2 cm and subsequently disposed

in the middle of Petri plates. The culture medium was poured into two layers in Petri plates, lower layer consists of culture medium free from bacteria and the upper layer being inoculated with the test bacteria, then incubated at 37°C and analyzed after 48 hours. Antifungal efficiency testing was carried out against *Candida albicans* (ATCC 90028) strain, according to ISO 20743:2007 standard, by absorption method of microbial inoculum on the functionalized fabrics. Textile materials were cut in samples with 1 cm<sup>2</sup> surface area, and placed in sterile tubes. Afterwards, 50 µL of microbial inoculum was pipetted on the surface of the material, with microbial concentration of 6×10<sup>3</sup> UFC/mL, followed by 24 h incubation at 36°C. After incubation period, samples were each washed with 3 mL of sterile deionized water, and each tube was vortexed for 20–30 seconds, followed by sampling of 100 µL from each tube and plated on Petri dishes with agarized Sabouraud media, with a Drigalski spatula. Plates were held at room temperature for 30 minutes, and then incubated, with the lid facing down (to avoid formation of condensation on the lid), for 24 h at 36°C.

## RESULTS AND DISCUSSIONS

### Photocatalytic effect

Colour differences attributes were determined considering as reference the samples treated both with the photocatalytic dispersion and the water/oil repellent product, before UV irradiation, the obtained values being shown in table 3.

Table 3

COLOUR DIFFERENCES ATRIBUTES BEFORE AND AFTER UV IRRADIATION				
Sample code	Observation	Colour difference		Sample color
		DL*	DE*	
V4	Before UV irradiation	Reference		
	UV irradiation 254 nm	-0.31	4.26	
	UV irradiation 365 nm	1.17	2.59	
M*	Before UV irradiation	Reference		
	UV irradiation 254 nm	0.27	2.17	
	UV irradiation 365 nm	0.40	1.21	

\* Sample is preliminary treated and dyed without any of the functional treatments

From the analysis of the values obtained for the difference in lightness DL\* it is found that the most obvious photocatalytic effect is registered in the case of the fabric treated with commercial photocatalytic dispersion (AERODISP W 740 X) and with the hydrophobic/oleophobic product (NUVA 2114) after irradiation at 365 nm, in this case, the highest value for this parameter (DL\* = 1.17) has been obtained. The relatively low value obtained for this parameter does not reveal a less efficient photocatalytic effect, this behavior can be attributed to the fact that the hydrophobic textile material absorbs a much diminished

amount of MB, which can be degraded by UV discoloration. In the case of the witness sample, which was not subjected to the functionalization treatment, a difference in lightness between the non-irradiated samples and those irradiated at the two wavelengths, with positive subunit values (lighter than the non-irradiated sample), is due to the sensitivity to UV radiation of dyes used for dyeing textile samples and less to the decoloration of the MB dye used to assess the photodegradation effect.

### Physical-mecanical characteristics

The main physical-mechanical characteristics are present in the table 4.

Table 4

PHYSICAL-MECANICAL CHARACTERISTICS					
Characteristic	Code				
	V1	V2	V3	V4	
PHYSICAL-MECANICAL CHARACTERISTICS					
Mass [g/m <sup>2</sup> ]	269	265	236	238	
Tensile strength, [N]	Warp	1423	1421	1411	1401
	Weft	1067	1087	880	942
Tear strength, [N]	Warp	49,8	57,8	53,30	54,9
	Weft	36,1	42,9	41,00	39,6

The tensile characteristics such us tensile strength and tear strength of finished samples are given in table 4. The comparative analysis of tensile strength and tear strength values obtained for all the treated samples shows that:

- tensile strength, for the samples treated according to the V3 experimental variant (hydrophobic/oleophobic/antibacterial combined treatment) decreases in the warp direction by 0,84% compared to V1 variant (antimicrobial treatment) and by 0,70% compared to V2 (hydrophobic/oleophobic treatment) and decreases in the weft direction by 17,52% compared to V1 variant, and by 19,04% respectively, compared to V2 variant;
- tear strength for the V3 variant records an increase in the warp direction compared to V1, by 6,5% in the warp direction and by 11,9%, respectively, in the weft direction and a decrease in the warp direction compared to V2 by 7,78% and, 4,42%, respectively, in the weft direction;
- tensile strength of the samples treated according to the V4 variant (photocatalytic/hydrophobic/oleophobic combined treatment) decreases by 1,4% in the warp direction and by 13,3% in the weft direction compared to the V2 variant (hydrophobic/oleophobic treatment);
- tear strength for V4 variant decreases by 5,01% in the warp direction and by 7,69% in the weft direction compared to the variant with V2 (hydrophobic/oleophobic treatment).

Table 5 shows the fabric comfort related characteristics, such as surface wetting resistance, air permeability, water vapour resistance and thermal resistance.

Table 5

FABRIC COMFORT CHARACTERISTICS			
Characteristic	V2	V3	V4
Wetting resistance			
ISO scale	5	5	5
AATCC photographic scale	100	100	100
Air permeability, [l/m <sup>2</sup> s] (100Pa)	31,65	38,48	44,11
Water vapour resistance under steady-state conditions, R <sub>et</sub> , [m <sup>2</sup> Pa/W]	-	7,05	7,47
Thermal resistance, R <sub>ct</sub> , [m <sup>2</sup> K/W]	-	0,0189	0,0180

All the finished fabrics show similar level of air permeability, water vapour resistance under steady-state conditions and thermal resistance. The multifunctional treated samples in different experimental variants and tested from the point of view of the hydrophobic effect by superficial wetting test (Spraytest), have shown maximum values of 100 on the AATCC photographic scale, regardless of the variant of finishing applied, thus indicating a very good hydrophobic effect. It has thus been demonstrated that hydrophobic treatment can be combined with photocatalytic effect treatment or with the treatment to obtain the antimicrobial effect, without diminishing the hydrophobic effect.

### Antimicrobial tests

**Antibacterial activity.** Images of Petri plates after 48 h incubation are shown in figure 1. The evaluation of antimicrobial activity consisted in highlighting the presence or absence of the inhibition zone around the samples, the size of the inhibition zone being calculated by the formula:

$$H = D - \frac{d}{2} \quad (1)$$

where:

$H$  is the inhibition zone (mm);

$D$  – the total diameter of the sample and the inhibition zone (mm);

$d$  – sample diameter (mm).

The results obtained from the evaluation of antimicrobial activity for the treated samples in different experimental variants are shown in table 6. For antibacterial activity testing it was considered as the witness sample (M) the dyed fabric without the functionalization treatment.

From the analysis of the data obtained by testing the antibacterial activity, it was found that for the samples tested with *Staphylococcus aureus* there were increases on the contact surface as observed on the entire culture medium area on all samples, except for the V1 sample having a 1.5 cm inhibition area. For samples tested with *Pseudomonas aeruginosa*, the only sample that totally inhibited growth was V2. The V3 sample has no antibacterial activity, both for Gram

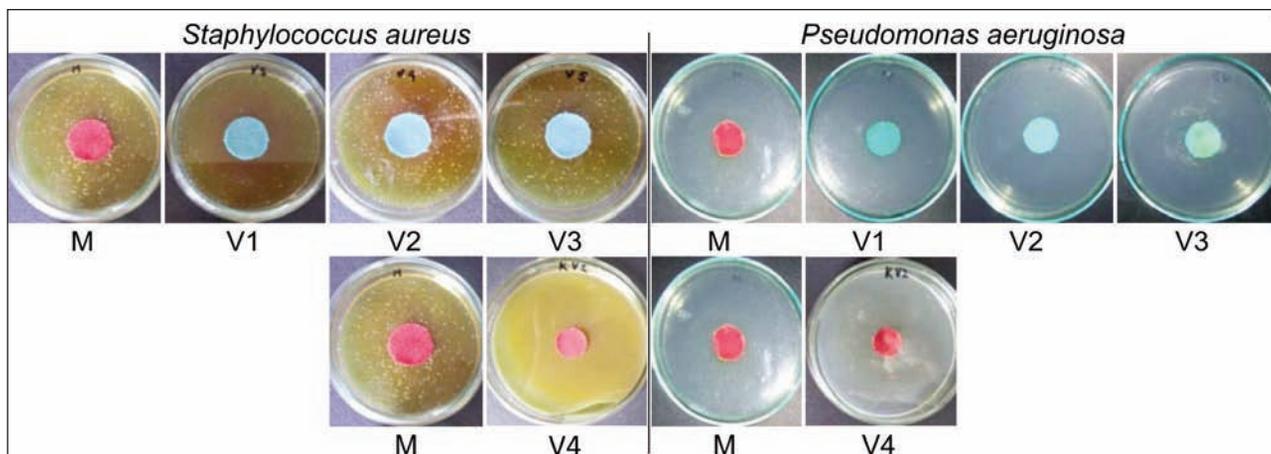


Fig. 1. Images of Petri plates after 48 h incubation

Table 6

EVALUATION OF THE ANTIBACTERIAL EFFECT				
Code	<i>Staphylococcus aureus</i>		<i>Pseudomonas aeruginosa</i>	
	Inhibition zone [cm]	Evaluation	Inhibition zone [cm]	Evaluation
V1	1.5	Satisfactory effect	-	Unsatisfactory effect
V2	-	Unsatisfactory effect	-	Satisfactory effect
V3	-	Unsatisfactory effect	-	Unsatisfactory effect
V4	-	Unsatisfactory effect	-	Satisfactory effect
M	-	Unsatisfactory effect	-	Unsatisfactory effect

positive and Gram negative microorganisms. The V4 sample has no antibacterial effect against the *Staphylococcus aureus* test strain but has completely inhibited the growth of *Pseudomonas aeruginosa*. **Antifungal activity.** For antifungal efficiency quantification, the percentage and logarithmic reduction rate of each sample was calculated, related to untreated material (control). Testing of the antimicrobial activity of functionalized textile materials highlighted different rates of microbial reduction (table 7), dependent on the type of treatment performed.

Table 7

QUANTITATIVE ANTIFUNGAL EFFICIENCY TESTING			
Sample	C <sub>24h</sub> (CFU/mL)	R%	Log <sub>10</sub> red.
Control*	5.4×10 <sup>3</sup> CFU/mL (C <sub>0</sub> )	13.34	0.06
V1	0 CFU/mL	100	3.80
V2	5.4×10 <sup>3</sup> CFU/mL	10	0.04
V3	0 CFU/mL	100	3.80

\*Preliminary treated fabric, dyed and non-functionalized

Antifungal tests results show maximum efficiencies of fabrics treated to confer multifunctional antimicrobial character (code V1) and multifunctional antibacterial/oleophobic/hydrophobic (code V3), for which percentage reduction rates of 100%, against *Candida albicans* population, were obtained. The fabric treated with fluorocarbon (C<sub>6</sub>) polymeric dispersions, for

oleophobic/hydrophobic treatment, according to V2 variant, shows weak antimicrobial activity, with only 10% percentage reduction rate. The result may be due to pronounced hydrophobic character of the textile material (compared to V1 and V3), which allowed the pearling of microbial inoculum on the material (or inoculum leakage on the walls of the test tube), thus not allowing an optimal contact surface. The non-functionalized fabric (code M), showed a poor inhibition activity on growth and development of *Candida albicans* population, with a microbial reduction rate of 13.34%, most likely due to mechanical retention of microbial cells on the surface of the material.

## CONCLUSIONS

Laboratory experiments performed on fabrics made of 50% cotton and 50% polyamide HT have demonstrated that hydrophobization treatment with fluoropolymer dispersions can be combined with treatment with titanium dioxide-based photocatalytic dispersions or dispersions based on silver chloride and titanium dioxide to obtain multiple photocatalytic, antibacterial and hydrophobic/oleophobic effects without diminishing the functionalization effects that would have been obtained by the individual treatments.

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# Study of electrospun cellulose acetate fibers

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## REZUMAT – ABSTRACT

### Studiu asupra fibrelor de acetat de celuloză electrofilate

Obiectivul acestui studiu a fost prepararea nanofibrelor de acetat de celuloză prin electrofilare, utilizând un amestec de solvenți. Soluțiile de acetat de celuloză au fost electrofilate din sisteme de solvenți binare și ternare, cum ar fi N,N-dimetilformamidă, acetonă și cloroform. S-au investigat efectele sistemelor de solvenți asupra caracteristicilor structurale, morfologice și mecanice ale fibrelor.

Cuvinte-cheie: electrofilare, acetat de celuloză, nanofibre electrofilate

### Study of electrospun cellulose acetate fibers

The objective of this work was the preparation of cellulose acetate nanofibers by electrospinning using a mixture of solvents. Cellulose acetate solutions were electrospun from binary and ternary solvent systems, such as N,N-dimethylformamide, acetone and chloroform. The effects of the solvent systems on the structural, morphological and mechanical characteristics of the fibers were investigated.

Keywords: electrospinning, cellulose acetate, electrospun nanofibers

## INTRODUCTION

In the recent years, nanoscience and nanotechnology have developed rapidly, leading to major advances in nanomaterials processing and characterization. From this category, one-dimensional materials – nanofibers – present extremely high specific surface area due to small diameters and nanofiber membranes showing high ratio of surface area to volume, high porosity, characterized by high pore interconnectivity [1]. The unique characteristics of nanofibers make them an important candidate for a large number of industrial applications [2–4].

A significant number of methods can be used to obtain nanofibers: interfacial polymerization [5], melt spinning [6], solution spinning [7] and electrospinning [8, 9]. Electrospinning is a simple and versatile process that uses the electrical field to obtain the polymeric nanofibers from solution. This method for preparing nanofibers allows the use of a large number of polymers [10–11]. Some of these polymers are polyvinyl pyrrolidone (PVP), polylactic acid (PLA), chitosan, polyester urethane (PEU), polyvinyl alcohol (PVA), polystyrene, polyacrylonitrile (PAN) and chitin [12–16].

From a wide range of polymeric materials, cellulose acetate (CA) belongs to the new generation of environmentally friendly products that fit into the new research directions due to the requirements of developing materials with minimal impact on the environment, using renewable resources as much as possible. Numerous single and binary solvent systems have been used for obtaining electrospun CA fibers.

By using traditional single solvent systems for preparing CA solution, such as N,N-dimethylformamide (DMF) [17], chloroform [18], acetic acid [19], N,N-dimethylacetamide (DMAc) [20] and acetone [21], some problems appear regarding to the obtaining of continuous and bead-free electrospun fibers. The physical properties of the solvent system can be improved by using a binary solvent system of two solvents with different dielectric constant and boiling point of both solvents. Such binary solvent systems for CA solution are acetone/ethanol [22], acetic acid/water [23] or DMAc/acetone [21].

In this paper, a new ternary solvent system consisting in DMF/Acetone/Chloroform was developed in order to obtain continuous CA fibers by electrospinning method. The influence of the solvents on the morphology and mechanical properties of CA fibers has been studied.

## EXPERIMENTAL WORK

### Materials

In this study, cellulose acetate (CA) with a molecular mass of 30,000 purchased from Aldrich was used as polymer source. The solvents used for dissolving CA were acetone (A) with 1.3 g/cm<sup>3</sup> density, purchased from Aldrich, N,N-dimethylformamide (D) with 0.94 g/cm<sup>3</sup> density, purchased from Alfa Aesar, and chloroform (C) with 1.485 g/cm<sup>3</sup> density, purchased from Chimreactiv. All materials were used without any purification.

## Preparation of electrospinning solutions

Five CA solutions with concentrations of 12% wt. were prepared in the binary system of solvents DMF/Acetone (table 1). Also, four CA solutions with concentrations of 12% wt. were prepared in the ternary system of solvents DMF/Acetone/Chloroform (table 2). A good dissolution of the CA in binary or ternary solvent system is very important in achieving good morphological properties of the electrospun fibers. Thus, CA was dissolved in the solvents system by magnetic stirring at room temperature for 2 hours, at a rotational speed of 480 rpm and the obtained solutions were used immediately in the electrospinning process.

Table 1

Solution	CA (% wt.)	D/A (v/v)
ACAD100	12	1/0
ACAD75	12	3/1
ACAD50	12	1/1
ACAD25	12	1/3
ACAD0	12	0/1

Table 2

Solution	CA (% wt.)	D/A/C (v/v/v)
DAC111	12	1/1/1
DAC112	12	1/1/2
DAC121	12	1/2/1
DAC211	12	2/1/1

Table 3

Solvent	Molecular weight [g/mol]	Boiling point [°C]	Electrical conductivity at 25°C [ $S \cdot m^{-1}$ ]	Latent heat [ $kJ \cdot mol^{-1}$ ]	Surf. tension at 20°C [ $mN \cdot m^{-1}$ ]	Abs. viscosity at 25°C [ $mPa \cdot s$ ]
Acetone	58	56	$5.0 \cdot 10^{-7}$	29.6	23.30	0.33
Chloroform	119	61	$< 1.0 \cdot 10^{-8}$	29.4	27.16	0.57
DMF	73	153	$6.0 \cdot 10^{-6}$	42.1	35.00	0.82

In order to obtain the nonwoven CA fibers mats on an aluminium foil substrate by electrospinning method, a NaBond unit was used with an applied voltage of 18 kV, a solution flow rate of 1.8 mL/h, nozzle size spinneret of 0.8 mm, spinneret-to-collector distance of 20 cm and a stationary substrate.

## Characterization

Morphological characterization of the CA obtained fibers was performed with scanning electron microscopy (SEM) using a FESEM/FIB/EDS Workstation Auriga produced by Carl Zeiss Germany, with an acceleration voltage of 5 kV, using the SESI detector.

The chemical structure of CA and CA fibers was determined by FTIR measurements performed by using a Jasco FTIR-4200 spectrophotometer, connected to

an ATR JASCO PRO 470-H module. All the samples were measured directly on the diamond crystal surface, in the range of 400–4000  $cm^{-1}$ , at a resolution of 4  $cm^{-1}$  and 50 scans for each spectrum.

Wettability testing of the obtained CA fibers mats was made using the sessile drop method. Contact angle of the polymeric nonwoven mats was determined with distilled water by using an optical microscope equipped with a camera for images acquisition on the computer and the images were processed using the software Image J, Drop Analysis - Drop Snake.

Mechanical properties (tensile strength) of the CA electrospun nonwoven fibers mats were measured by using a mechanical testing machine, model LFM 30 kN, Walter & Sai AG Switzerland.

## RESULTS AND DISCUSSIONS

The physical properties of the solvents (table 3) [24], especially the volatility, have a major influence on the formation and the morphology of fibers obtained by electrospinning method. By using volatile solvents such as acetone, the tip of the needle can be easily blocked with the polymer because the solvent evaporates quickly. In these researches, the partial elimination of this problem was obtained by using a binary solvent system for dissolving CA with close volatility, such as acetone (boiling point 56°C) and chloroform (boiling point 61°C). This small difference of solvents volatility did not lead to the obtaining of uniform and beads-free CA fibers (the study is not presented here).

Partial solving of this problem was accomplished by using a binary solvent system. This binary mixture contains two solvents in different ratios that show a

higher difference of volatility. Therefore, the binary DMF/Acetone system in various ratios can be used to dissolve CA and to obtain fibers through electrospinning. In this research, we went further in order to obtain continuous and uniform CA fibers and we used a ternary solvent system such as DMF/Acetone/Chloroform.

## FTIR characterization

The FTIR spectrometry (figure 1) was used to study the influence of the solvent type on the chemical structure of cellulose acetate (CA). The recorded spectra look similar and present the characteristic bands of cellulose acetate [25–27]: the bands from the 2800–3000  $cm^{-1}$  region assigned to the stretching vibrations of C–H ( $CH_2$  groups [28], 1735  $cm^{-1}$

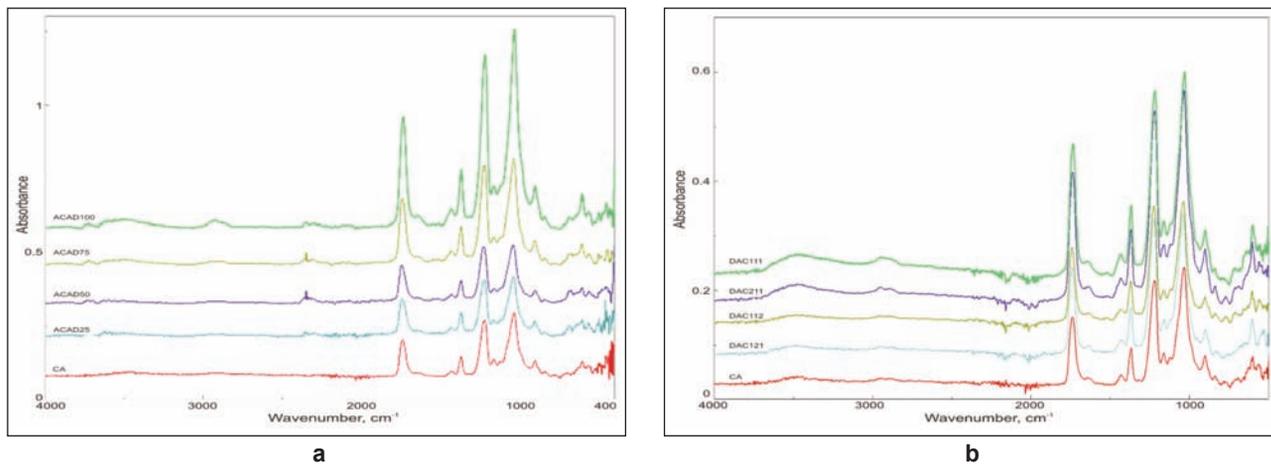


Fig. 1. FTIR spectra of CA crystals and of the obtained CA fibers from D/A binary solvent system (a) and D/A/C ternary solvent system (b)

(C=O stretching of acetyl or carboxylic acid),  $1435\text{ cm}^{-1}$  ( $\text{CH}_2$  or OH in plane bending),  $1370\text{ cm}^{-1}$  (CH deformation from  $\text{CH}_3$ ),  $1220\text{ cm}^{-1}$  (C–O stretching of acetyl group),  $1163\text{ cm}^{-1}$  (C–O–C anti-symmetric bridge stretching),  $1033\text{ cm}^{-1}$  due to C–O–C (ether linkage) of the glycosidic unit,  $903\text{ cm}^{-1}$  ( $\beta$  glycosidic linkages between the sugar units)). The presence of a wide band in the region  $3100\text{--}3500\text{ cm}^{-1}$  (stretching vibrations of OH) and the presence of a band at  $1647\text{ cm}^{-1}$  (due to H–O–H bending) indicate the presence of water adsorbed on the fibers surface [27, 28]. A variation of the intensity of OH stretching band can be observed, which can be attributed rather to the water adsorption on the surface of CA fibers than to chemical modification induced by solvents [26]. The different water adsorption degrees could also indicate some modifications on the crystalline structure of the CA fibers induced by solvents (higher crystallinity degree, lower water adsorption) [26].

### Morphological characterization

Figure 2 shows the SEM images of the electrospun CA fibers prepared from binary solvent system DMF/Acetone at different v/v ratios: 1/0 (figure 2, a), 3/1 (figure 2, b), 1/1 (figure 2, c), 1/3 (figure 2, d), 0/1 (figure 2, e). By using DMF as single solvent (figure 2, a) and by adding acetone in a ratio of 3/1 v/v, only beads with micron size were obtained (figure 2, b). By increasing the concentration of acetone to 1/1 v/v, very thin fibers with a diameter of about 74 nm start to appear on big beads with diameters around 2000 nm (figure 2, c). By using a binary solvent system of DMF/acetone with a ratio of 1/3 v/v, fibers with an average diameter of about 298 nm with discrete beads were obtained (figure 2, d). By using acetone as single solvent, fibers without beads with an average diameter of 3750 nm were obtained (figure 2, e). The physical properties of solvents have a major influence on the electrospun products. Because DMF

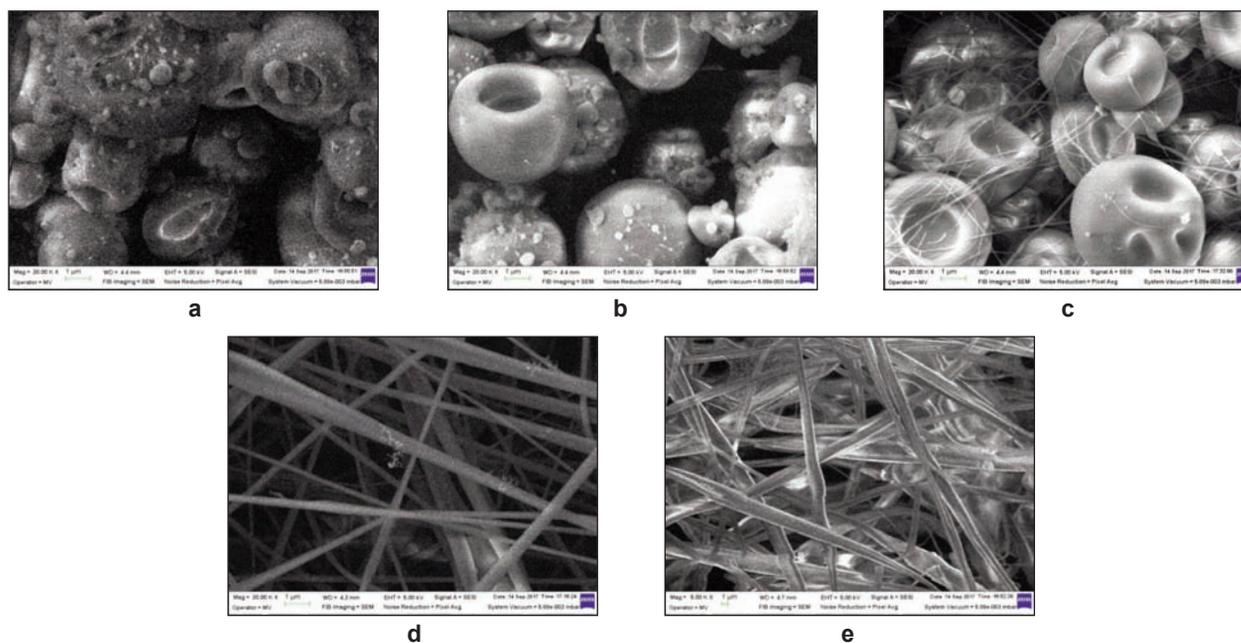


Fig. 2. SEM images of electrospun CA fibers obtained from D/A binary solvent system at different v/v ratios: 1/0 (a), 3/1(b), 1/1 (c), 1/3 (d) (20 kx magnification), and 0/1 (e) (5 kx magnification)

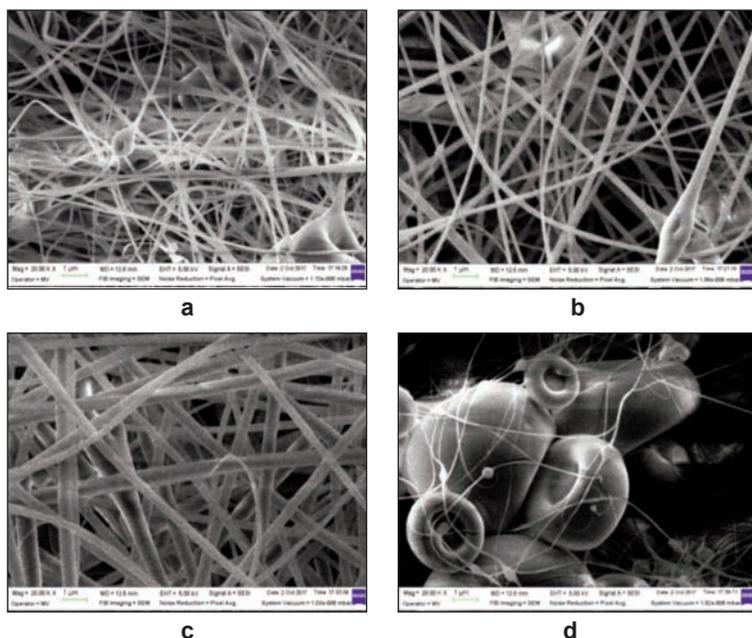


Fig. 3. SEM images of electrospun CA fibers obtained from D/A/C ternary solvent system at different v/v/v ratios: 1/1/1 (a), 1/1/2(b), 1/2/1 (c), and 2/1/1 (d) (20 kx magnification)

has a higher boiling point and a higher surface tension, only beads were obtained in comparison to acetone that presents a lower value of these physical parameters. For this reason, when we used acetone as solvent, we obtained fibers. Because of the high boiling point of DMF, during the electrospinning process the ejected charging jet of the solution do not have enough time to dry, and for this reason only droplets were obtained. When using acetone as solvent for obtaining CA fibers, some problems appeared because the needle tip was easily blocked with polymer due to the fast solvent evaporation. By using a binary solvent system of DMF/acetone in a ratio of 1/3 v/v these problems were partially solved, the overall volatility of the solvent system was reduced, and as a consequence fibers with discrete beads were obtained.

Figure 3 illustrates the SEM images for electrospun CA fibers prepared from ternary solvent system DMF/Acetone/Chloroform at different v/v/v ratios: 1/1/1 (figure 3, a), 1/1/2 (figure 3, b), 1/2/1 (figure 3, c) and 2/1/1 (figure 3, d). Chloroform physical properties (table 3) exhibit values that are intermediate between DMF and acetone, and by adding this in the ternary solvent system we tried to control the overall properties of the final solution.

By using a mixture of solvents in equal parts (D/A/C solvent system with the volumetric ratio 1/1/1 – figure 3, a), fibers with 198 nm average diameter and with droplets were obtained. By increasing the amount of chloroform in the solvent system (D/A/C solvent system with the volumetric ratio 1/1/2 – figure 3, b), fibers with higher average diameter of about 235 nm were obtained, and the droplets diameter increased as well. When using a ternary solvent system with a higher concentration of acetone (D/A/C solvent system with the volumetric ratio 1/2/1 – figure 3, c), fibers

with smooth surface, uniform diameters and beads-free were obtained, but the fibers have higher diameters of about 440 nm. By increasing the amount of DMF (D/A/C ratio 2/1/1 – figure 3, d), beads with discrete fibers with average diameters of about 58 nm were obtained.

### Wettability testing of the fibers

To analyse the wettability of the different nonwoven electrospun CA fibers using D/A solvent system, water contact angles were measured (figure 4). A tendency of the obtained nonwoven CA fibers is the decreasing of the contact angle with the increasing of the amount of acetone in the solvent system. The decrease of the contact angle starts from 130.9° for the CA fibers prepared with D/A solvent system 1/0 to 126.5° for the CA fibers prepared with D/A solvent system 1/1, until 124.5° for the CA fibers prepared with D/A solvent system 0/1. For electrospun CA samples obtained from the D/A solvent system 3/1 and 1/0 it was observed

that after 20 seconds the contact angle decreased to the value of 55°, respectively 53°. So, the nonwoven fiber mats exhibit an intermediate hydrophilic behaviour. This behaviour appeared most probably due to the morphology of the electrospun CA fibers and the presence of the droplets which results in lower contact angles.

In the case of electrospun nonwoven CA fibers prepared from DMF/Acetone/Chloroform ternary solvent system, the contact angle (table 4) did not present a

Table 4

Sample	Contact angle (degree)
DAC111	128.5
DAC112	129.4
DAC121	131.2
DAC211	130.0

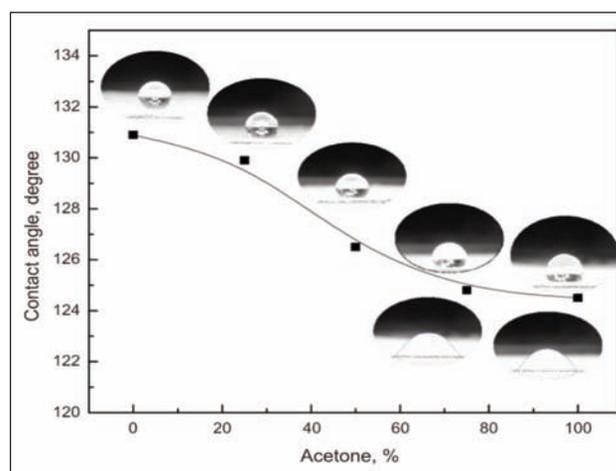
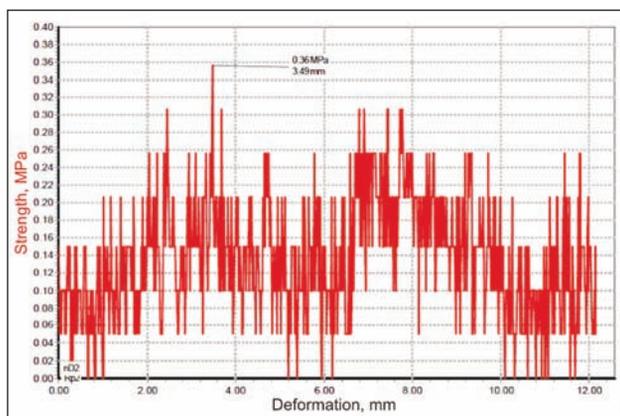
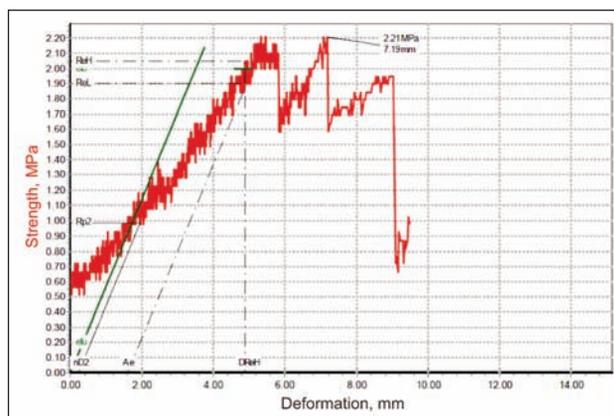


Fig. 4. Contact angle for the electrospun nonwoven CA prepared from DMF/acetone binary solvent system



a



b

Fig. 5. Tensile strength curves of the CA fibers prepared using D/A/C ternary solvent system 1/1/2 (a) and 1/2/1 (b)

considerable variation, which remained constant at about  $128^{\circ}$ – $131^{\circ}$ . By adding chloroform in the solvent system we noticed a small influence to the hydrophobicity by increasing the contact angle of the sample prepared with the highest amount of acetone.

### Mechanical characterisation

In order to perform the tensile strength measurement, the CA fibers were electrospun for 6 hours on a textile substrate (gauze fabric). Tensile strength was performed only for the samples prepared with ternary solvent system D/A/C 1/1/2 (figure 5, a) and 1/2/1 (figure 5, b), because these samples could be prepared and handled for test with minimal damage on the structure of the mats. The rest of the samples prepared using the solvent systems D/A/C ratio 1/1/1 and 2/1/1 were quite weak and were damaged very easily when handled.

Tensile strength tests were carried out with a drawing speed of 50 mm/minute on rectangular samples with the size of 100 mm  $\times$  20 mm. For each type of material five tests were carried out and then an average value of the obtained parameters was calculated.

In the case of the samples obtained using the solvent system D/A/C 1/1/2, the tensile strength was 0.44 MPa and it increased until 2.77 MPa for the samples prepared using the solvent system D/A/C 1/2/1. This increase appears due to the smooth and uniform diameter fibers obtained and the absence of the beads in the mats. The presence of beads in the

fibers mats acts as defects and leads to a lower number of fibers and lower interactions between them and therefore lower values of the tensile strength are obtained.

### CONCLUSIONS

In the research which was carried out, the cellulose acetate fibers were obtained by electrospinning using polymer solutions prepared from simple, binary and ternary solvents systems containing the following solvents: N,N-dimethylformamide, acetone and chloroform.

When using the DMF/Acetone binary solvent system, fibers with discrete beads were obtained by electrospinning. When using the ternary solvent system DMF/Acetone/Chloroform for preparing the electrospinning cellulose acetate solution, a uniform morphology of the fibers was obtained, these fiber mats having a tensile strength up to 2.77 MPa.

During these experiments, the morphology of electrospun cellulose acetate fibers was controlled by modifying the physical parameters of the solvent systems (binary or ternary), thus continuous, uniform and smooth fibers were obtained by using a cellulose acetate solution prepared with the solvent system DMF/Acetone/Chloroform in a volumetric ratio of 1/2/1.

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## REZUMAT – ABSTRACT

### Investigarea proprietăților antibacteriene ale țesăturilor din lână vopsite cu conuri de pin

*Tendențele în modă și culoarea reprezintă elementul primordial al primei etape de selecție a produselor textile. Acesta este motivul pentru care, în industria textilă, vopsirea are un mare succes. În special vopsirea naturală a dus la dezvoltarea de noi tendințe în ultima perioadă și a dobândit importanță în acest sens. În acest studiu, conurile de pin au fost utilizate pentru vopsirea țesăturilor din lână cu ajutorul a cinci agenți de mordansare diferiți și au fost realizate și vopsiri fără mordant. În plus, țesăturile din lână vopsite cu conuri de pin au fost evaluate din punctul de vedere al rezistenței la lumină și la spălare. S-a constatat că diferite culori și nuanțe pot fi obținute prin utilizarea unor agenți de mordansare și, în final, s-a observat că pot fi utilizate conurile de pin ca sursă de vopsire naturală. Mai mult decât atât, eficiența antibacteriană a probelor vopsite a fost investigată pentru a studia efectul sursei de colorant natural utilizat și al agenților de mordansare. S-a constatat că probele vopsite au proprietăți antibacteriene diferite în funcție de agentul de mordansare utilizat și de bacteriile analizate.*

*Cuvinte-cheie: lână, material textil, colorant natural, antibacterian, pin, con de pin*

### Investigation of antibacterial properties of wool fabrics dyed with pine cones

*Fashion and color are the foreground of the case in the first stage of the selection of textile products. That is why coloring in textiles has a great appeal. In particular, natural dyeing, which has caught up with new trends in recent times, has gained importance in this regard. In this study, pine cones were used in dyeing of wool fabrics with the help of five different mordanting agent and also mordant-free dyeings were performed too. Besides, wool fabrics dyed with pine cones have been evaluated in terms of light and washing fastnesses. It was found that different colors and shades can be obtained with the use of different mordanting agents and finally it was observed that pine cones can be used as a natural dye source. Moreover the antibacterial efficiencies of the dyed samples were investigated to see the effect of the used natural dye source and the mordanting agents. It was seen that the dyed samples have different antibacterial properties depending on the used mordanting agent and the bacteria tested on.*

*Keywords: wool, textile, natural dye, antibacterial, pine, pine cone*

## INTRODUCTION

Color pervades all aspects of our lives, influencing our moods and emotions [1]. The coloring of textiles for value addition, look and desire of the customers was anciently initiated by using colors of natural source [2]. It is an ancient art and it was primitively managed by sticking plants to fabric or rubbing crushed pigments into cloth [3]. But the introduction of synthetic dyes led to an almost complete replacement of natural dyes because of the several advantages [4]. The effluents from finishing processes contain high concentrations of biologically difficult-to-degrade or even inert auxiliaries and chemicals and there is pressure on dye manufacturers to develop dyes that can reduce environmental problems [5–6]. Because of the increased environmental awareness to avoid some hazardous synthetic dyes, the use of natural dyes has become a matter of significant importance [7]. In this respect this study was aimed to show the usability of pine cones for coloration of wools and the antibacterial efficiencies of the dyed samples. Anatolian black pine is one of the most common and important forest tree species in Turkey [8]. *Pinus nigra* belonging to *Pinaceae* family is grown in west and south regions of Anatolia [9]. Large quantities of pine cones are produced annually throughout

the world, especially in pine plantations grown for the pulp and paper industry [10]. The antimicrobial activity is an important property for some functional fabrics [11]. The large surface area and the ability to retain moisture of textile structures enable microorganisms' growth, which causes a range of undesirable effects, not only on the textile itself, but also on the user [12]. So, there is a great demand for antimicrobial finishes of textiles to control the growth of microorganisms [13]. There are several studies available on usability of different natural dye sources to ensure antibacterial efficiencies. For example, Gupta et al. (2004) were tested eleven natural dyes against three types of Gram-negative bacteria and they were declared that seven of them showed activity against one or more of the tested bacteria [14]. Şapcı et al. (2017) reported antimicrobial and antifungal activity of fabrics dyed with *viburnum opulus* and onion skins [15]. In other study, Khan et al. (2012) were examined the effect of *Rheum emodi* L. as dye and its dyed wool yarns against two bacterial and two fungal species. They were declared that the dyed samples showed very effective antimicrobial properties [16]. Likewise, Singh et al. (2005) tested four natural dyes against common pathogens and found *Quercus infectoria* dye indicating the best antimicrobial activity [17].

## MATERIAL AND METHODS

### Material

In the experiments, 100% woolen fabric was used in a 2/1 twill construction with a weight of 160 g/m<sup>2</sup>. The fabrics were in pretreated form and ready for dyeing process so no additional process was conducted prior to dyeing.

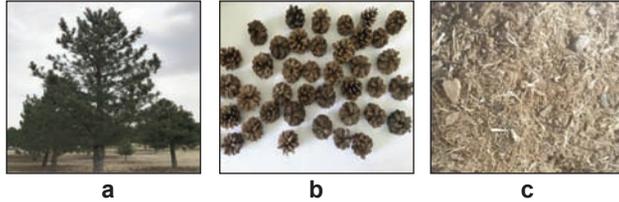


Fig. 1. (a) Black pine (*Pinus nigra*) tree; (b) The cones; (c) The milled cones

As a natural dye source, the idle cones (figure 1, b) of the black pine (*Pinus nigra*) tree (figure 1, a) that grows in Central Anatolia region is used. The cones were collected and dried up in the shadow after they completed the developmental period and poured. These dried cones were then milled (figure 1, c) and these milled cones were used in the dyeing of the wool fabrics.

### Method

The dyeings were conducted in a laboratory type sample dyeing machine according to exhaustion method. In dyeing procedure firstly the dye bath was adjusted to pH 7 and then the fabric, natural dye source and mordanting agent have been added to this bath as seen from the figure 2.

The dyeing and mordanting of the fabrics has been managed simultaneously and as mordanting agents; 3% Copper (II) sulfate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ), 3% tin (II) chloride ( $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ ), 3% iron (II) sulfate ( $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ ), 3% potassium dichromate ( $\text{K}_2\text{Cr}_2\text{O}_7$ ) or 20% Alum ( $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ ) have been used. Moreover, dyeings without use of any mordanting agent have been tested too. The natural dye sources used in the experiments were directly added to the dye bath without any previous extraction in three different concentrations: 1:0.5; 1:1; 1:2 (fabric to natural dye source ratio). During the dyeing procedure the liquor ratio was adjusted to 1:50 (fabric to dye bath ratio). After the dyeing process, the samples were allowed to dry at room temperature following the washing process

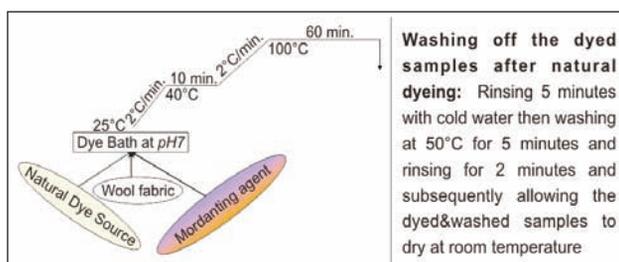


Fig. 2. Natural dyeing procedure

and then various measurements were conducted to them.

CIE L \* a \* b \* values and color efficiencies (K/S) of the dyed fabric samples were measured to evaluate the usability of the pine cones as a natural dye source. Konica Minolta 3600d spectrophotometer was used for this purpose. Moreover, the samples were photographed by scanning the samples to better observe the colors obtained. In addition, after dyeings the samples were evaluated for light fastness (according to ISO 105-B02) [18] and washing fastness (according to ISO 105-C10 standard) [19]. Moreover to see the effect of pine cones in terms of the antibacterial activity, the samples dyed with pine cones at the highest dye concentration of the 1:2 have been analyzed in terms of the antibacterial efficiencies.

The antibacterial test method used in this study is described in detail elsewhere [20]. **For determination of antibacterial effect of the fabrics;** the naturally dyed samples were tested against Gram-negative bacteria (*Escherichia coli* ATCC 25922) and Gram-positive bacteria (*Staphylococcus aureus* ATCC 29213) according to ASTM E 2149 01 standard [21].

The antibacterial efficiency of the samples was measured by using the equation presented below. This equation represents the bacteria reduction (%) caused by the contact with the sample for 24 hours.

$$\text{Bacteria Reduction (\%)} = 100 \times (BC_0 - BC_{24}) / BC_0$$

$BC_{24}$ : Bacteria concentration (CFU/ml) of the jar after "24 hours" contact time with the naturally dyed sample.

$BC_0$ : Bacteria concentration (CFU/ml) of the jar at "0" contact time (before the addition of the naturally dyed sample).

## RESULT AND DISCUSSION

In order to talk about the usability of any vegetable source in textile dyeing, this source should be able to color the textile materials as competently and exhibit sufficient fastnesses at the same time. In this context, the colors obtained after dyeings in the framework of the experiments were measured firstly and collected in table 1. Table 1 also contains the scanned photos of the samples too.

It was generally seen from the table 1 that the amount of dyestuff used in obtaining different colors is not important and that different colors can not be obtained by changing the amount of pine cone used in dyeing. On the other hand, it has been observed that the color efficiency and the lightness-darkness values ( $L^*$ ) change with the change in the concentration of the natural dye source. The increase in  $L^*$  value gives the lightness of the color, while the decrease indicates that the color is darker. The  $L^*$  value ranged from 48.55 to 79.88 for the samples of woolen fabrics dyed with pine cone. When the  $L^*$  values were taken into account it was found that a reduction in  $L^*$  values could come across in case of

THE COLORS AND COLOR VALUES OF THE FABRICS DYED WITH PINE CONES								
Natural dye source concentration	Mordanting agent	K/S	CIE L*a*b* (D65)					Scanned samples
			L*	a*	b*	C*	h°	
1:0.5	<i>No mordanting agent</i>	2.42	60.34	12.72	16.47	20.82	52.32	
1:1	<i>No mordanting agent</i>	3.26	57.53	13.9	18.16	22.86	52.57	
1:2	<i>No mordanting agent</i>	4.21	56.81	12.78	20.58	24.23	58.15	
1:0.5	<i>Copper (II) sulfate</i>	7.52	52.69	5.67	24.15	24.81	76.79	
	<i>Tin (II) chloride</i>	1.3	79.88	2.12	22.1	22.2	84.52	
	<i>Iron (II) sulfate</i>	2.54	62.29	9.63	18.59	20.93	62.61	
	<i>Potassium dichromate</i>	2.79	65.21	5.88	21.16	21.97	74.46	
	<i>Alum</i>	1.55	72.77	4.95	19.42	20.04	75.7	
1:1	<i>Copper (II) sulfate</i>	8.02	51.57	7.78	25.03	26.21	72.74	
	<i>Tin (II) chloride</i>	1.85	73.65	9.05	23.05	24.76	68.56	
	<i>Iron (II) sulfate</i>	3.83	56.73	11.88	19.57	22.89	58.75	
	<i>Potassium dichromate</i>	2.83	63.61	4.69	19.62	20.17	76.56	
	<i>Alum</i>	2.06	68.7	7.34	20.38	21.66	70.2	
1:2	<i>Copper (II) sulfate</i>	9.57	48.55	9.67	25.27	27.06	69.05	
	<i>Tin (II) chloride</i>	2.96	69.9	6.52	25.93	26.74	75.9	
	<i>Iron (II) sulfate</i>	4.55	56.84	10.45	21.81	24.18	64.39	
	<i>Potassium dichromate</i>	3.24	63.26	6.59	21.98	22.95	73.32	
	<i>Alum</i>	2.71	65.72	7.1	22.12	23.24	72.21	

the increase the used natural dye source concentration in dyeings. This can be shown as proof that the color becomes darker with the increase of used dye concentration. The increase in the K/S value indicates that the color efficiency is high while the decrease shows that the color efficiency is low. The K/S value for woolen fabric samples dyed with pine cone varies between 1.30 and 9.57. When the color efficiencies of the dyed samples were compared, it was found that the K/S values increase as the dyeing concentration increases and this tendency is also seen in the dyeings made with different mordanting agents as well. If an example of the dyeing done without use of any mordanting agent want to be given; it could be seen that the color efficiency for the 1:2 dye concentration was 4.21, and the color efficiency for the dyeing concentration 1:0.5 was 2.42. From table 1, it can be seen that the variation in dyeing efficien-

cy was mainly due to the natural dye source concentration and mordanting agent type. Along with the increase in dyeing concentration, an increase in color efficiency is an expected feature. Moreover, it has been observed that the highest color efficiency could be achieved by using copper (II) sulfate as a mordanting agent and the lowest color efficiencies were generally observed with tin (II) chloride and alum. On the other hand, the effect of the mordanting agents on obtaining different colors was obvious. When table 1 has been examined, it could be easily seen that different colors and shades can be obtained with the use of different mordanting agents. It was seen from table 1 that yellow, khaki, pinkish orange and brown colors and tones could be obtained in the dyeings made with the use of pine cones. By taking the hue angles ( $h^\circ$ ) in table 1 as reference, it was determined that the hue angles were

LIGHT AND WASHING FASTNESS OF FABRICS DYED WITH PINE CONES									
Mordanting agent	Natural dye source concentration								
	1:0,5			1:1			1:2		
	Light	Washing		Light	Washing		Light	Washing	
<i>No mordanting agent</i>	3/4	C.C.	5	3/4	C.C.	5	4	C.C.	5
		Sta.	5		Sta.	5		Sta.	5
<i>Copper (II) sulfate</i>	3/4	C.C.	5	3	C.C.	5	4	C.C.	5
		Sta.	5		Sta.	5		Sta.	5
<i>Tin (II) chloride</i>	4	C.C.	5	4/5	C.C.	5	4/5	C.C.	5
		Sta.	5		Sta.	5		Sta.	5
<i>Iron (II) sulfate</i>	3/4	C.C.	5	3/4	C.C.	5	3/4	C.C.	5
		Sta.	5		Sta.	5		Sta.	5
<i>Potassium dichromate</i>	3/4	C.C.	5	4	C.C.	5	4	C.C.	5
		Sta.	5		Sta.	5		Sta.	5
<i>Alum</i>	4	C.C.	5	4	C.C.	5	4/5	C.C.	5
		Sta.	5		Sta.	5		Sta.	5

Sta.: Staining on wool; C.C.: Color Change

around 52 in all mordant free dyeings. For example, in a mordant free dyeing process at a 1:1 dye concentration, the  $h^\circ$  was 52.57,  $a^*$  was 13.9 and  $b^*$  was 18.16 and the color is perceived as a pinkish orange. After dyeing at 1:0.5 dye concentration by using copper sulphate mordanting agent, the  $h^\circ$  value of the obtained color was 76.79, and  $a^*$  and  $b^*$  values were measured as 5.67 and 24.15 respectively. Therefore, a yellowish and a lesser red color than the color obtained from the mordant free dyeing emerged and a khaki color was observed. In dyeings with tin chloride mordanting agent at 1:0.5 natural dye source concentration, the obtained color was perceived as in light yellow shade. The  $a^*$  and  $b^*$  values were measured 2.12 and 22.1 respectively and  $h^\circ$  angle was found 84.52. As can be seen, different colors and color tones could be obtained with the pine tree cones using different mordanting agents. It is possible to obtain different colors with a single natural dye source, for example herein with pine tree cones, thanks to this unique feature of the natural dyes.

Another important parameter in terms of textile dyeing is the fastnesses, which were also tested for the samples dyed with milled pine tree cones. Table 2 shows the light fastness and wash fastness of the samples.

When table 2 has been examined, it was seen that mordant types and natural dye source concentration were effective in terms of light fastness values. But generally in coloring made by using pine tree cones, the light fastnesses were medium-good. If a generalization is made, it was found that the increase of the natural dye source concentration generally means the increase of the light fastness values. The reason of this was thought to be due to the high tolerability of the dyestuff portion of the light-decayed in dark colors. When the washing fastness values were

checked, very good/excellent washing fastness results were obtained. All wash fastness results have a value of 5 for staining and color change. This showed that wool fabrics dyed with pine cone natural dye source have no staining or discoloration during washing process. In other words, it could be said that the mordant material and the dyeing concentration have no effect on washing fastness for dyeing with pine cone natural dye source and that sufficient fastness results could be obtained.

As detailed in the method part of the study, the wool fabrics dyed with pine cones at concentration of 1:2 were analyzed in terms of antibacterial efficiencies against *E. coli* and *S. aureus*. From table 3, it was observed that there was no bacteria reduction in undyed wool fabrics.

Table 3

THE ANTIBACTERIAL ACTIVITY OF WOOL FABRICS DYED WITH PINE CONES AT A CONCENTRATION OF 1:2		
	Bacteria reduction (%)	
	<i>S. aureus</i>	<i>E. coli</i>
Undyed wool fabric	-	-
Wool fabric dyed without use of any mordanting agent	99.7	-
Wool fabric dyed with use of Copper (II) sulfate	99.9	99.9
Wool fabric dyed with use of Tin (II) chloride	99.9	-
Wool fabric dyed with use of Iron (II) sulfate	99.9	99.9
Wool fabric dyed with use of Potassium dichromate	50	-
Wool fabric dyed with use of Alum	99.9	99.9

These antibacterial test results clearly showed that depending on the tested bacteria and used mordanting agents the reduction (%) of the bacteria population has differed but generally it can be told that natural dyeing with pine cones ensured better antibacterial efficiency against *S. aureus*. As seen from the table 3, except the samples dyed in presence of Potassium dichromate mordanting agent the bacterial reduction of *S. aureus* is higher than 99% so not only the effect of the mordanting agents but also the extracts of pine cones have an antibacterial efficiency against *S. aureus*. However when the antibacterial efficiency against Gram-negative bacteria (*E. coli*) has been examined, it was observed that natural dye source of pine cones have not antibacterial efficiency against *E. coli* and only if the Copper (II) sulfate, Iron (II) sulfate or alum based natural dyed samples exhibit significant bacterial reduction of *E. coli*. So this antibacterial efficiency against *E. coli* is more related to the used mordanting agent and the tested natural dye source solely did not cause a positive effect on it.

## CONCLUSIONS

In this study, it was aimed to show the usability of pine cones in natural dyeing of the wool fabrics. As a

result of the dyeing experiments, it has been determined that the pine tree cones can bring out different colors with the use of different mordanting agents. When a summary is made by looking at the generic; the color of pinkish orange was obtained in the mordant free dyeings. Moreover, depending on the type of mordant in the mordant based dyeings; yellow-beige, khaki, pinkish orange and brown tones had been observed. In addition, it was observed that same washing but different light fastnesses can be obtained with the use of different mordanting agents. Beyond the use of pine cones as a natural dye source, the effect of pine cone based natural dyeing has also been analyzed in terms of antibacterial efficiencies. The dyed samples' antibacterial efficiencies were tested against *E. coli* and *S. aureus*. It was found that while the fabrics not dyed did not exhibit antibacterial efficiency, the samples dyed with pine cones has shown an antibacterial efficiency depending on the used mordanting agent and the bacteria tested. But briefly, wool fabrics dyed with pine cones in presence of Copper (II) sulfate, Iron (II) sulfate or alum showed antibacterial efficiency for both *E. coli* and *S. aureus* and the mordant free dyed samples were showed an antibacterial efficiency against only *S. aureus*.

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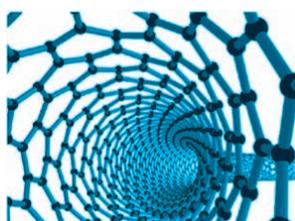
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# Investigating pull-out characteristics of tubular fabrics with different tightnesses in drilling and sampling process

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## REZUMAT – ABSTRACT

### Investigarea caracteristicilor de tracțiune ale țesăturilor tubulare cu diferite grade de compactitate în procesul de perforare și eșantionare

În acest studiu, caracteristicile de tracțiune ale țesăturilor tubulare cu patru grade diferite de compactitate au fost investigate prin intermediul sistemului de testare prin tracțiune automată. Între timp, principiul fenomenului lipire-alunecare în timpul procesului de perforare și de eșantionare a fost clarificat. Rezultatele experimentale obținute de la acest sistem au indicat că forța de tracțiune a țesăturilor tubulare a prezentat tendința de creștere în stadiul inițial, după care de scădere și, în cele din urmă, a avut tendința spre o valoare constantă non-zero. De asemenea, s-a demonstrat că, cu cât este mai mare compactitatea țesăturilor tubulare, cu atât este mai mare forța de susținere. În plus, conceptul de coeficient de netezire a fost introdus pentru a înțelege mai bine gradul de dificultate al țesăturilor tubulare care sunt extrase din conducta rotundă. Calculele au arătat că o compactitate mai mică a conferit o performanță mai bună la netezire și, în mod natural, coeficientul de netezire a fost mai mare.

**Cuvinte-cheie:** forța de tracțiune, coeficient de netezire, țesături tubulare, grade diferite de compactitate, fenomen de lipire-alunecare, perforare și eșantionare

### Investigating pull-out characteristics of tubular fabrics with different tightnesses in drilling and sampling process

In this paper, the pull-out characteristics of tubular fabrics with four different tightnesses were investigated via our self-designed pull-out testing system. Meanwhile, the principle of stick-slip phenomenon during the drilling and sampling process was clarified. Experimental results obtained from this system indicated that pull-out force of tubular fabrics presented a tendency of rising in the initial stage then decreasing, and finally tended towards a constant non-zero value. It also revealed that the bigger the tightnesses of tubular fabrics are, the larger its bearable force is. Moreover, the concept of smoothness coefficient was introduced to further understand the degree of difficulty that tubular fabrics pulled out from round pipe. The calculations showed that smaller tightnesses gave better smoothness performance, and naturally the smoothness coefficient was bigger.

**Keywords:** pull-out force; smoothness coefficient; tubular fabrics; different tightnesses; stick-slip phenomenon; drilling and sampling

## INTRODUCTION

In recent years, with the outstanding properties of non-laddering and seamless comparable to conventional fabrics, there has been an increasing interest in tubular fabrics for practical applications in the fields of medical materials, pipeline rehabilitation [1–5], etc. Besides, tubular fabrics used as thin strip sampling apparatuses and sample collection bags to implement stratigraphic drilling and to collect samples of soils and small solids have also attracted much attention in the fields of space technology, geological assay and archaeological discovery [6–7].

Because the interaction relationship between tubular fabrics and round pipe in actual use is very complex, research on the force of tubular fabrics pulled out from round pipe in drilling process is needful and the development of corresponding equipment or device is also indispensable. Moreover, there are many factors that are impacting on the pull-out process of tubular fabrics along the wall of round pipe. For the purpose of better analyzing movement condition of tubular fabrics along the wall of round pipe, Zhao

Chao revised Euler's formula. In addition, a test device for measuring pull-out force of tubular fabrics was designed based on Instron instrument, it was found that the coated length of tubular fabrics and positive pressure to round pipe had positive correlation with pull-out force [8]. Qian Xiuyang and colleagues measured the friction coefficient between tubular fabrics and the wall of round pipe applying elastic element and elastic pressure method [9]. Through establishing mechanical model of drilling process, Luo Shihua explored the relationship between the stress of tubular fabrics at the end of round pipe and the length that coated. As well, dynamic numerical simulation was conducted to investigate pull-out process of tubular fabrics based on anisotropic constitutive model of materials, they also discovered that wall thickness and inner diameter of round pipe and the thickness of tubular fabrics all influenced pull-out process, in turn, made it unfavorable for tubular fabric to pull out from round pipe [10]. However, a specialized system for testing pull-out force of tubular fabrics during drilling and sampling hasn't been developed and the principle of

volatility in this process does not reflect reasonably and most of these evaluation standards concentrated on the physical properties of tubular fabric or round pipe, characterization techniques and analysis method aimed at the structure of tubular fabrics were less reported.

Pull-out process of tubular fabrics is a nonlinear deformation process, which has very complex influence factors. If there are failure conditions of self-locked or blocked, it will make the drilling and sampling process difficult to carry out. Hence, investigating pull-out characteristics of tubular fabrics in drilling and sampling process is significant. This paper deals with the fabrication of four different tightnesses tubular fabrics and design of pull-out system so as to investigate pull-out force of tubular fabrics along the wall of round pipe, and then to explore smoothness coefficient.

## EXPERIMENTAL DETAILS

### Nomenclature

The parameters used in the derivation process are listed in table 1.

### Manufacturing of tubular fabrics

Herein, the basic parameters of materials used to fabricate tubular fabrics are shown in table 2. The diameters  $d$  of the four tubular fabrics are identically equal to 24 mm.

The parameters of the four tubular fabrics with different tightnesses and the corresponding calculative formulas [11] are shown in table 3.

As shown in figure 1, a and 1, d, when weaving begins, according to the weave design in figure 1, c, under the action of pneumatic pressure, picking rod is drawn out with the special shuttle in figure 1, b connected from picking mouth, passes through shed into the opposite card slot, then picking rod comes back into picking mouth left behind the shuttle in card slot, and the first weft insertion is completed. When next shed is formed, picking rod is thrown out from picking mouth, and passes across shed towards card slot, where shuttle is released and clamped with oncoming picking rod, then picking rod carries shuttle back again into picking mouth, and the second weft insertion is accomplished. Picking rod and shuttle repeat this process again and again thereby weft yarns are inserted and tubular fabrics are manufactured [12].

### Self-designed pull-out testing system of tubular fabrics

As shown in figure 2, a pull-out system for measuring pull-out force of tubular fabrics is self-designed. The system mainly consists of two modules, one is drilling and sampling mechanism in figure 2, b and the other is a signal receiving system [13].

Before the movement of drilling and sampling mechanism, hollow bit is in contact with soil particles but not drills, one end of tubular fabric is sheathed on the outer wall of round pipe, the other end is inverted into the corner and clamped by a rope. Round pipe is placed into hollow auger drill pipe. During drilling and sampling, lifting plate moves downward along the four guide rails with the traction of adjusting speed

Table 1

Symbols	Meaning	Symbols	Meaning
$N_D$	Fineness of Kevlar filament	$Z$	Cycle numbers of basic weave
$\delta_y$	Density of Kevlar filament	$S_w$	Progression of weft direction
$d_y$	Diameter of Kevlar filament	$E_z$	Total tightness
$M_j$	Total warp ends	$E_j$	Warp tightness
$P_j$	Warp density of a single layer	$E_w$	Weft tightness
$R_j$	Ends of basic weave cycle	$P_w$	Weft density of a single layer

Table 2

Materials	$N_D$ (D)	$\delta_y$ (g/cm <sup>3</sup> )	$d_y$ (mm)	$d_y = 0.01189 \sqrt{\frac{N_D}{\delta_y}}$
Kevlar filament	800	1.44	0.28	

Table 3

THE PARAMETERS OF TUBULAR FABRICS				
Tubular fabrics	$M_j$ (roots)	$P_j$ (roots/10 cm)	$E_z$ (%)	$M_j = \pi d \cdot P_j, \quad M_j = R_j \cdot Z + S_w$ $E_z = E_j + E_w - \frac{E_j \cdot E_w}{100}$ $= d_y \cdot P_j + d_y \cdot P_w - \frac{d_y \cdot P_j \cdot d_y \cdot P_w}{100}$
1#	167	220	85.3	
2#	151	200	80.6	
3#	137	180	75.4	
4#	121	160	69.5	

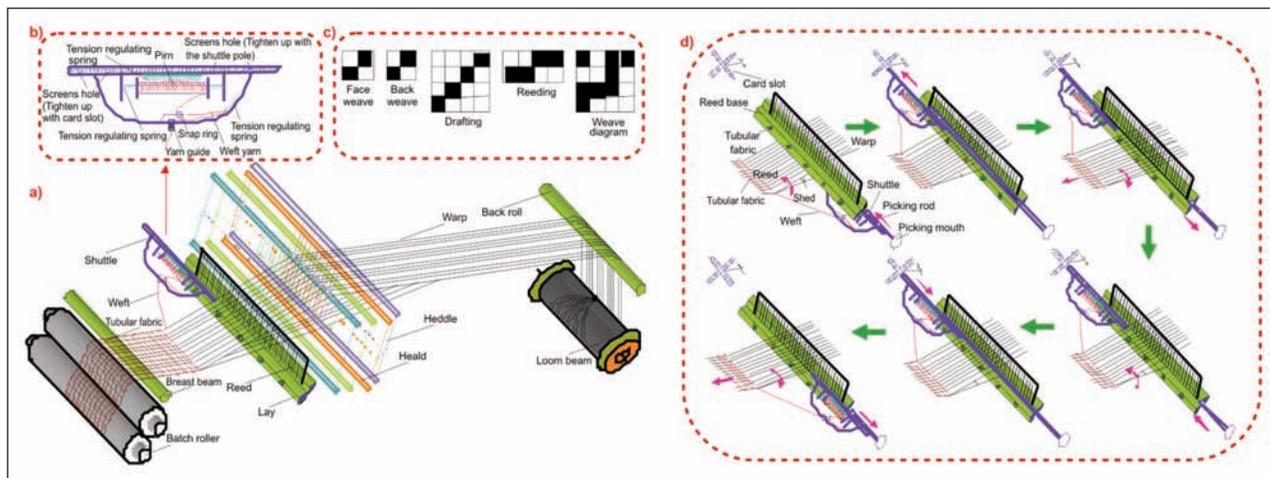


Fig. 1. (a) Weaving process of tubular fabric; (b) special shuttle; (c) diagram of weave design; (d) process of picking

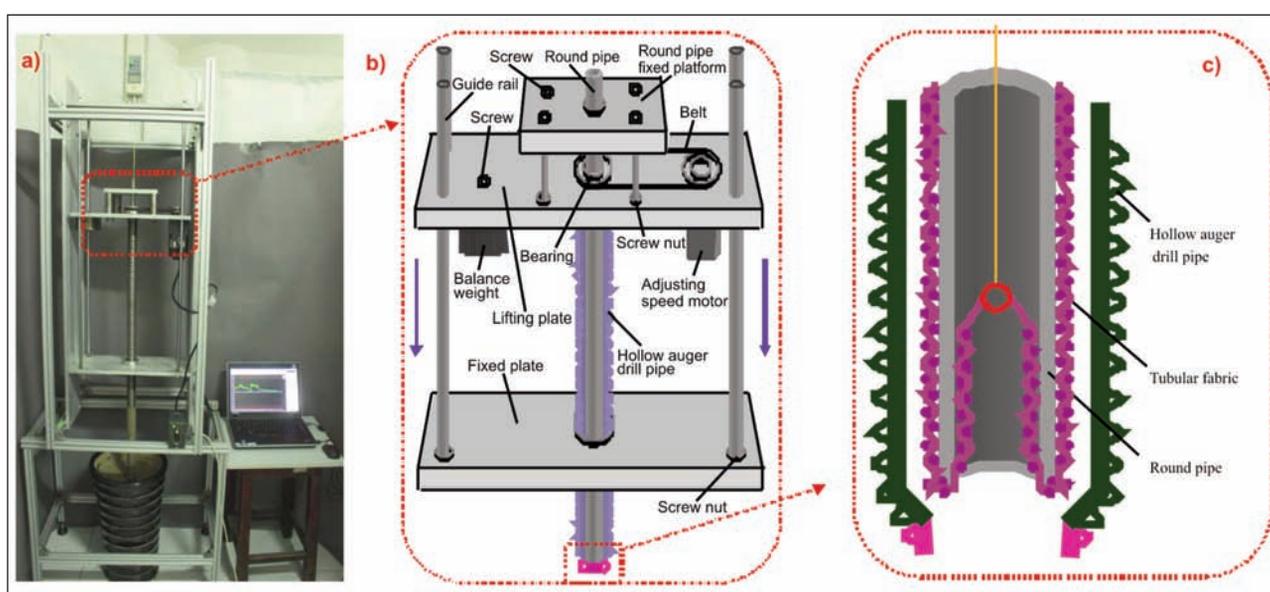


Fig. 2. (a) Self-designed pull-out system of tubular fabrics; (b) Drilling and sampling mechanism; (c) Location relationship of tubular fabric, round pipe and hollow auger drill pipe

motor, and drives hollow auger drill pipe to make spiral movement downward together, but round pipe only does vertical motion under the action of fixed platform. Along with tubular fabric completely entered into the inner of round pipe, soil particles are coated, as demonstrated in figure 3.

## RESULTS AND DISCUSSION

### The principle of stick-slip phenomenon

During drilling, the upper end of rope is fixed, round pipe moves downward uniformly with speed  $v_0$ . For ease of analysis, it is assumed that round pipe is stationary and rope does upward movement relative to round pipe at a speed of  $v_0$ , as shown in figure 4, a. As shown in figure 4, b, after drilling, rope is gradually tightened, force  $F$  increases but not yet in excess of maximum static friction force  $f_{max}$ , hence tubular fabric is still in static state. As drilling proceeds,  $F$  progressively increases till to exceed  $f_{max}$  at point A,

when tubular fabric begins to move and is gradually flattened out from folded state and friction force  $f$  therewith decreases. Then, tubular fabric does accelerated movement that acceleration  $a$  increases gradually and speed  $v$  aggrandizes accordingly. At point B,  $v$  is equal to  $v_0$ , thus, rope is tightened,  $F$  and  $a$  reached maximum value. However, with disappear of folded state at point C,  $f$  is minimum and equal to  $F$ . For the fact that  $v$  is still greater than  $v_0$ , hence  $F$  gradually reduces. However,  $f$  becomes bigger with the increase in length of tubular fabric inserted into the inner of round pipe. Thus,  $v$  begins to reduce, till to point D. Considering that  $F$  is less than  $f$ ,  $v$  continuously reduces till to zero, when tubular fabric stops moving and  $f$  turns into static friction force. Then, as round pipe continues downward, it comes into next cycle movement [14–15].

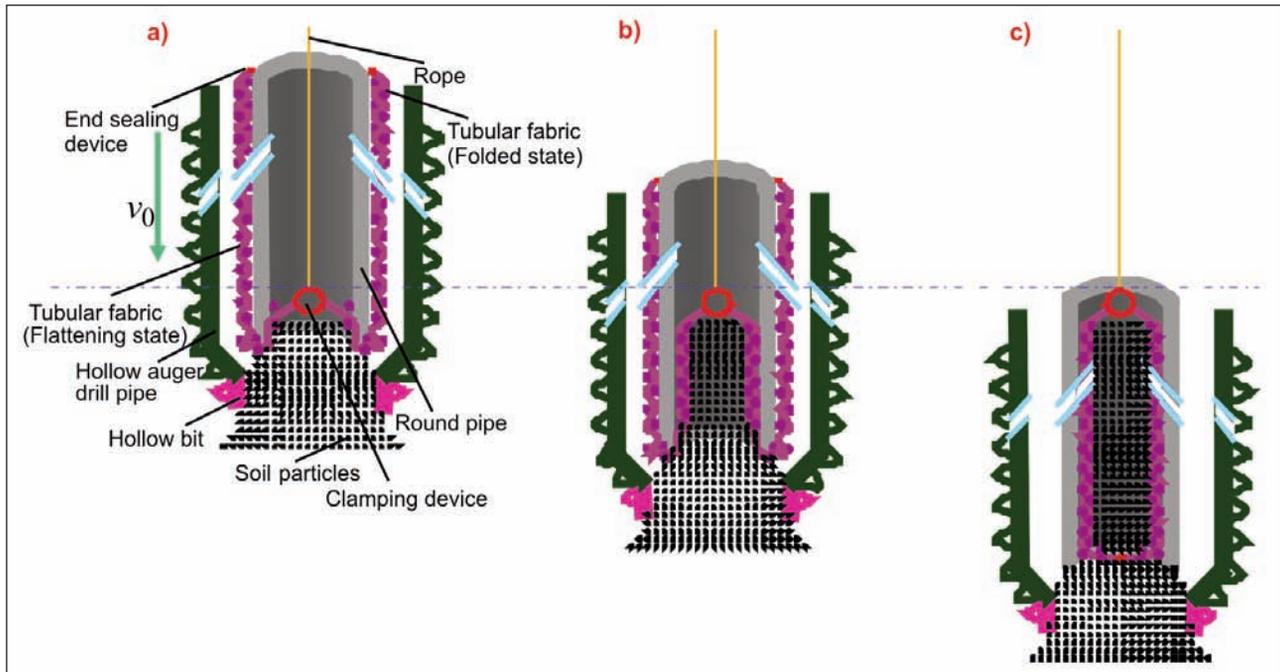


Fig. 3. The principle and process of drilling and sampling: (a) folded state exists; (b) folded state completely vanishes; (c) the end of drilling and sampling process

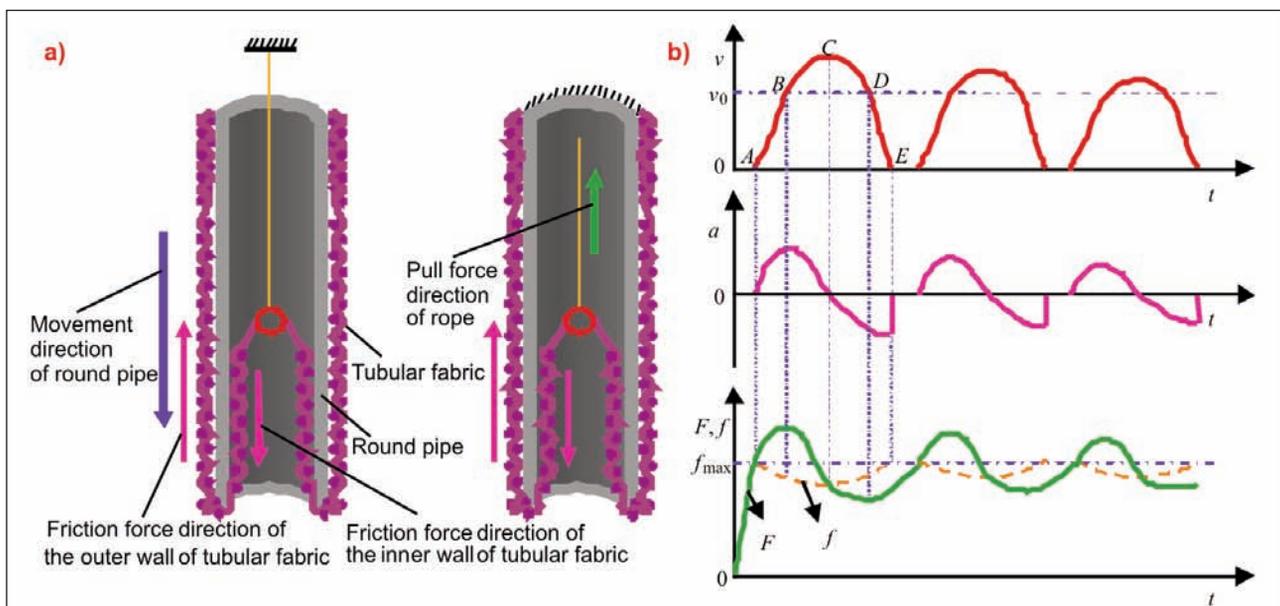


Fig. 4. (a) Equivalent diagrams of the motion of round pipe and rope; (b) the principle of stick-slip phenomenon

### The concept of smoothness coefficient

In order to further explore and analyze the interaction force between tubular fabric and the wall of round pipe, a concept of smoothness coefficient is introduced and the formula is [12]:

$$S = 1 - \frac{F_{max}}{F_b} \quad (1)$$

Where,  $S$  is smoothness coefficient of tubular fabrics,  $F_{max}$  – maximum pull-out force obtained from the experiment,  $F_b$  – breaking strength of tubular fabrics obtained from Instron instrument.

Smoothness coefficient is the degree of difficulty that tubular fabrics pulled out from round pipe and the values range from 0 to 1. The greater the value, the smaller the effect of tubular fabric on the wall of round pipe, that is to say, the characteristic of smoothness is better.

### Pull-out force-time and smoothness coefficient-time curves

As can be seen from figure 5, a, the tendency of the four curves is approximately coincident and every curve demonstrates a tendency that first rises and then falls. During drilling and sampling, because the existence of stick-slip phenomenon, it can be clearly

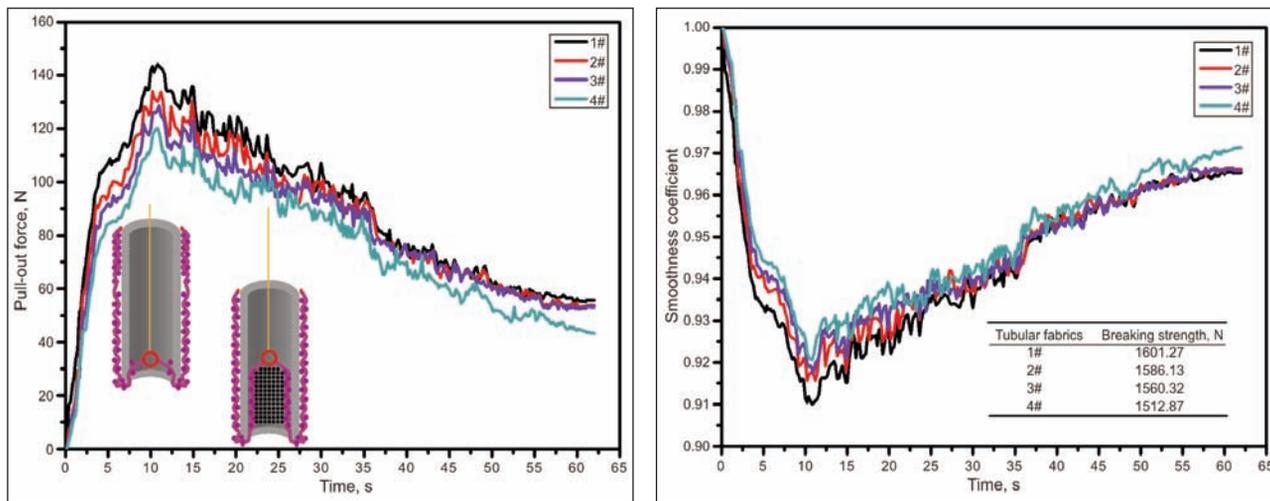


Fig. 5. (a) Pull-out force-time curves; (b) Smoothness coefficient-time curves

seen that pull-out force-time curves are highly volatile in the whole process. Moreover, it can also be seen that forces increase quickly compared to time from zero to a maximum value, after which point, forces decrease and finally move toward a constant value that is greater than zero. Actually, the process can be divided into two stages, folded state exists (about 0~11 s) and folded state vanishes (about 11~62 s), so pull-out forces of tubular fabrics presented a tendency of rising in the initial stage then decreasing, and finally tended towards a constant non-zero value. Pull-out force reaches maximum value when tubular fabrics in folded state are completely dismissed.

It can also be found that high tightnesses exhibit higher pull-out forces compared to low ones, however, an opposite way to smoothness coefficients, as shown in figure 5, b. The differences can be attributed to the construction of tubular fabric, in reality, the interaction force between tubular fabrics and round pipe is the squeezing and friction between filaments of tubular fabrics and the wall of round pipe, hence, the bigger the filaments contact with the wall, the more intense the interaction [16–18]. High tightnesses have a big contact area, which leads to large

pull-out force consequently. There is also another phenomenon from figure 5, b, smoothness coefficients all exceed 0.90, which gives a signal that characteristics of smoothness is good for tubular fabric when pulled out from round pipe.

## CONCLUSIONS

In conclusion, this paper manufactures a high-performance tubular fabric and designs a pull-out system, with which a pull-out test was conducted. Because of the existence of stick-slip phenomenon, when tubular fabrics pulled out from round pipe, pull-out forces are highly volatile. From folded state to completely flattening state, pull-out forces presented a tendency of rising in the initial stage then decreasing, and finally tended towards a constant non-zero value. The results also indicate that high tightnesses have higher pull-out forces compared to low ones, but an opposite way to smoothness coefficients.

## ACKNOWLEDGEMENTS

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# Sunlight exposure: the effects on the performance of paragliding fabric

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## REZUMAT – ABSTRACT

### Expunerea la soare: efectele asupra performanței țesăturii pentru parapante

Acest studiu a fost conceput pentru a explora relația dintre expunerea la soare și proprietățile mecanice ale țesăturilor pentru parapante, care au diferite culori, densități, tipuri de finețe și materiale de acoperire. În acest studiu s-au prezentat 5 culori diferite de țesături pentru parapantă (roșu, turcoaz, albastru închis, portocaliu și alb) care au fost expuse la lumina puternică a soarelui, timp de 150 de ore în timpul verii, de la ora 9:00 până la 3:00 p.m., timp de 5 zile pe săptămână, 5 săptămâni. Înainte și după procesul de îmbătrânire din cauza radiațiilor UV, s-au efectuat testele de permeabilitate la aer, rezistență la tracțiune, rezistență la sfâșiere și rezistență la plesnire. Rezultatele testelor au fost, de asemenea, evaluate folosind metode statistice. Conform rezultatelor, s-a constatat că țesătura turcoaz a avut cel mai ridicat grad de estompare dintre țesăturile studiate. S-a constatat că există o scădere semnificativă a proprietăților mecanice ale țesăturilor după expunerea la soare. După îmbătrânire, țesăturile devin considerabil mai puțin rezistente în ceea ce privește proprietățile mecanice din cauza degradării atât a colorantului, cât și a structurii macromoleculare a fibrei.

Cuvinte-cheie: expunere la soare, fotodegradare, fotoîmbătrânire, proprietăți mecanice, țesătură pentru parapante

### Sunlight exposure: the effects on the performance of paragliding fabric

This study was designed to explore the relationship between sunlight exposure and the mechanical properties of paragliding fabrics which have different colors, densities, yarn counts, and coating materials. This study exposed 5 different colors of paragliding fabrics (red, turquoise, dark blue, orange, and white) to intense sunlight for 150 hours during the summer from 9:00 a.m. to 3:00 p.m. for 5 days a week for 5 weeks. Before and after the UV radiation aging process, the air permeability, tensile strength, tear strength, and bursting strength tests were performed. Test results were also evaluated using statistical methods. According to the results, the fading of the turquoise fabric was found to be the highest among the studied fabrics. It was determined that there is a significant decrease in the mechanical properties of the fabrics after sunlight exposure. After aging, the fabrics become considerably weaker in the case of mechanical properties due to the degradation in both the dyestuff and macromolecular structure of the fiber.

Keywords: sunlight exposure, photodegradation, photoaging, mechanical properties, and paragliding fabric

## INTRODUCTION

Textile materials are usually designed and produced to be used in environmental conditions, outside, where the fabrics are subjected to different sources of radiation. One of the most important sources of radiation found in the environment is UV radiation. It can affect many properties of the materials, including the mechanical behavior. Exposure of UV radiation was found to affect the supermolecular structure of the fibers. Changes in the internal structure of the fibers lead to variation of the color absorption behavior [1]. As it is known dyes absorb UV light, which helps in reducing exposure. Darker colors tend to absorb more UV light than lighter colors, including whites and pastels, but vivid colors such as red can also substantially absorb UV rays [2]. The more vivid the color, the greater the protection; a bright yellow shirt is more protective than a pale one. But even a pale fabric can offer good protection if the weave, material, weight, etc. are effective at keeping out UV light. And many white fabrics have “optical whitening agents”, chemical compounds that strongly absorb UV radiation, especially UV-A [3–4].

Damage by UV radiation is commonly the main reason for the discoloration of dyes and pigments, weathering, yellowing of plastics, loss of gloss and mechanical properties (cracking), sun burnt skin, skin cancer, and other problems associated with UV light. The manufacturers of paints, plastics, contact lenses, and cosmetics have a great interest in offering products that remain unaltered for long periods under conditions of light exposure [5–8]. Exposure to ultraviolet (UV) radiation may cause significant degradation of many materials. UV radiation causes photo-oxidative degradation which results in breaking of the polymer chains, produces free radical, and reduces the molecular weight, causing deterioration of the mechanical properties leading to useless materials, after an unpredictable time [9–10]. When polymers are used in outdoor applications, the environment negatively influences the service life. This process is called weathering [11–12]. During weathering, the term “lightfastness” becomes a significant indicator for the quality of material. Lightfastness is the ability of a fabric to stand up to light. Dyed fabrics that are exposed to light can, in time, fade or change color. Both natural sunlight and

artificial lights can cause damage to color. In general, light pastel colors fade more easily than dark ones, especially pink and turquoise. But dark colors crock more than light ones [13].

The damage of light depends on the intensity of the light source and the amount of exposure, as well as the properties of the dyestuff. Weather conditions, the season of the year, height above sea level, and the distance from the equator will all affect sunlight intensity [13].

In order to measure lightfastness in the home conditions, aging under sun light exposure could also be performed under direct sun light. All-day exposure is better, however, in this method, the samples need to be placed behind glass and exposed to sun at least from 9:00 a.m. to 3:00 p.m., in which sunlight intensity is the highest. The samples should be placed where shadows from objects in the vicinity will not fall upon them. The test should be repeated for approximately five summer days or eight winter days. Running the test for consistent lengths of time allows for making comparisons between a number of exposures [13].

Drapery fabric, on the other hand, is tested for eighty hours, canopy fabric for 160 hours, and automobile fabrics go up to 300 hours [13].

Paragliding fabrics are used under the exposure of sunlight and it causes photo degradation in the fiber structure, which can cause change in tensile properties and air permeability. Tensile properties have vital importance and air permeability is an indicator of fabric porosity, which directly changes the lift force of air. Since the mechanical properties and air permeability of these fabrics have great importance, the effect of sunlight on the physical properties of paragliding fabrics is an important parameter which should be investigated.

## MATERIALS AND METHODS

### Materials

The experiments were conducted using 5 different paragliding fabrics, which were produced in different densities, yarn counts and colors. They were selected since the most commonly used paragliders are made from them and are commercially available. The

basic properties of the fabrics are summarized in table 1.

As seen in table 1, all fabrics were coated with a special coating material (polyurethane), a thin film of resin, to increase their strength and resistance to solar radiation and abrasion. In addition, the aim is to decrease air permeability. Therefore, coating is an important part that affects the performance of the paragliding fabric. Although there are numerous materials used for this purpose, polyurethane is one of the most used polymers. Polyurethane coated fabric offers advantages over other polymeric coatings such as low-temperature flexibility, overall toughness (very high tensile, tear strength, and abrasion resistance, requiring much less coating weight), and softer handle [14]. There are various types of it and some polyurethane based coating materials exhibit high strength combined with high flexibility, good cold flexibility, and high elasticity, however, it has a poor to oxygen and light [15]. They could develop extensive yellowing and photo-oxidation in the sunlight which is an important disadvantage during usage [16–17]. One of the solutions to avoid this disadvantage is to apply a silicone layer on the polyurethane coating. Silicones are chemically inert and maintain their properties for a long time at temperature extremes [14]. Silicone elastomers and silicone dispersions consist of polydimethylsiloxane with reactive groups. They are water-repellent and, dirt-repellent, thermally stable between –50 and +200 °C, flame retardant, have a high resistance to chemicals, and are transparent [15]. There are aging studies that indicate their performance durability over time [14]. Therefore, a combined coating of polyurethane and silicone on a paragliding fabric was included (Turquoise sample) in the study to compare its performance.

### Methods

For the experiment, paragliding fabrics were tested for their physical properties such as, breaking strength, tearing strength, bursting strength, and air permeability. Later, they were exposed to sun light for 150 hours in August. For this purpose, the samples were placed behind glass and exposed to sun from 9:00 a.m. to 3:00 p.m. for 25 days. The solar angle was adjusted to 60° (degrees from vertical), which is

Table 1

	Red	Turquoise	Dark blue	White	Orange
<b>Weft Density (picks/cm)</b>	41.0	22.3	27.2	27.3	26.3
<b>Warp Density (ends/cm)</b>	49.5	24.2	43.5	41.3	48.8
<b>Fabric Density (yarns/cm<sup>2</sup>)</b>	90.5	46.5	70.7	68.6	75.1
<b>Total Mass per Square Meter (g/m<sup>2</sup>)</b>	39.2	44.4	44.1	41.5	55.1
<b>Thickness (mm)</b>	0.132	0.188	0.126	0.202	0.184
<b>Material</b>	PA 6.6	PA 6.6	PA 6.6	PA 6.6	PA 6.6
<b>Construction</b>	Ribstop	Ribstop	Ribstop	Ribstop	Ribstop
<b>Coating Material</b>	Polyurethane	Polyurethane + Silicone	Polyurethane	Polyurethane	Polyurethane

the optimum angle for the solar collectors to have the best solar aging for the system in the selected month (figure 1).

After aging, they were tested again, to determine the change before and after exposure. Tests were performed under standard atmosphere conditions ( $20 \pm 2^\circ\text{C}$  temperature and  $65\% \pm 4\%$  relative humidity).

The tensile properties of the fabrics were measured using a Zwick Z010 Tensile Strength Tester. The breaking strength was conducted according to the EN ISO 13934-1 strip method. Tearing strength was conducted according to the ISO 13937-2 standard. Fabric thickness was measured according to EN ISO 5084 and the bursting strength test was determined according to EN ISO 13938-1. The air permeability test was performed according to the ISO 9237 standard using a Textest FX 3300 air permeability tester with 2.5 kPa pressure through a 5 cm<sup>2</sup> specimen area. The Specular Component Included (SCI) Hunter Lab Ultra Scan PRO Spectrophotometer (Measurement geometry: d8, observation angle: 10°, and light source: D65) was used for color measurement. Each measurement was repeated four times and the average value was recorded. The results were expressed as CIE (Commission Internationale de l'Eclairage) L\*, a\*, and b\* coordinates.

In order to determine the "Strength Loss (SL)" of the fabrics mechanical properties, "Breaking Force Loss", "Tearing Force Loss", and "Bursting Strength Loss" values were calculated by the given formula (equation 1).

$$SL = \frac{\text{Strength (BE)} - \text{Strength (AE)}}{\text{Strength (BE)}} \times 100 \quad (1)$$

SL = Strength Loss, indicating "Breaking Force Loss", "Tearing Strength Loss" and "Bursting Strength Loss";  
Strength (BE) = Strength Before Exposure;  
Strength (AE) = Strength After Exposure.

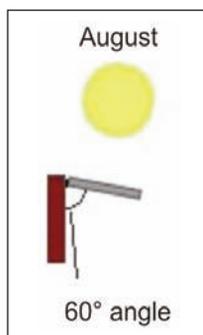


Fig. 1. Solar angle degrees from vertical

## RESULTS AND DISCUSSION

### Statistical evaluation

The breaking strength, bursting strength, and tearing strength values of the fabrics were measured and after all the fabric performance tests, an ANOVA and Student-Newman-Keuls tests were conducted to determine whether the effect of paragliding fabric types on fabric properties is statistically significant at a 95% confidence level ( $p < 0.05$ ). In addition to this, an Independent Samples T-Test was conducted to analyze the differences between the before and after sun light exposure. The results are given in table 2, table 3 and table 4.

The relationships between the mechanical properties and color changes were examined. Since the turquoise sample indicates completely different performance, correlations were performed with and without this sample. The related R<sup>2</sup> values of the correlations were calculated and are given in table 5.

### Results of colour measurement

The results of the color measurements (L\*, a\*, and b\* coordinates) are given in table 6. The overall colour change,  $\Delta E$ , was calculated using the CIE 2000 formula.

Color fading is photo degradation and the color which is seen is based upon the chemical bonds and the amount of light that is absorbed in a particular wavelength. Ultraviolet and infrared rays can break down the chemical bonds and thus fade the colors. It may be more noticeable in brighter and more intense colors [18].

The data in table 2 indicates that, the color change of the turquoise sample was found as the greatest among the samples, when photo aging was carried out. This result was followed with the results of the dark blue, orange, and red samples.

### Results of air permeability

The fabrics were tested for their air permeability under a pressure difference of 2500 Pascal and a test area of 5 cm<sup>2</sup> before and after sunlight exposure. However, none of fabrics was found as air permeable according to the measurement procedure of the TS 391 EN ISO 9237 standard.

Table 2

	Red	Turquoise	Dark blue	White	Orange
Breaking Strength on Warp Direction	0.096	0.028*	0.010*	0.104	0.282
Breaking Strength on Weft Direction	0.000*	0.075	0.041*	0.349	0.379
Elongation on Warp Direction	0.043*	0.063	0.017*	0.008*	0.010*
Elongation on Weft Direction	0.003*	0.025*	0.024*	0.394	0.230
Bursting Strength	0.000*	0.000*	0.003*	0.001*	0.020*
Tearing Strength on Weft Direction (Warp Tearing)	0.002*	0.000*	0.008*	0.005*	0.010*
Tearing Strength on Warp Direction (Weft Tearing)	0.003*	0.000*	0.001*	0.258	0.455

\* Significant according to  $\alpha = 0.05$

Table 3

	Paragliding fabric type	N	Before exposure			After exposure		
			Subset			Subset		
			1	2	3	1	2	3
Breaking force on warp direction	Turquoise	5	243.24			Turquoise	197.53	
	Orange	5		325.86		Red	280.69	
	Red	5		368.12		Orange	291.83	
	White	5			468.04	Dark Blue	299.94	
	Dark Blue	5			522.29	White		449.670
	Sig.		1.000	.117	.052		.110	1.000
Breaking force on weft direction	Orange	5	344.56			Dark Blue	190.09	
	Dark Blue	5	349.88			Red	200.20	
	Turquoise	5	379.06	379.06		Orange	294.04	294.04
	Red	5		402.81		Turquoise		313.38
	White	5		408.31		White		402.56
	Sig.		.103	.177		Sig.	.119	.180
Elongation (Warp direction) (%)	Orange	5	18.4533			Orange	13.0800	
	Turquoise	5	20.1967			Dark Blue	13.6633	
	White	5	20.5967			Red	16.8067	16.8067
	Red	5	20.8000			Turquoise		17.7533
	Dark Blue	5	21.6500			White		18.4533
	Sig.		.209			Sig.	.056	.490
Elongation (Weft direction) (%)	Red	5	21.1833			Red	14.6767	
	White	5	21.1833			White	15.4633	
	Dark Blue	5	21.5167			Dark Blue	18.2033	
	Orange	5	22.1400			Orange	19.1933	
	Turquoise	5		24.7533		Turquoise	19.2200	
	Sig.		.814	1.000		Sig.	.432	

Table 4

	Paragliding fabric type	N	Before exposure				After exposure			
			Subset				Subset			
			1	2	3	4	1	2	3	4
Bursting strength	Orange	5	664.8				Turquoise	581.9		
	Turquoise	5		743.3			Orange		677.9	
	Red	5			841.1		Red		679.9	
	Dark Blue	5				945.9	Dark Blue			766.16
	White	5				950.6	White			794.90
	Sig.		1.000	1.000	1.000	.772	Sig.	1.000	0.923	0.222
Warp tearing strength	Red	5	29.6				Turquoise	16.7		
	Orange	5	30.7				Red		21.0	
	Turquoise	5		36.0			Orange			24.3
	White	5			41.9		Dark Blue			30.0
	Dark Blue	5			44.4		White			37.8
	Sig.		.557	1.000	.202		Sig.	1.000	1.000	.640
Weft tearing strength	Dark Blue	5	22.9				Turquoise	11.5		
	Turquoise	5	24.2				Dark Blue	15.9		
	Red	5		26.1			Red		22.8	
	Orange	5			29.7		Orange			28.8
	White	5				38.4	White			37.7
	Sig.		.192	1.000	1.000	1.000	Sig.	0.834	1.000	1.000

Table 5

	Color change ( $\Delta E$ )	
	With all samples	Without turquoise sample
Breaking Strength Loss on Warp Direction	0,12	0,82
Breaking Strength Loss on Weft Direction	-0,11	0,58
Elongation Loss on Warp Direction	-0,25	0,99
Elongation Loss on Weft Direction	0,23	0,80
Bursting Strength Loss	0,82	-0,83
Tearing Strength Loss on Weft Direction (Warp Tearing)	0,91	0,80
Tearing Strength Loss on Warp Direction (Weft Tearing)	0,89	0,74

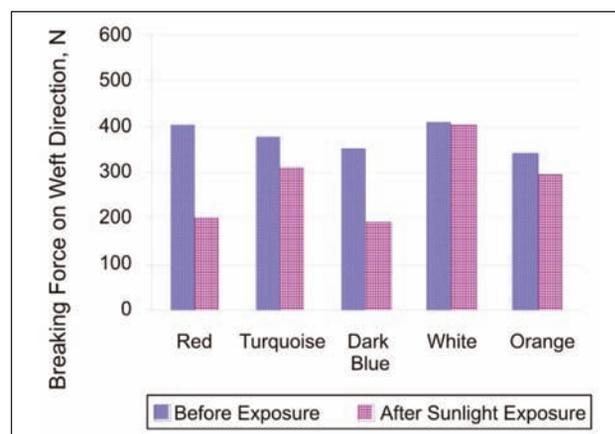
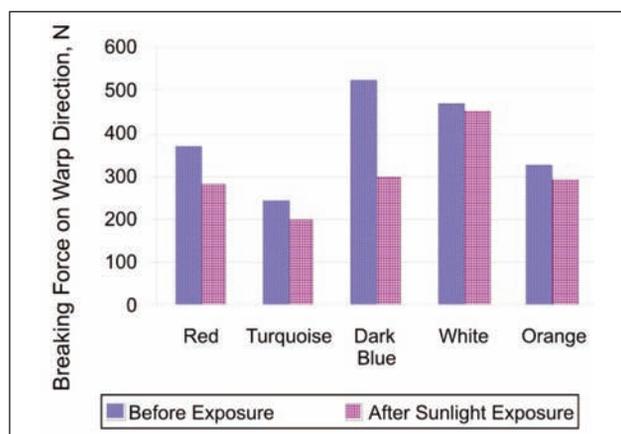
Table 6

Fabrics	Status	L*	a*	b*	$\Delta E^*$
Red	Before Exposure	41.28	52.59	27.18	3.17
	After Exposure	44.4	52.33	27.64	
Turquoise	Before Exposure	61.16	-39.52	-25.07	35.33
	After Exposure	75.3	-25.56	4.14	
Dark Blue	Before Exposure	38.1	15.43	-51.92	7.92
	After Exposure	39.46	9.69	-46.63	
Orange	Before Exposure	57.03	57.88	53.51	5.15
	After Exposure	57.11	52.86	52.4	
White	Before Exposure	91.39	-1.26	4.9	0.44
	After Exposure	91.28	-1.17	4.49	

### Results of breaking force and breaking elongation

According to table 3, it can be stated that in most cases there is a significant difference between the results measured before and after sunlight exposure. As seen in figure 2, the breaking force results for both the warp and weft directions decrease in all fabric types after sunlight exposure. According to figure 2, *a*, it can be denoted that the dark blue and white samples have the highest breaking force for warp direction values, while the turquoise sample has the lowest before sunlight exposure. However, after expo-

sure, the white sample has significantly higher breaking strength than the other samples. The decrease in the breaking strength value seen both before and after the exposure was not found statistically significant for the white and orange samples as seen in table 3. Fabrics generally lose their tensile strength after sunlight exposure. In the case of the breaking force on the weft direction results (figure 2, *b*), the white sample which has the highest fabric thickness value (table 1), again has the highest force value both before and after sunlight exposure.

Fig. 2. Breaking Force Results: *a* – on Warp Direction; *b* – on Weft Direction

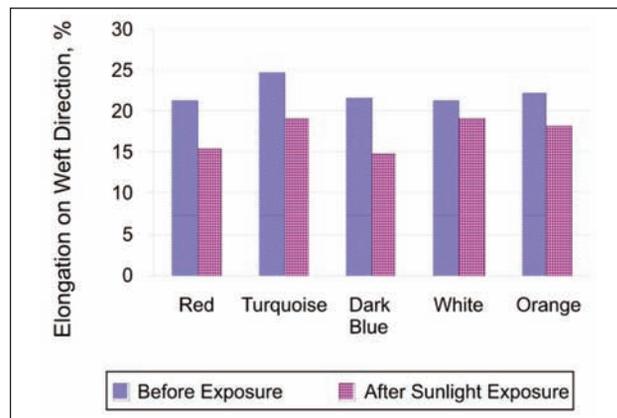
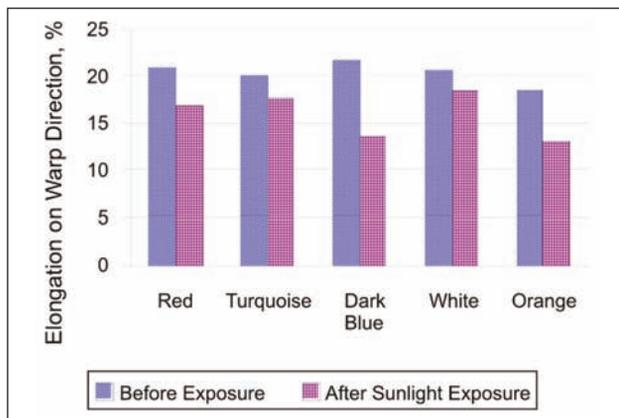


Fig. 3. Elongation Results: a – on the Warp Direction; b – on the Weft Direction

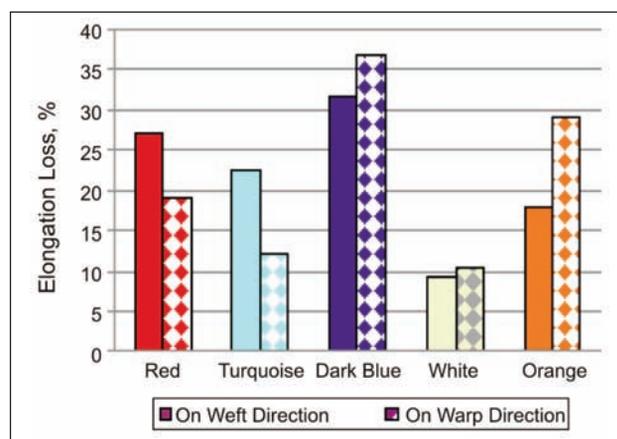
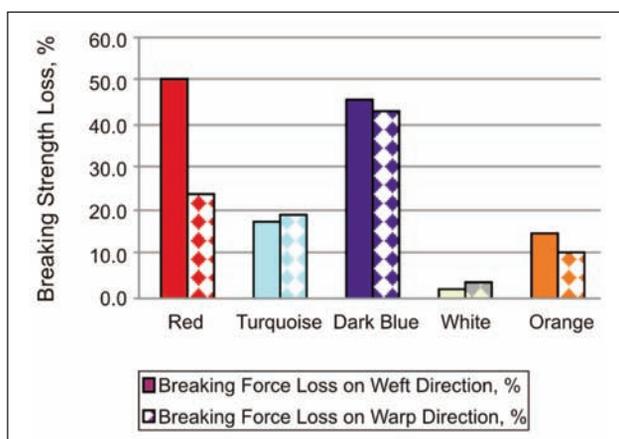


Fig. 4. Breaking Strength Loss and Elongation Loss of the Fabrics

When the elongation values were evaluated, the results generally decrease after sunlight exposure (table 3) and the differences between the values were mostly found statistically insignificant. The turquoise sample has a higher elongation value on the weft direction before exposure; however it loses it after exposure. In most cases, the measured values were found close to each other.

The paragliding fabrics were affected with the exposure to sun light differently and to compare them with each other the “Breaking Strength Loss” and “Elongation Loss” values were calculated according to equation 1. The results are given in figure 4.

It is thought that; dark blue and red paragliding fabrics absorb more solar radiation. For this reason, dark blue and red colored fabrics are more affected with sunlight exposure (photo aging) than the other fabrics. The breaking strength loss of the turquoise sample is relatively lower than the dark blue and red samples. Besides its lighter color, this result is also related with the additional silicone coating on the fabric surface. It supports the material and therefore the breaking strength loss of this fabric is lower. Since the solar radiation absorption is lower for the white and orange samples, the breaking strength loss of these fabrics were found as the lowest.

### Results of bursting strength

According to the bursting strength values given in figure 5 and the statistical evaluation results given in table 5, it was pointed out that the white and dark blue samples have the highest values and the difference between them was found statistically insignificant. Similar to the breaking strength results, the turquoise sample has a comparatively lower bursting strength. For all fabric types, sunlight exposure has a significant decreasing effect on the bursting strength (table 3).

According to the bursting strength loss values, which were calculated according to equation 1, the highest strength loss was observed in the dark blue, turquoise, and red samples, however, the orange sample has a comparatively lower strength loss. The white sample has the lowest loss among the studied fabrics. Although the tensile strength loss of the turquoise sample is lower, the bursting strength loss value was found to be higher. It is thought that the additional silicone coating on the turquoise sample decreases the elasticity of the fabrics and that the fabric reaches the bursting limit easily, which results in a low bursting strength value [19–20].

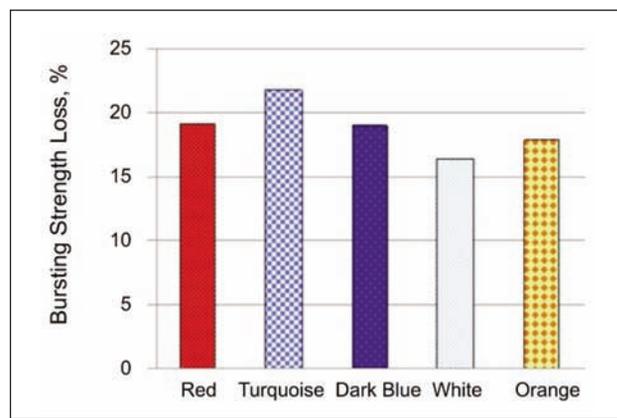
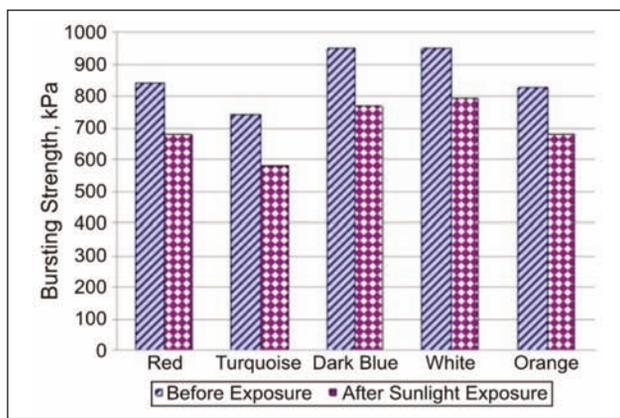


Fig. 5. Bursting Strength Results Before and After Sunlight Exposure (a); Bursting Strength Loss After Sunlight Exposure (b)

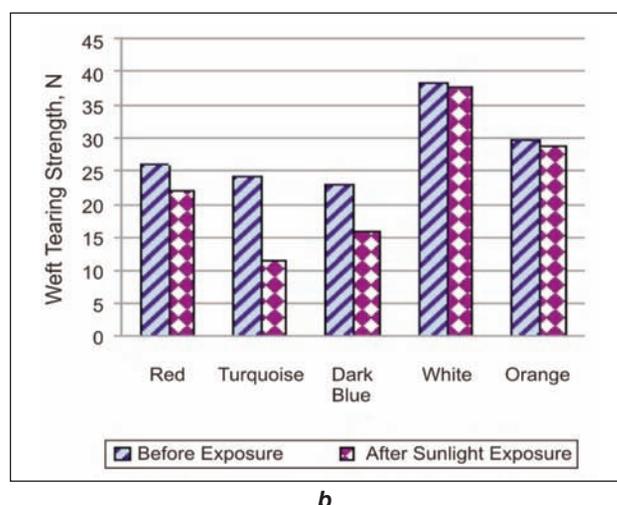
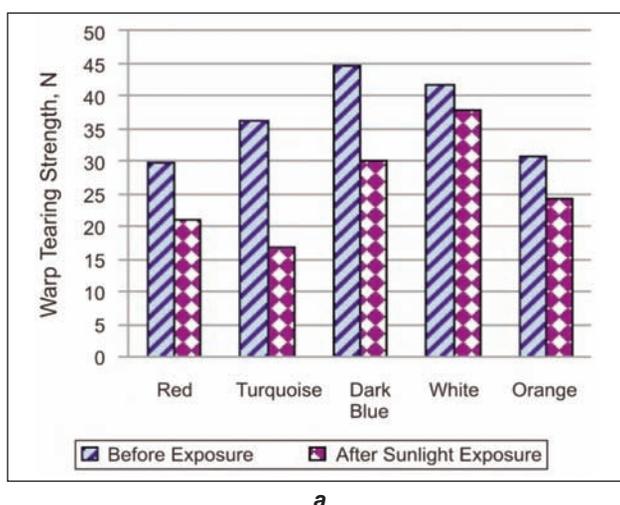


Fig. 6. Tearing strength test results

### Results of tearing strength

When the warp tearing strength values were examined (figure 6, a), it can be seen that the white sample has a higher tear strength value before and after sunlight exposure. The results were found to be parallel to the results of the breaking strength for the dark blue sample.

The dark blue fabric has the highest warp tearing strength, however, it has a very low value on the weft direction. The turquoise sample with its lowest fabric density has a low tearing strength in both directions. The tear strength in both directions generally decreases after sunlight exposure (table 3) apart from the white and orange samples. Tearing strength values of these two samples did not decrease significantly after exposure.

The calculated tearing strength loss values (figure 7) were found parallel to the results of the bursting strength loss values. Figure 7 emphasizes the low loss in the white and orange samples and a high loss in the red, blue, and turquoise samples. The high tearing strength loss of the turquoise sample is associated with the lower elongation of the yarns. The breaking elongation of the yarns used in the fabric

structure affects the distance between the yarns in the tearing point and therefore, changes the dimensions of the tearing triangle and the number of yarns which are torn. In conclusion, the higher breaking elongation increases the tearing strength of the fabric [21]. The additional silicone coating on the turquoise sample decreases the elongation of the yarns and causes a higher loss in the tearing strength. Moreover, yarns that can group together by lateral movement during tearing give a better tearing resistance. Since, the silicone coating on the turquoise

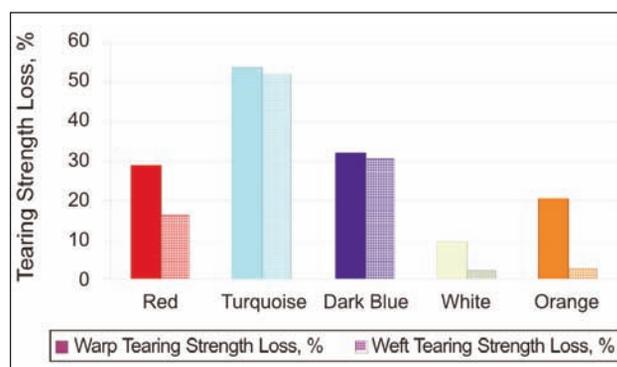


Fig. 7. Tearing strength loss after sunlight exposure

sample also decreases the grouping ability, a lower tearing strength value was measured for this type of fabric [22].

## CONCLUSIONS

In this article, 5 different paragliding fabrics different in colors, densities, and yarn counts were investigated. In order to simulate the usage during flights, all fabrics were exposed to sunlight for 150 hours. Changes in the breaking, tearing, bursting strength, and air permeability were researched using statistical methods. Another aim was to clarify how different colors affect the absorption of light in a material and change the aging. The following results were found in this study:

- In normal usage, polyamide fiber is not durable to solar radiation and for this reason lamination materials such as polyurethane and silicone are used to increase its resistance. Nevertheless, it was determined that there is a significant decrease in the mechanical properties of the investigated fabrics after sunlight exposure. The fabrics aged under sun light become considerably weaker in the case of their mechanical properties. UV and IR radiation results in an important amount of degradation in both the dyestuff and macromolecular structure of the fiber.
- According to the breaking strength test results, photo aging affects the results of the blue, red, and turquoise samples under the conditions used in the experiments significantly. However, for the orange and white samples the breaking strength change was not found statistically significant.
- The turquoise sample lost approximately 50% of its initial tearing strength, while dark blue was over 30%. Tearing strength change of orange sample was found lower than 20%. The white sample was

not nearly as affected from sun light exposure, by means of tearing strength, which emphasizes the importance of color in aging.

- Polyurethane and silicone coatings which were used on the turquoise sample support the material and therefore, the breaking strength loss of this fabric was found to be lower. However, these coatings decrease the elasticity of the fabrics and result in a higher bursting strength loss. In addition to this, they cause higher tearing strength loss, due to the decrease in the elongation of the yarns and decrease in the grouping ability of the yarns.
- In the air permeability test, no permeability value could be detected under the measurement conditions. Therefore, it is thought that 150 hours of photo aging does not change the air permeability value that could cause a safety problem during flight.
- It is thought that the blue, turquoise, and red paragliding fabrics are affected by sunlight exposure (photo aging) more than the other fabrics. This results in higher tensile strength loss in the fabric structure.

The use of paragliding fabrics exposes them to sunlight and therefore, there is an important amount of aging which changes the fabric properties significantly. The decrease in tensile properties and the increase in air permeability of the fabric can be accepted, to some extent, as a result of photo aging. However, if the air permeability of the fabric increases to a certain point, the paraglider loses altitude rapidly, which is not desired. In addition to this, a decrease in the tensile properties can also be of vital importance for not supplying the requirements for the needed strength values.

According to the results, it was concluded that selected paragliding fabrics lost some of their mechanical properties after sun light exposure, while the air permeability of the fabrics did not increase.

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## REZUMAT – ABSTRACT

### Captarea energiei din mișcările umane pentru aplicații portabile

Captarea energiei biomecanice din mișcarea umană este o soluție alternativă pentru a alimenta eficient sistemele electronice portabile. În acest studiu au fost dezvoltate două dispozitive de captare a energiei piezoelectrice, pe bază de impact, care pot fi integrate în tălpile de încălțăminte și pot fi, de asemenea, adaptate pentru a fi integrate în covoarele comercializate sau în carosabilul exterior pentru a capta energia mecanică masivă de la vehiculele care se deplasează sau de la un grup de oameni la frecvențe reduse. Pentru un studiu cuprinzător, au fost selectate și testate două tipuri de dispozitive de captare PVDF. S-a demonstrat că răspunsurile mecanice ale prototipului tip arc și ale prototipului de tip C sunt diferite. În plus, răspunsul mecanic al tipului C poate fi afectat de înălțimea verticală a tipului C. Tensiunea de vârf a tipului C crește odată cu scăderea înălțimii verticale a tipului C. Tensiunea de vârf a tipului arc este aproape aceeași cu cea a tipului C, când înălțimea verticală este de 25 mm. Stabilitatea tensiunii de ieșire a tipului arc este cea mai scăzută în comparație cu cea a celor trei tipuri C. Stabilitatea tensiunii de ieșire a tipului C în cazul în care înălțimea verticală este de 25 mm este cea mai scăzută dintre cele trei înălțimi verticale diferite.

*Cuvinte-cheie:* piezoelectric, dispozitiv de captare a energiei, mișcări umane, frecvență scăzută, portabil

### Energy harvesting from human motions for wearable applications

Harvesting biomechanical energy from human's movement is an alternative solution to effectively power the wearable electronics. In this paper, two impact-driven piezoelectric energy harvesters were developed which can be integrated within human shoe-soles and also can be tailored to integrate in commercial carpets or outdoor roadway to harvest the massive mechanical energy from the passing vehicles or people in crowds at low frequencies. For a comprehensive study, two buckling types of PVDF harvesters were selected and tested. It has been shown that the mechanical responses of the arch type prototype and the C type prototype are different. In addition, the mechanical response of the C type can be affected by the vertical height of the C type. The peak-peak voltage of the C type increases with the vertical height of the C type decreases. The peak-peak voltage of arch type is almost the same with the C type when the vertical height of which is 25 mm. The stability of the output voltage of the arch type is the worst when compared with that of the three C types. The stability of the output voltage of the C type when the vertical height of which is 25 mm is the worst among the three different vertical heights.

*Keywords:* piezoelectric, energy harvester, human motions, low frequency, wearable

## INTRODUCTION

Energy harvesting from human body's movement has become a widespread issue due to its versatile capability serving as a clean and renewable energy system to power some low-power consumption electronic devices [1–3]. Several working mechanisms or concepts can be used to fabricate a human energy harvester, such as electromagnetic [4], electrostatic [5], thermoelectric [6], triboelectric [7], piezoelectric [1] and biofuel cell [8]. Among them, the piezoelectric energy harvester has attracted much more research attention because of its simple structure and great potential to convert the mechanical energy into electricity in an direct and high-efficiency manner [9–10]. The piezoelectric energy conversion occurs because the piezoelectric molecular structure is oriented such that the material exhibits a local charge separation, known as an electric dipole. When a stress is applied to the material it cause a strain, so it results in a deformation of the electric dipole and the formation of a charge that can be removed from the material and used to power various electric devices [11]. Perhaps

the most common and effective type of piezoelectric human energy harvester is integrating the piezoelectric materials into shoes due to the large amount of power generation from walking or running in daily life [12–14].

To adapt to the low-frequency walking condition and overcome the limitations of the shoes' structure, most current studies of shoe-equipped piezoelectric transducers focus on the following three types, namely flat plate type, arch type and cantilever type [10]. Kymissis explored an insole made of eight-layer stacks of PVDF sheets with a flexible plastic substrate to harness the energy dissipated in bending of the sole and the average power reached 1.1 mW at 1 Hz [14]. Zhao developed a shoe-embedded piezoelectric energy harvester, which was readily compatible with a shoe [1]. The harvester was based on a sandwich structure and there was a multilayered PVDF film sandwiched between the two wavy surfaces. Moro presented a rectangular piezoelectric cantilever based on PZT, which was mounted inside the shoe heel using a conventional clamp system

without loss of comfort or radical change in shoe design [15]. Ansari designed horizontal and vertical buckling harvesters placed on the road to scavenge the mechanical energy from passing vehicle or walking people [16]. To the best knowledge of the authors, there is no literature mentioned on the study of vertical buckling harvester mounted on the shoe to harvest human's biomechanical energy, especially the comparison study of horizontal and vertical buckling harvesters.

In this research, two kinds of piezoelectric buckling harvesters, which were horizontally and vertically configured were investigated and compared. A series of experiments were carried out to characterize the harvester prototypes, which finally proved that the fabricated harvesters can be served as a sustainable and wearable power supply for low power consumption electronic devices.

### HARVESTER DESIGN AND FABRICATION

Lead zirconate titanate (PZT) and polyvinylidene difluoride (PVDF) are the most common piezoelectric materials for energy harvesting owing to their high piezoelectric performance. Considering the PVDF has a considerably better flexibility compared with the PZT, the harvester proposed in this paper is based on the PVDF. The selected PVDF units are integrated with aluminum layer electrodes.

Two prototypes of the 31-mode harvesters are fabricated for two different purposes. Prototype 1 is horizontally configured, which designed as an arch type, the schematic diagram and the finished prototype are shown in figure 1, a. In this regard, the PVDF will contact directly the heel. Prototype 2 is vertically configured, which designed as a C type, the schematic diagram and the finished prototype are shown in figure 1, b. The PVDF will not contact the heel in a direct way and thus this reduces the damage of the PVDF compared with the prototype 1. The prototype 1 is a common type in previous studies, the purpose of this design in here is to compare with prototype 2.

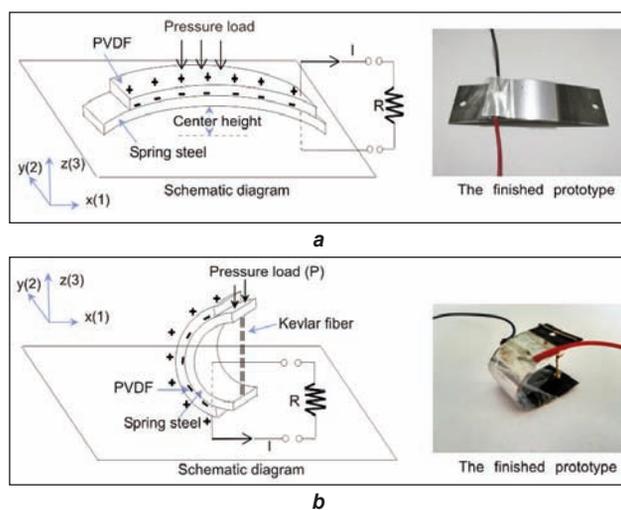


Fig. 1. Schematic diagram of different structural piezoelectric energy harvesters: a – Prototype 1: arch type; b – Prototype 2: C type

The two kinds of harvesters all can be mounted to the insole of the heel. The PVDF layer could generate electricity when it is deformed and returns back to the non-deformed shape periodically. When the external force is removed, the working unit returns to its original position.

The two harvesters consist of two major components: the top PVDF layer and the bottom spring steel substrate. These two layers are bonded together by a thin adhesive layer. The PVDF layer is in the middle of the curved spring steel layer, whose length, width, and thickness dimensions are 70, 20, and 0.33 mm, respectively. The center height of arch type harvester is set as 5 mm. When the descent height of the two prototypes is fixed to 5 mm, the height of C type harvester is more adjustable. Considering the shape of the shoe's heel, three specifications of the vertical height of C type harvester are chosen, which are 30 mm, 25 mm, 20 mm. The corresponding C type harvesters are named prototype 2a, prototype 2b and prototype 2c, respectively. The kevlar fiber is chosen to adjust the height of the prototype 2 due to its high strength and high modulus, which resulted in the vertical height of C type harvesters can be controlled well. The other geometric and material parameters of PVDF are listed in table 1.

Table 1

Parameters	Value
$d_{33}$ (PC/N)	21
$d_{31}$ (PC/N)	17
Coupling coefficient $k_{33}$ (%)	10~14
Density ( $\text{kg/m}^3$ )	$1.78 \times 10^3$
Relative permittivity	$9.5 \pm 1.0$
Elastic modulus (MPa)	2500
Length×width×thickness (mm×mm×mm)	$30 \times 20 \times 0.03$

### EXPERIMENTS

To compare these two kinds of harvesters, the power harvesters tested was based on a simple force excitation generator which can provide an impulse force and can constrain the axial deformation easily, the illustration of the force excitation generator is shown in figure 2. The output voltages of two prototypes were measured by the oscilloscope. During testing, the harvester was fixed to the buffer board and the impulse force was set to 500 N. The axial deformation of prototype 1 and prototype 2 were constrained to 5 mm by adjusting the location of buffer board. For the purpose of preventing the damage of the PVDF under excessive deformations, we limited the amount of axial deformation.

To test the output voltages of the harvesters, the force frequency of the force excitation generator was set as 1 Hz. During testing, the output of the harvesters was terminated into the oscilloscope to measure the output voltage. The internal resistance of the oscilloscope is 10 M $\Omega$ , serving as a resistive load.

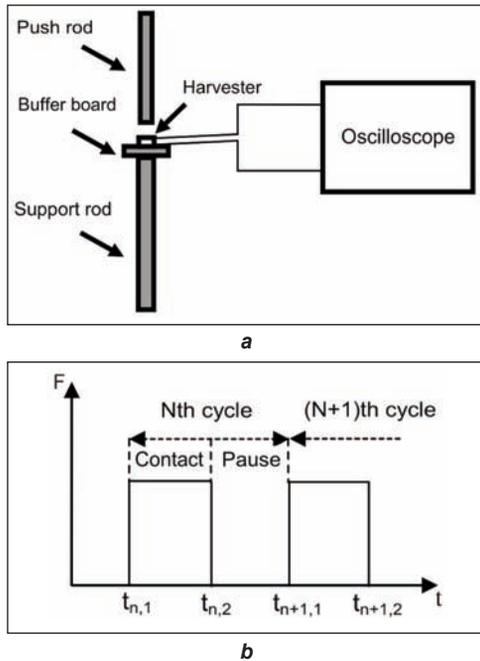


Fig. 2. The illustration of the simple force excitation generator: *a* – structure; *b* – schematic

## RESULTS AND DISCUSSION

The single voltage waveforms created by the harvesters at a 1 Hz impulse force can be seen in figure 3, which shows the impulse response of the harvesters. It can be seen different from that of prototype 2, which means that the mechanical responses of the two types are different. The similarity of the plots between the prototype 2a and prototype 2b indicates that there are no significant differences between the mechanical responses of the prototype 2a and prototype 2b. But the plot of prototype 2c is different from that of the prototype 2a and prototype 2b, which means that when the vertical height reaches a certain value, it can influence the mechanical responses of the prototype 2.

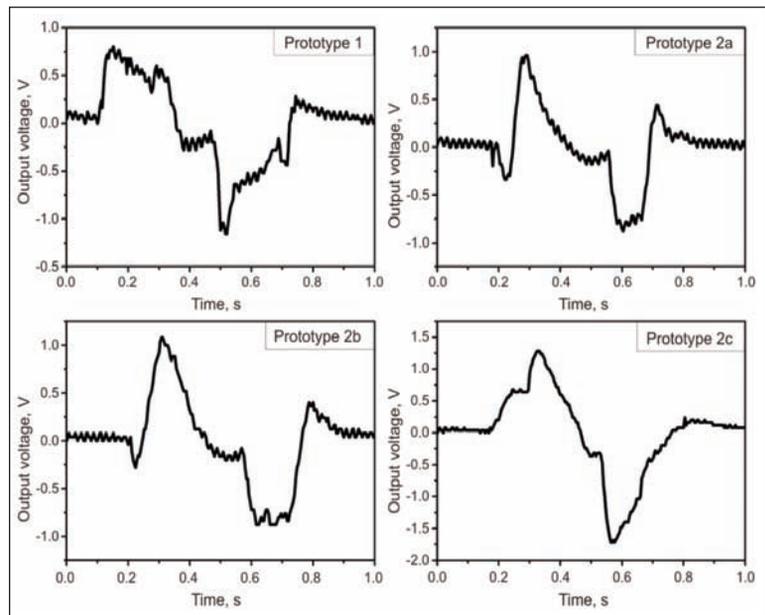


Fig. 3. The single actuation of the two harvester

Figure 4, *a* compares the results of the peak-peak voltages of the two prototypes. It can tell that the corresponding peak power which calculated by the equation  $P = U^2/R$  of the harvesters is at the  $\mu W$  level. In addition, the figure 4, *a* shows that the prototype 1 can produce approximately the same voltage of the prototype 2b, the differences are not so significant. For the prototype 2, the peak-peak voltage of the prototype 2c is the biggest, that of the prototype 2a is the smallest. From the test results it can be seen that the peak-peak voltage increases with the vertical height of prototype 2 decreases. The coefficient of variation (CV) of output voltage reflects the stability of the output voltage of the harvester. Figure 4, *b* shows the CV of the peak-peak output voltages of the prototypes, it can be seen that the CV of the prototype 1 is the biggest and the prototype 2a is the

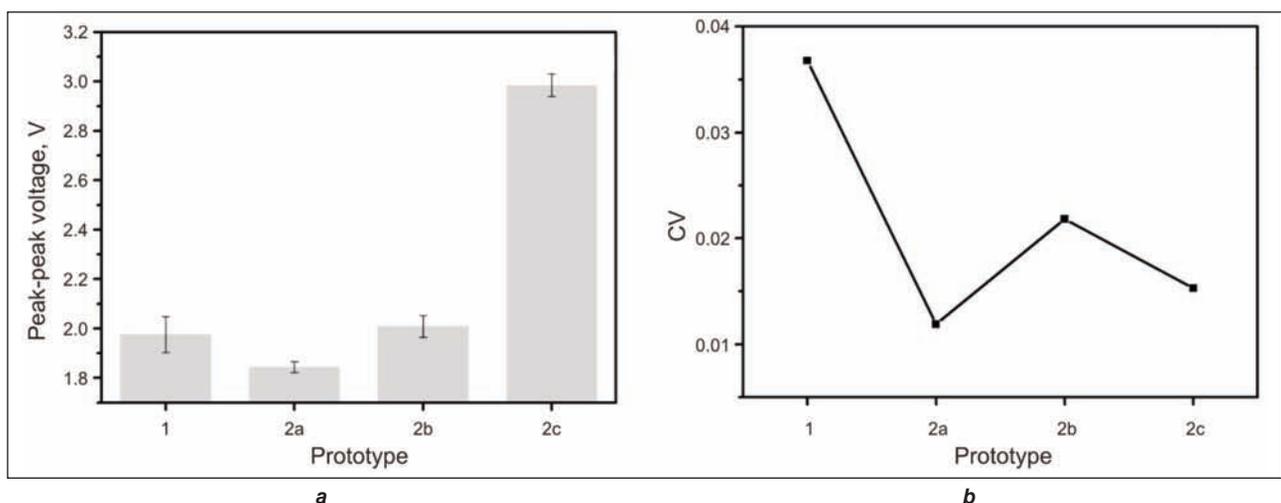


Fig. 4. *a* – The peak-peak output voltages of the prototypes; *b* – The CV of the peak-peak output voltages of the prototypes

smallest, which means the stability of the output voltage of the prototype 1 is the worst and that of the prototype 2a is the best. For the prototype 2, the CV of the output voltage are not the same, the results reveal that the vertical height of prototype 2 effect the stability of the output voltage.

## CONCLUSIONS

This paper fabricated two mechanical energy harvesters based on piezoelectric effect driven by the impulse excitation. We performed the experiment under 500 N impulse force at 1 Hz. The experimental results revealed that the two energy harvesters were

capable of generating the electricity under low-frequency excitation and the obtained devices exhibit many compelling advantages for practical applications in real environment. The two designed energy harvesters can be used not only in a shoe's heel, but also they are scalable and can be tailored to integrate in commercial carpets or outdoor roadway that converts the massive mechanical energy from the passing vehicles or people crowds into electricity. Though the output power is not very big, N of the same prototypes can be connected in series or parallel to improve output power in the future work, so the two prototypes are promising and could provide a potential possibility for future green energy.

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# Raincoat design for children for age group 7-8 years: A design development case study

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## REZUMAT – ABSTRACT

### Proiectarea jachetei de ploaie pentru copiii cu vârsta de 7–8 ani. Studiu de caz pentru dezvoltarea modelului

*Această lucrare propune un proces de proiectare și dezvoltare a jachetei de ploaie pe baza unei analize obiective a nevoilor utilizatorilor. Jacheta de ploaie propusă este concepută pentru copiii cu vârsta cuprinsă între 7 și 8 ani. Procesul de proiectare propus începe cu interviuri personale și cu examinarea participanților. Analiza necesităților utilizatorilor va fi realizată în acest proces cu referire la categoriile de nevoi ale utilizatorilor, și anume cele funcționale, expresive și estetice. Aceste nevoi au dus la dezvoltarea ulterioară a criteriilor de proiectare. Aceste criterii au fost transformate în caracteristici ale jachetei de ploaie și utilizate în dezvoltarea unui prototip de jachetă de ploaie. În final, a fost evaluat prototipul jachetei de ploaie în ceea ce privește criteriile de proiectare. Designul final combină proprietățile funcționale, expresive și estetice dorite, așa cum sunt evidențiate prin criteriile de proiectare.*

*Cuvinte-cheie: considerații FEA, model de îmbrăcăminte pentru copii, proces de proiectare, analiza obiectivă, proiectarea jachetei de ploaie*

### Raincoat design for children for age group 7–8 years: a design development case study

*This paper proposes a rain coat design and development process based on objective analysis of user needs. The proposed raincoat is developed aiming at children for age group 7–8 years. The proposed design process starts with personal interviews and participant observation. The user needs analysis will be realized in this process regarding the user need categories of functional, expressive, and aesthetic needs. These needs led to the further development of design criteria. These criteria were then translated into raincoat attributes and used in the development of a raincoat prototype. Finally, the raincoat prototype was evaluated regarding the design criteria. The final design combines the desired functional, expressive, and aesthetic attributes as outlined by the design criteria.*

*Key words: FEA considerations, children's garment design, design process, objective analysis, and raincoat design*

## INTRODUCTION

Raincoat is one of the most widely used functional garments used by children for age group 7–8 years. These children are undergoing a period of slow but steady growth, during which they are encouraged to learn some physical skills by themselves. Normally, children at this age period are very active compared with the previous age group, but they don't have a lot of outdoor activity experience. In this condition, they are easily influenced by the uncertainty of the weather. Current raincoat for children for age group 7–8 years supplied in market is adapted from that of adults, which cannot fully satisfy the needs of children. The general shape of these raincoats is in A line. Their sleeves are very big. Fabrics chosen for these raincoats are heavy and unbreathable. Color story of these raincoats is also not well considered. Normally one color is used for these raincoats. Both children and their caregivers (parents, teachers...) are demanding a new functional raincoat: comfortable, highly breathable, lightweight... There is a huge

gap between the consumer's perception and supplied product.

In order to improve the current design of raincoat for children for age group 7–8 years, FEA (functional, expressive, and aesthetic) needs of this group is analysed. These needs led to the further development of design criteria of the desired raincoat. FEA Consumer Needs Model is proposed by Lamb and Kallal (1992) as a consumer needs model that assesses user needs and wants by incorporating functional, expressive, and aesthetic considerations (FEA) [1]. This model has been recognized to have implications in different research. For example, Watkins (1995) extracted a design process that strengthens user needs by the use of their model [2]. Bye and Hakala (2005) developed ankle braces designed and sized specially for women which shows the critical impact of user needs [3]. Cristiano Ciappei and Christian Simoni (2009) used the FEA Consumer Needs Model to identify the key success factors engrained in the new product development practices of sport shoe companies [4].

Current research related to the application of the FEA Consumer Needs Model presents the fact that, even though the client defined the problem initially at the beginning of the process, designers should work through the design step of analysis and determine what the client viewed as the problem. In this process, product factors which are related to user needs can be highlighted. In this research, a FEA Consumer Needs Model is used to analysis the user needs of raincoat for children for age group 7–8 years. A group of user represents, who have deep understanding of the user need of this group is selected. Then a set of user needs criteria will be determined but this group of represents use the FEA analysis. Then, these criteria will be announced to a group of selected designers. Based on the knowledge and experience of the selected designers, these design criteria will be translated into relevant raincoat attributes. These raincoat attributes will contribute to the development of a raincoat prototype. Finally, the raincoat prototype will be evaluated regarding the user needs criteria provided by the group of user represents. Using this design process, the final obtained design is integrated with the desired functional, expressive, and aesthetic attributes as outlined by the design criteria.

The rest of the paper is structured as follows. In Section 2, the overall experiment design and related concepts used in the experiment design is explained. In Section 3, experiment results have been given and analysed. Finally, a conclusion is provided in Section 4.

### Experiment design and related concepts

This research proposes a novel design process based on the FEA Consumer Needs Model. Two groups of different designers and user represents are involved. In this section, the overall experiment and the relevant concepts will be explained.

### The proposed design process

User involvement is a widely accepted principle in a successful product development process [5]. A lack of user involvement has been repeatedly associated with failed product development projects and the benefits of user involvement have been shown in several studies [6, 7]. Analysis and integration of user needs can largely increase user involvement and lead to the success of product development [8]. The proposed design process is based on the user needs analysis using FEA Consumer Needs Model. Figure 1 describes the proposed design process.

There are two groups of people involved in this process: user represents and selected designers. The user represents are care-givers of children at the age group of 7–8. They are full-time mothers/fathers, kindergarten teachers, or primary school teachers. All of them have experience in taking care of children at the age group of 7–8. An announcement of the research purpose of this study is delivered to them and they are willing to participate in this research. There are 20 user represents who are invited. The selected designers are chosen from children's wear

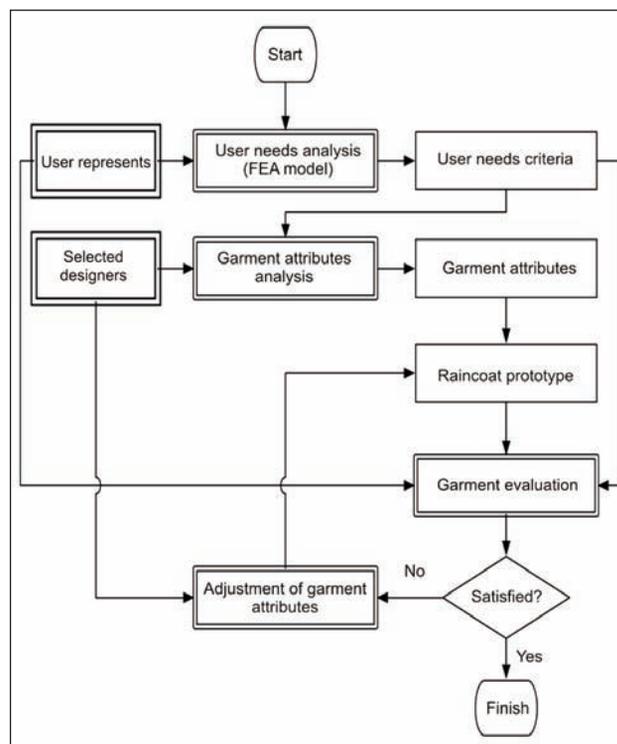


Fig. 1. Overall experiment design and data processing flow chart

fashion brands. The selected designers meet the following three requirements: (1) he/she has worked in children's wear for more than 5 years; (2) he/she has clear understanding of the physical, mental, emotional, and social characteristics of children at the age group of 7–8; (3) he/she is very experienced in garment design solutions for children's wear. There are 20 user represents who are selected.

The proposed design process starts with the user need analysis using a FEA Consumer Needs Model. The section is performed by user represents. After this section, a set of user needs criteria will be obtained. After that, based on these user need criteria, the selected designers will have a brand storm together. Related raincoat attributes will be defined based on these user need criteria. Subsequently, a raincoat prototype can be proposed when the designers reach a common conclusion. The last section of the proposed design process is the raincoat prototype evaluation. The proposed prototype will be presented to the selected user represents. They will evaluate the proposed raincoat prototype regarding the set of user needs criteria they offered. If they agree with the proposed prototype, the design process will be finished. If they don't agree, designers will adjust the raincoat attributes based on the feedback of the user represents. The sequence of design-evaluate-adjustment will be repeated for several times until the final result is satisfied by the user represents.

### FEA Consumer Needs Model

The FEA Consumer Needs Model is developed by Lamb and Kallal in 1992. They believe that considerations of functional, expressive and aesthetic criteria

should be taken into apparel design process. Since 1992, the FEA Consumer Needs Model has been accepted in both research and industrial application. It is regarded as a theoretical framework to guide designers for the better understanding of the user. In applications of this conceptual framework, authors used the consumer needs focus to assess FEA criteria for products for various consumer groups. Some altered the model by not including all three criteria, or adding additional criteria. Design solutions included functional design for health and well-being, sports apparel and smart clothing, fashion apparel, textiles, costumes, fashion history as inspiration, and non-apparel items.

### Subjective evaluation and fuzzy set theory

Experiments for the proposed design process are based on sensory evaluation. In this procedure, uncertain and imprecise linguistic expressions are often used by both designers and user represents. Thus, fuzzy set theory can be a relevant method for processing uncertain data obtained from sensory evaluation. Fuzzy set, as an intelligent technique, is developed to handle the vagueness of human thought which is full of uncertainty and imprecision [9]. Fuzzy set theory has wide application in the area of sensory/subjective evaluation since it has obvious advantages in dealing with uncertain data, such as linguistics and clustering [10–12]. Using fuzzy set tools, a set of linguistic terms, describing the evaluation criteria, can be quantitated in the universe of respective and discourse membership function [13]. Triangular fuzzy numbers (TFN), as a classic fuzzy set tool, are used to quantitate the utilized linguistic terms in this research.

### Data process using fuzzy set theory

Based on fuzzy set theory, linguistic terms of the proposed linguistic rating scale  $L_k$  can be quantified into Triangular Fuzzy Numbers (TFNs). A Triangular Fuzzy Number (TFN),  $M$ , can be denoted using n-tuples formalism as  $M = (l/m, m/u)$  or  $M = (l, m, u)$ . The parameters  $l$ ,  $m$  and  $u$ , respectively, denote the smallest possible value, the most promising value, and the largest possible value that describe a fuzzy event. Each TFN has linear representations on its left and right side such that its membership function can be defined as:

$$\mu_m(x) = \begin{cases} 0, & x \in [-\infty, l] \\ \frac{x-l}{m-l}, & x \in [l, m] \\ \frac{x-u}{m-u}, & x \in [m, u] \\ 0, & x \in [u, +\infty] \end{cases} \quad (1)$$

If  $M_1 = (l_1, m_1, u_1)$  and  $M_2 = (l_2, m_2, u_2)$  are two TFNs, the operation laws between them can be defined as:

$$M_1 + M_2 = (l_1 + l_2, m_1 + m_2, u_1 + u_2) \quad (2)$$

$$M_1 * M_2 = (l_1 * l_2, m_1 * m_2, u_1 * u_2) \quad (3)$$

$$t * M_1 = (t * l_1, t * m_1, t * u_1) \quad (4)$$

$$(l_1, m_1, u_1)^{-1} = (1/u_1, 1/m_1, 1/l_1) \quad (5)$$

Using TFNs, evaluation scores given by each of the evaluators can be quantified. Table 1 presents the quantified TFNs of the proposed linguistic rating scale.

Based on the operation rules given by equation (3), (4) and (5), the evaluation scores given by each evaluator  $e_j$  can be aggregated as  $\{a_{ijh} \mid i=1, \dots, 7, j=1, \dots, 7, h=1, \dots, m\}$ , where  $a_{ijh}$  represents the number of evaluators who choose one certain degree. Therefore,

$$a_{ij} = \left( \frac{1}{m} \sum_{j=1}^7 a_{ijh} t_1, \frac{1}{m} \sum_{j=1}^7 a_{ijh} t_2, \frac{1}{m} \sum_{j=1}^7 a_{ijh} t_3 \right) \quad (6)$$

where  $t_1$ ,  $t_2$  and  $t_3$  correspond to the value of the triangular fuzzy numbers, and they take values from table 1. Table 3 presents the aggregated evaluation matrix of the relations between different FEA considerations.

## EXPERIMENTS AND RESULTS DISCUSSION

There are three experiments that are designed for the realization of the proposed design process. Experiment I is designed to obtain the set of user needs criteria by user represents. Experiment II is proposed to define the related raincoat attributes regarding the obtained user needs criteria by the selected designers. Experiment III is designed to evaluate the proposed raincoat prototype and give appropriate adjustment.

### Experiment I: User need analysis using the FEA model

Experiment I is designed to identify the needs of children for age group 7–8 years based on FEA considerations. The invited 20 user represents form an evaluation panel for this experiment. There are two steps in Experiment I: (1) generation of user needs criteria, and (2) selection and evaluation of these user needs criteria.

First, a training section was performed. The purpose of this experiment about sourcing need of children for age group 7–8 years of raincoat was announced to all the panelists. After that, a brainstorming process was performed. During the brainstorming process, each of the panelists is free to access open resources (books, internet, literature...) to get information about needs for raincoat design for children of the age group of 7–8. After the brainstorming process, each trained member of the panel generated an extensive list of user needs criteria, which are in the form of words/short sentences. Then, the generated words/short sentences were collected and screened for all the members of the panel. A “round table” discussion among all the participants was carried out to vote for all the words/short sentences. There were two main principles in the election: (1) words/short sentences with repeated meaning were avoided, and (2) the selected words should try to cover all the possible design solutions. After each step, the panel leader announced the discussion result to all the panelists. Only the discussion result

Table 1

User needs criteria	Related raincoat attributes
S <sub>1</sub> Avoiding wind	Avoid the clothes swing caused by the wind
S <sub>2</sub> Avoiding facial rain	Avoid rains go onto the face
S <sub>3</sub> Good water vapor permeability	Allow water vapor pass through the garment fast
S <sub>4</sub> Light weight	Make sure the raincoat is not so heavy
S <sub>5</sub> Childlike pattern and color	Make the raincoat more childlike (such as use cartoon characters)
S <sub>6</sub> Soft contact with skin	Make the contact part with the skin more soft

approved by all the panelists could be used in the following step. After that, a list of design solutions was determined, as presented in table 2.

### Experiment II: Definition of raincoat attributes regarding the obtained user needs criteria

Experiment II is performed by the selected designers. Experiment II has the similar procedure of Experiment I. There are also two steps of Experiment II: (1) generation of raincoat attributes, and (2) selection and evaluation of these raincoat attributes. After the two steps, a list of raincoat attributes was determined, as presented in table 1.

Based on these raincoat attributes, a raincoat prototype is proposed as shown in figure 2. The overall shape is similar to a jacket, which is closed by a plastic zipper with light weight. The proposed shape and construction of the raincoat can effectively avoid wind, as required by S<sub>1</sub>. There is a transparent mask put on the face area, which can avoid rains to go onto the face, as required by S<sub>2</sub>. Material with great vapor pass ability [14] is chosen to satisfy S<sub>3</sub>. Such kind of material is also with light weight, which can satisfy S<sub>4</sub> as well. Designers proposed that the final products will be designed with different cartoon prints as a collection. As the illustration is a prototype, cartoon characters are not proposed. There are two layers for

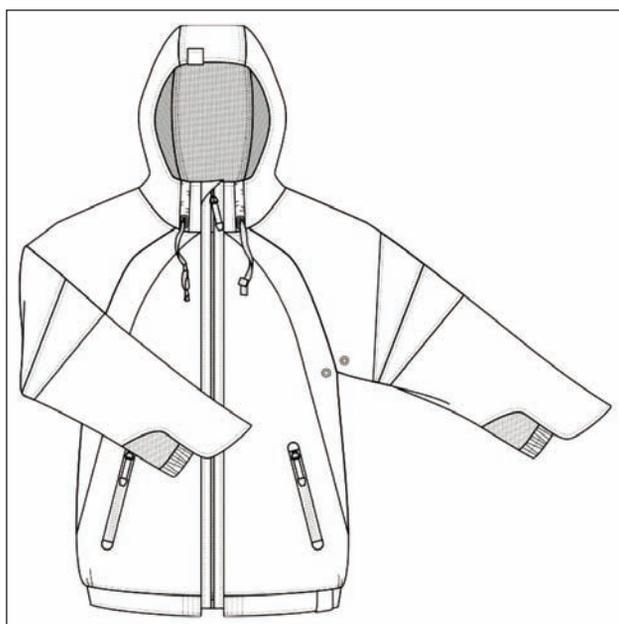


Fig. 2. Raincoat prototype for 7-8 years old children

the proposed raincoat, and the lining is polyester material with soft contact.

### Experiment III: Garment prototype evaluation and adjustment

In Experiment III, each of the user represents is required to give a score of the proposed raincoat prototype regarding user needs criteria shown in table 1. There is a label provided with the fabric content information. A scale of five evaluation degrees, ranging from A to E (A, B, C, D, E) are given by each user represents based on the overall evaluation of these dresses (table 3). "A" means that the best among all these dresses, while "E" means that the worst among all these dresses. A set of linguistic terms of the level of performance is applied to describe the evaluation degrees. In order to quantify the evaluation degrees, a set of fuzzy numbers is assigned to each of the linguistic term. The involved evaluation degrees, their corresponding linguistic term and fuzzy numbers are described in table 2.

Table 2

Evaluation degrees	Linguistic term	Fuzzy numbers
A	Best (BE)	(2.5,3,3.5)
B	Relatively good (RG)	(2,2.5,3)
C	Average (AV)	(1.5,2,2.5)
D	Relatively poor (RP)	(1,1.5,2)
E	Worst (WO)	(0.5,1,1.5)

Using this method, evaluation results of the designer can be quantified and aggregated into a group decision related to the fit effect of the proposed garment block displayed in the 3D virtual try-on.

For example, for the group perception of the invited user represents in terms of S<sub>1</sub> of the raincoat prototype can be formulated as a new triangular fuzzy number using equation 6:

$$\left( \frac{1.5 \times 2 + 2 \times 3}{5}, \frac{2 \times 2 + 2.5 \times 3}{5}, \frac{2.5 \times 2 + 3 \times 3}{5} \right) = (1.8, 2.3, 2.8)$$

Using the same calculation process, the perception of all the designers can be aggregated in terms of different KFMs, as presented in table 4.

After that, in order to investigate the design effect of proposed raincoat prototype regarding various user needs criteria, the distance of all the aggregated evaluation scores are measured to the "Perfect" condition,

User needs criteria	Aggregated evaluation result	Distance to "Perfect" condition
S <sub>1</sub> Avoiding wind	(1.8,2.3,2.8)	0.404145188
S <sub>2</sub> Avoiding facial rain	(2.3,2.8,3.3)	0.115470054
S <sub>3</sub> Good water vapor permeability	(1.4,1.8,3)	0.567646212
S <sub>4</sub> Light weight	(2.2,2.9,3.4)	0.11055416
S <sub>5</sub> Childlike pattern and color	-	-
S <sub>6</sub> Soft contact with skin	(2.4,2.9,3.4)	0.057735027

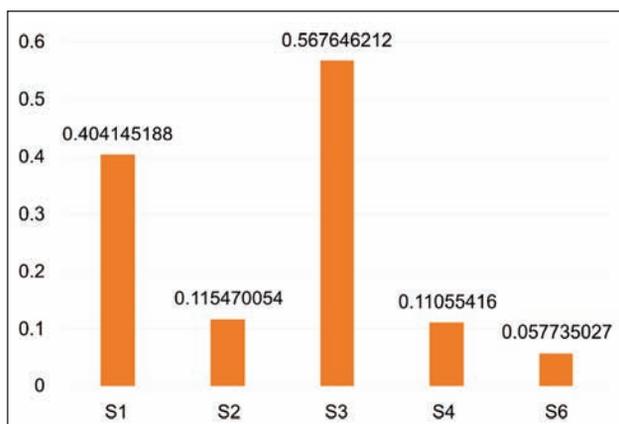


Fig. 3. Design effect of the proposed raincoat prototype regarding the user needs criteria

whose corresponding TFN is (2.5,3,3.5). When the distance is shorter, the satisfaction is higher. For example, using equations 2–6, the fit effect of  $F_{A1}$  can be calculated as:

$$\sqrt{\frac{1}{3}[(1.8 - 2.5)^2 + (2.3 - 3)^2 + (2.8 - 3.5)^2]} = 0.4$$

Similarly, all the aggregated TFNs and corresponding distance to the "Perfect" condition can be formulated as presented in table 3.

Figure 3 presents the design effect of the proposed raincoat prototype regarding the user needs criteria. It can be found that, the distances of S<sub>1</sub> and S<sub>3</sub> to the "perfect" condition is rather long, which means that the design effect of the proposed raincoat prototype is not satisfied by the user represents regarding S<sub>1</sub> and S<sub>3</sub>.

Based on the result obtained, a group discussion was organized again to adjust the proposed raincoat prototype. The same evaluation procedure that was applied at the previous stage was carried out again. The sequence of *Design – Evaluation – Adjustment* can be performed repeatedly until a satisfying design solution is obtained. Figure 4 presents the modified raincoat prototype.

For the adjusted vision of raincoat prototype, a vest is designed inside. The inside vest is more stick to the

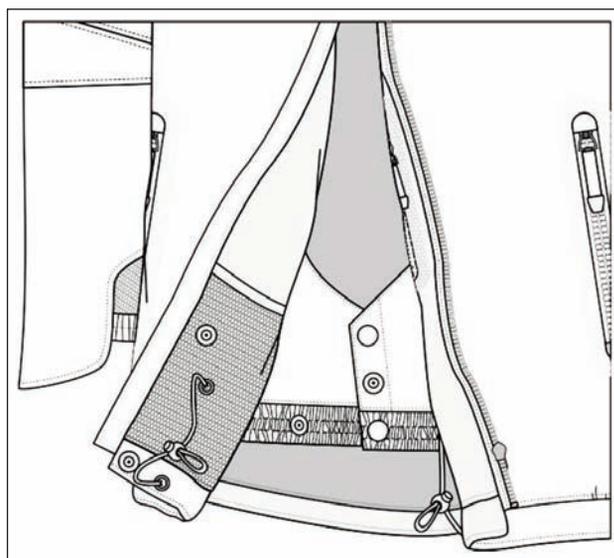


Fig. 4. Modified raincoat prototype with enhanced functional design

body, which can effectively avoid wind, as required by S<sub>1</sub>. Besides, as the outer layer of the raincoat is a little bit loose. There is a gap between inside and outside layers, which can make it possible to the vapor release, as required by S<sub>3</sub>.

## CONCLUSION

In this research, a raincoat design and development process is developed based on FEA Consumer Needs Model. The proposed design process starts with personal interviews and participant observation. The user needs analysis will be realized in this process regarding the user need categories of functional, expressive, and aesthetic needs. These needs led to the further development of design criteria. These criteria were then translated into raincoat attributes and used in the development of a raincoat prototype. Finally, the raincoat prototype was evaluated regarding the design criteria. A case study is given for the development of a raincoat aiming at children for age group 7–8 years. The final design combines the desired functional, expressive, and aesthetic attributes as outlined by the design criteria.

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# The use of neoprene fabric evaluation in terms of comfort in child tracksuit production

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## REZUMAT – ABSTRACT

### Evaluarea confortului țesăturilor din neoprene destinate producției de treninguri pentru copii

Țesătura din neopren, utilizată la fabricarea echipamentelor pentru scufundări, wind surfing și pescuit, și-a făcut o intrare rapidă în industria modei. Această țesătură este rezistentă la produsele petroliere, la apă și la vânt. În același timp, este, de asemenea, rezistentă la temperaturile cuprinse între  $-50^{\circ}\text{C}$  și  $+120^{\circ}\text{C}$ . A fost utilizată la confecționarea îmbrăcăminte de zi cu zi datorită structurii sale flexibile. În acest studiu, au fost comparate țesăturile din neopren cu proprietăți diferite privind caracteristicile de confort și fizice pentru a determina dacă o țesătură din neoprene este adecvată în producția de treninguri pentru copii.

Cuvinte-cheie: neopren, țesături tricotate, îmbrăcăminte funcțională, confort termic, confort în mișcare

### The use of neoprene fabric evaluation in terms of comfort in child tracksuit production

Neoprene fabric, which used in the manufacture of diving, wind surfing and fishing clothes, made a rapid entry into the fashion industry. This fabric is resistant to petroleum products, water and wind. At the same time it is also resistant to temperatures between  $-50^{\circ}\text{C}$  and  $+120^{\circ}\text{C}$ . It has been used in daily wear because of the flexible structure. In this study, neoprene fabrics having different properties are compared in terms of comfort and physical properties in order to determine whether a neoprene fabric is suitable in the child tracksuit production.

Keywords: neoprene, knitted fabrics, functional clothing, thermal comfort, motion comfort

## INTRODUCTION

People for centuries use their textile products in almost every aspect of daily life. Previously only used for protecting and veil textiles, then it was used for the purpose of appealing to the beauty of the human spirit as fashion [1].

Many companies operating in the textile and apparel industry in the world and Turkey, products have begun to turn to 'high value added'. Smart and technical textiles with functionality in clothing are between the expectations. Waterproof and sunlight, anti-bacterial and breathable fabrics can be given as examples of functional textiles (figure 1).

Textile materials which are used depending on the time and fashion are changing the direction of raw and the structural characteristics frequently. Neoprene fabric is used in daily life in the examples of this situation.

Neoprene is the trade name of polychloroprene material developed by Dupont [2]. In 1931, the United States, Arnold Collins, discovered synthetic rubber neoprene. Neoprene was produced first commercially by the Dupont company under the name of Duprene and 1 pound as the main \$ 1,250 tonnes in 1932. In the early years people were not interested with this rubber because of insufficient properties of synthetic rubber. But in 1934, Dayton Rubber Manufacturing Co. company mentioned that the

rubber can be used in car tires and its usage increased in industry [3].

In this study, a variety of tests made to decide whether it is appropriate for the child tracksuit production of neoprene fabric which is in functional textile groups. For this purpose fabrics are compared and evaluated for physical and thermal comfort properties.

### Neoprene in garment production

Neoprene was worn for the first time in 1950 as a garment. Brothers Bob and Bill Meistrell discovered neoprene to keep their body temperature warm and protect them from the cold in the water during surfing. Neoprene with Dive N 'Surf name has begun selling in Southern California. Thus the modern neoprene wetsuit was born [13].

Later, the brothers created swimmers, sports bras and accessories which name was Body Glove in increasing popularity in the 1980s. Thus, neoprene, which used in wetsuit, has been used in different areas and brands. Anymore, neoprene represents textures which are easily accessible to everyone and may be preferred in everyday wear. Neoprene, which has become the most comfortable part of everyday clothing, is considered as an alternative to leather fabric surfaces [13].

Neoprene fabric is produced open width. In this way, fabrics crease does not occur in the tube knitted



Fig. 1. Neoprene garment samples [12]

fabrics and fabric utilization efficiency considerably increased. Neoprene fabric has a more stable structure than the other knitted fabric and so there is not turning aside problem. Therefore, sewing process can be made more comfortable.

In order to obtain a flexible structure vivid color in the collection and laser-cut details, have come to the fore in recent years. Neoprene skirt, sweatshirts, dresses, skinny cigarette pants, vests and jackets as products were studied in the winter collection intensively [14]. Neoprene's technical specifications are as follows:

- Neoprene is resistant to atmospheric conditions, the petroleum products in the form of liquids and gases, ozone, water and salt water [5].
- Neoprene can resist to temperatures of  $-50\text{ }^{\circ}\text{C}$  to  $120\text{ }^{\circ}\text{C}$  [4].
- Neoprene is a synthetic elastomer such as latex or solid state within the flexible foam. Rather than sulfur, it is vulcanized with metal oxide. It raises the flammability modified isocyanate [6].
- Non flammable characteristic is good [7].
- Washable, tensile strength is high. Cold, heat conductivity is poor [8].
- Neoprene is resistant to oil, chemicals, light, high temperatures and electric current [9].
- The coloring of neoprene is difficult. Digital printing can be done on all products [7].

Today, neoprene is used in a variety of fields. Usage areas of neoprene are as follows:

- The solid state of neoprene used in the manufacture of mechanical rubber parts, fuel hoses, electric cables, and the coating of special equipment, as a binder in rocket fuel, in the manufacture of gaskets and seals, conveyor belt and production of protective material [6].
- Neoprene used in air bags, life jackets, protective clothing and aircraft interiors [7].
- Neoprene used in sports clothing and garments, such as diving, wind surfing, fishing and the manufacture of medical products such as vests and knee braces [5].
- Neoprene used in cooking gloves, cup cooler, computer bag, mobile phone pouch, bottle cap, mouse pad, the American service production [11].

## PREVIOUS STUDIES

Neoprene studies about smart textiles, multi-functional textiles, wetsuit are as follows. Vahapoğlu V. (2006) gave information about the historical development of the synthetic rubber industry and the current

the general characteristics of most consumption synthetic rubber [3]. Bulut and Sular (2007) has made work about coating and lamination methods, uses, production techniques and performance testing of coated laminated fabrics [6]. Halaçeli (2008) gave information about high-performance textiles, which sports clothes and space research results developed, use together breathable, waterproof fabric in daily wear [16]. Bulgunand Yılmaz (2009) has done tests on the structure of fire suits, thermal protection within the scope of performance evaluation and have explored firefighter clothing innovation in the design [17]. Karahanet et al. (2009) gave information about textile structures used in space applications and the latest studies in this area [18]. Öndoğanet et al. (2014), evaluated the clothing, the fabric, the fit to the body, their production techniques and models of athletics sport the environmental requirements [20]. Erdoğanet et al. (2014) did collection work about diving suits, characteristics, uses, accessories, materials used in the clothing [21].

## MATERIALS AND METHOD

### Material

Neoprene knitted fabrics were used in this study. Neoprene production is made to form double face circular knitting machine. Both sides of the neoprene fabric may be manufactured in different contents or the same color. Thus, it is possible to produce a double face fabric. For example polyester-elastane, both sides; a face gray melange dark gray melange, other side; polyester-elastane face as the other side of viscose-elastan. In between the monofilament structure that connection will provide air circulation, improves volumetric structure and provides the breathing of the fabric.

Yarn in the production of neoprene fabric is used approximately two times more than the production of other knitted fabrics. Neoprene fabric production yield is 50% lower than the other knitted fabric [10]. Four different types of fabrics are selected in the scope of this research. Neoprene fabrics approved for use in the production tracksuit is preferred in the choice of fabrics. All of the fabrics have been formed double faced fabrics. All fabrics contained elastane, fabrics according to the fibers type and ratio are given in table 1 below.

### Method

In this study, the structural characteristics and specifications, which are effective on thermal comfort properties of the fabrics used in this study, were measured with using objective methods and in accordance with the relevant standards and device instructions.

Especially, due to abrasion of knee area of children tracksuits, abrasion properties of fabric, which is used in production tracksuit, are important. Therefore the fabric abrasion resistance was measured under pressure 9kp according to EN ISO 12947-2 standard

Table 1

Fabric code	Type of fabric (%)	Weight (gr/m <sup>2</sup> )	Thickness (m)	Front/back side content
1	90 PES -10 EA	340.06	0.002080	Front/Back: PES-EA
2	60 PES-30 COTTON-10 EA	402.48	0.003151	Front/Back: PES-COTON-EA
3	70 PES-20 VISCON-10 EA	390.58	0.002660	Front: PES Back: VISCON
4	95 PES-5 EA	305.14	0.000967	Front/Back: PES-EA

in the Martindale abrasion tester. One of the most important properties of comfort is also air permeability. Air permeability measurements were made according to TS 391 EN ISO 9237 standard (and 20 cm<sup>2</sup> test area). The pressure difference is 100 Pa in the Textest FX 3300 device. The fabrics used in this study should not hinder the movement, but it should fit properly on the body at the end of the movement. So the ability of the stretch-return is expected to be good. This reason, stretch-return characteristics of the fabric were tested to determine stretching and deformation. A mechanism is prepared according to ASTM D 2594-04 standard. Two nails are mounted on a flat surface. Nails distance between was determined according to standard by calculating the stretch ratio of the fabric sample size. Fabric samples were sewed in the form of a tube and passed to the hanger and hanger are also attached to nails and tension was provided. Samples of fabric were tested for comfortable products by considering the width and length elongation value. All of the fabric is knitted. Bursting strength was measured in order to assess the effect of stress caused by wear versatile force. Measurement was performed in Truburst Bursting Strength Testing device according to TS 393 EN ISO 13938-1 standard. Dimensional change of the fabrics was evaluated after washing. Therefore, the fabrics were washed with liquid detergent at 40 °C in daily wash programme. The fabrics were centrifuged at 800 rpm and dried in the air. Width and height measurements were made with a tape measure. In order to evaluate the attitude of the fabric, circular flexure test was made according to ASTM D 4032 standard. The average of forces, pushing the fabric circle, was calculated. The weight of the fabric according to the TS EN 12127 standard was determined on sensitive scales. Average value was multiplied by 100 and g/m<sup>2</sup> weight was determined. Alambeta and MMT devices have been used to determine the thermal properties of the fabric. Thermal resistance, thermal conductivity and heat absorbance values were found with Alambeta tester. Thermal properties of the fabric are evaluated according to the obtained data were compared. The body temperature of the daily clothing such as tracksuit is expected to keep the balance. Therefore, the liquid caused by body heat is very important in terms of outer surface to move quickly and give people a feeling of dryness for thermal comfort. MMT (Moisture Management Tester) tester was used to measure the moisture transport properties of the fabric.

## EXPERIMENTAL RESULTS

Abrasion and thread breakage in the fabric samples wasn't observed on the results of 50,000 abrasion cycles. Therefore, the fabrics were analyzed to evaluate the test results whether weight loss. Abrasion test results are shown in table 2. Maximum weight loss was observed in the number 1 fabric. Air permeability test results are shown in figure 2.

Table 2

Fabric code	Previous weight (gr/m <sup>2</sup> )	Next weight (gr/m <sup>2</sup> )	% Weight loss
1	37.8	36.4	3.7
2	45.7	45.2	1.09
3	44.7	44.2	1.11
4	35.2	34.8	1.13

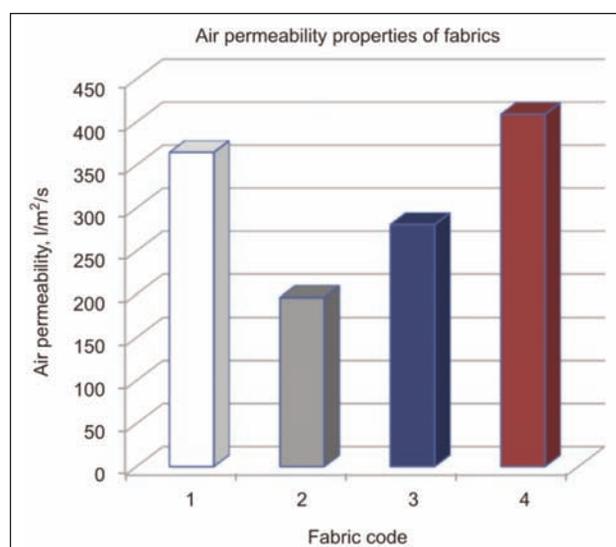


Fig. 2. Air permeability properties of fabrics

Width and length stretch-return values of the fabrics are shown in table 3. Burst or deformation wasn't observed in any of the fabrics according to the pressure 800 kpa in the burst strength measurement test conducted. The dimensional change wasn't observed according to test results of the washing dimensional exchange on all fabrics at the end of the measurements. The force, which is required to push the number 2 fabric, was the maximum level value of 18.9 Newton according to the circular flexure test result. This was followed by respectively 3, 1 and 4 fabrics (figure 3).

Table 3

Fabric code	Width stretch (cm)			Length stretch (cm)		
	Initial length	Recent length	% Change	Initial length	Recent length	% Change
1	15	15	0	15	15	0
2	15	15.15	1	15	15	0
3	15	15	0	15	15	0
4	15	15.25	1.66	15	15.1	0.66

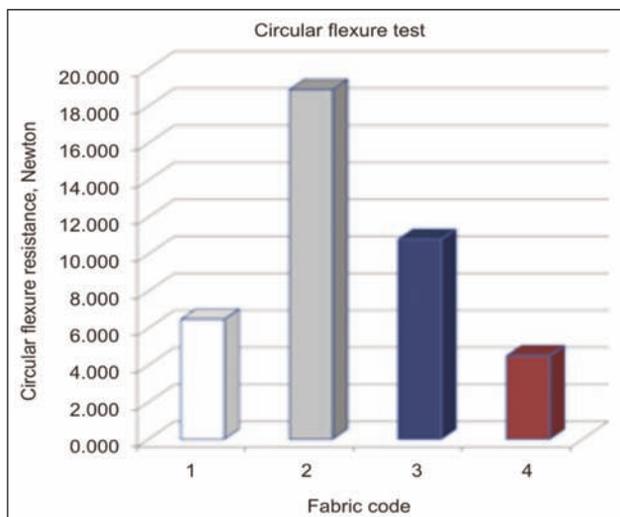


Fig. 3. Force values applied to fabric (Newton)

The results of the Alambeta device are shown in table 4. The thermal resistance of number 2 fabric, which is the thicker fabric, is highest (figure 4). Values of thickness, weight, and thermal resistance are linearly as expected.

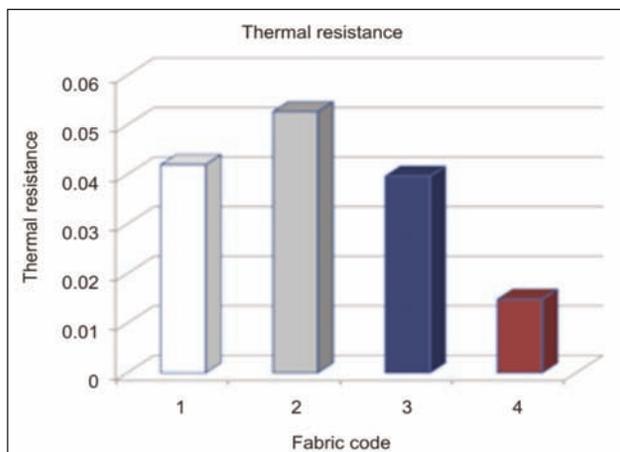


Fig. 4. Thermal resistance values of fabrics

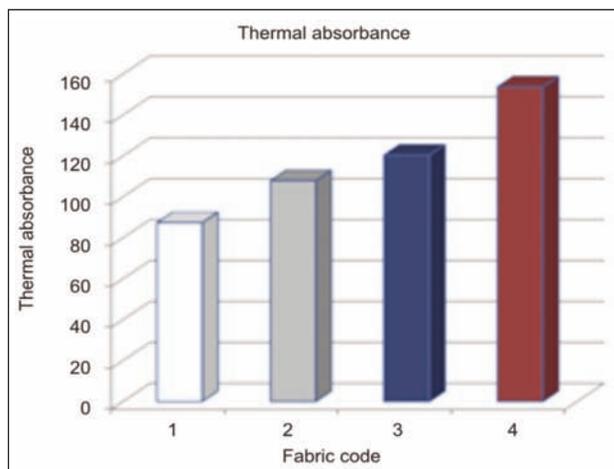


Fig. 5. Thermal absorbance values of fabrics

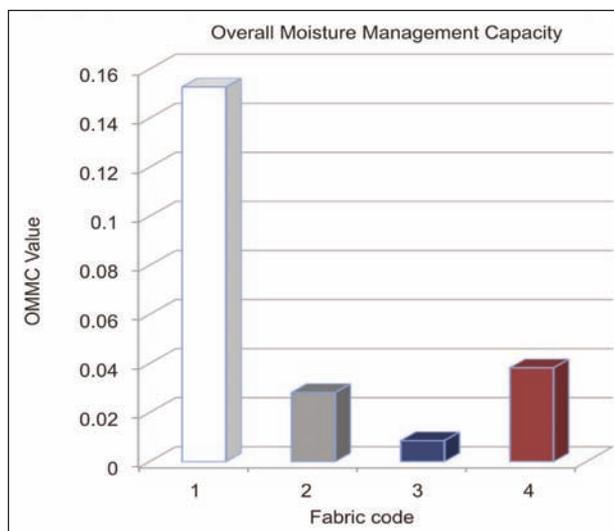


Fig. 6. OMMC Graph

Thermal absorbance values of all the fabrics were below than 400 (figure 5). During the initial contact of the skin with the fabrics, it was found that it does not give a very cold feeling.

Overall Moisture Management Capacity (OMMC) value is shown in figure 6, according to MMT test. Four number fabric has the best moisture transmission fabric as shown in figure 6.

## CONCLUSIONS

Four different fabrics were analyzed in this study. It was planned to be used in the production of

Table 4

Alambeta	Thermal resistance	Thickness	Thermal absorbance	Thermal conductivity
1	0.042	0.00208	87.42	0.049
2	0.0527	0.003151	107.966	0.0599
3	0.0397	0.00266	120.466	0.067
4	0.01487	0.000967	153.79	0.065

children's tracksuit, and so assessments were made according to the criteria sought in tracksuit.

Abrasion of child tracksuits especially can become much in the knee area. Therefore, it was made test to determine abrasion of the fabric and it was observed that no abrasion on the test results. This is among the expected results in the production of a tracksuit. This is advantageous in children's products and this provides the possibility for longer use.

The air permeability values of the Neoprene fabric are good generally due to monofilament which is an intermediate layer between the front and back surfaces of the fabric. It was determined that there is the linear ratio between air permeability with fabric weight-thickness on the test results. Fabric content of %95 PES-5EA is thinner than other types of fabrics. Due to both thinner fabric and the least weight, code 4 fabric has got the highest the air permeability value. For this reason the code 4 fabric can be advisable for warmer climates.

When the stretch-return ability test results in the study was conducted to evaluate fabrics, any distortion wasn't observed in the fabrics. In this case it can be said that neoprene fabrics have a good return feature. Thus, occurring deformation will not happen frequently in the knee or elbow area of the tracksuit. In this study, although the criteria are evaluated in children tracksuit, the same criteria are also required in the adult tracksuit. Even, the visuals in this direction, has a far greater significance for adults. The load on the knee is higher in adults and the knee trail is much more pronounced. This problem is possible to eliminate with neoprene knitted fabrics, content of %10 elastane. Voluminous soft touch fabric also shows a soft barrier between the surface of the knee. For this reason particularly, it can be reduced pain sensation of feeling with the design double-layer models of the child tracksuits for the knee area when children fall on the ground. When the rate of %5 elastane fabric, the return wasn't also about %1 at the end of the width-length stretching. Therefore %10 elastane is

more suitable for tracksuits fabric. Thus, tracksuits, which are produced neoprene fabric, will keep a long time in the visual aesthetics of everyday wear. And thus the use of neoprene fabric can get round their everyday outerwear design.

When looking from the circular resistance test results, it was observed that %60 COTTON-30PES-10EA content of fabrics showed largest load bending. When the weight of neoprene fabric increases, the circular bending resistance increases. In recent years, joining heavy weight products are possible with advanced sewing machines without any problems. Rigid properties of the fabrics also provide ease of manufacture.

When the thickness of fabric was increased, it was determined to the thermal resistance increased, according to Alambeta test results to the thermal conductivity, which is an important factor for child garment fabric. Especially, due to the code 2 fabric, which is the thickest fabric, has high thermal resistance and low air permeability, it will increase the preference for cold climates. Because of heat absorbance values below 400 of examined, all fabrics will not be cold feeling in contact with the first skin, it means that children feel good during wearing their clothes.

Another important factor is moisture absorption children's clothing items. Best moisture absorption was observed %60 COTTON-30PES-10EA containing fabric. This is probably because of the relatively low weight compared to other fabrics and fine fabric. In fact, all of OMMC values were below 0.2. Therefore, OMMC of neoprene fabric used in this study is not good. It is expected to say to be well that the values of moisture transmission is above 0.4. Therefore it mustn't be preferred neoprene fabric in areas where there is intense physical activity.

Neoprene fabric can easily be used in the manufacture of clothing or tracksuit due to advantages as an aesthetic in normal daily life, easily not to be deformed, the usage is durable and printing process is applied with vivid colors.

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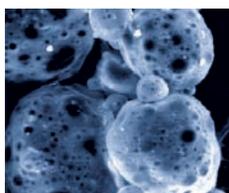
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# Developing a software calculating fabric consumption of various bathrobe models

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## REZUMAT – ABSTRACT

### Dezvoltarea unui software de calculare a consumului de țesătură pentru diferite modele de halat de baie

Având în vedere cererile clienților și dinamica sectorului textil, ar trebui stabilite prețuri corecte în foarte scurt timp atunci când clienții le solicită pentru diferite stiluri de îmbrăcăminte. Clienții solicită adesea oferte de preț de la producătorii de îmbrăcăminte. În acest caz, producătorul de îmbrăcăminte trebuie să se miște rapid și precis pentru a determina consumul unitar al confecției. Este foarte important să se cunoască costul corect al țesăturii în stabilirea prețului produsului de îmbrăcăminte care urmează să fie creat. În general, costul țesăturii utilizate în articolele de îmbrăcăminte reprezintă 60–70% din costul total. Producătorii își asumă riscuri atunci când stabilesc prețul îmbrăcăminte pe baza consumului aproximativ al țesăturilor. Toleranța la consumul de țesături poate fi luată mai mult ca un beneficiu, dar atunci comanda nu poate fi plasată de client din cauza prețului ridicat. În sistemele CAD, calculul consumului de țesături nu se poate face cu ușurință. În cadrul acestei cercetări, software-ul a fost dezvoltat pentru a calcula rapid consumul pe unitatea de îmbrăcăminte. Materialul pentru modelele de halat de baie a fost selectat și s-au folosit datele de la o fabrică care produce halate de baie. Rezultatele programului, dezvoltate împreună cu software-ul, sunt comparate cu cifrele experimentale. Astfel a fost posibil să se determine consumul de țesătură cu o precizie de 98,2% într-un timp foarte scurt, prin utilizarea sistemului dezvoltat ( $R^2 > 0,982$ ).

Cuvinte-cheie: consum de țesătură, determinare rapidă a prețurilor, raport de utilizare a țesăturii, software

### Developing a software calculating fabric consumption of various bathrobe models

Considering the customer requests and speed in the textile sector, very fast and accurate pricing should be done when the customers ask for very urgent prices for different styles. Customers often ask for sample pricing from apparel manufacturers. In this case, the garment manufacturer has to move quickly and accurately in determining the unit consumption of the garment. It is very important to know the correct fabric cost in pricing the garment to be produced. In general, the cost of fabric in garments accounts for 60–70% of the total cost. Manufacturers take risks when pricing the garment with the approximate fabric consumption. Fabric consumption tolerance can be taken higher to be a benefit, but then the order may not be placed by the customer due to high price. In CAD systems, calculation of fabric consumption can not be done easily. In this research, the software has been developed to calculate the unit usage of a garment quickly. Bathrobemodels were selected as a material and the data of a factory that produces bathrobe was used. The results of the program, which is developed with the software, are compared with the experimental figures. As a result, it was possible to determine fabric consumption with a reliability of 98.2% in a very short time by using the developed system ( $R^2 > 0.982$ ).

Keywords: fabric consumption, faster pricing, fabric utilization ratio, software

## INTRODUCTION

In textile industry, where customers ask for very urgent prices, companies have to inform them the accurate prices in a very short time. Especially in the sectors such as the apparel sector where high competition and variable factors are intensive, the use of effective production methods has become compulsory [1]. Fabric is generally the most significant factor in costing of garment. Fabric accounts for 60 to 70% of the total cost of basic-styled garments. The cost of fabric depends on the type of fabric is going to be used to make the garment.

The fabric consumption of a garment is affected by the model, the measurements, the fabric width, and the size breakdown. Even for the same garment, the fabric consumption can vary in different fabric widths [2]. The garment manufacturer has to know the fabric consumption to be able to calculate the fabric cost of

the garment and this is very important to make the correct costing, in today's world where the competition between the companies is highly increased. When the fabric width is known for a particular style, the length of the fabric to produce this style is called fabric consumption. Yesilpinaret al. have developed the software that enables the fabric consumptions of different shirt models to be estimated in a speedy way [3], [6]. Similarly, the software developed enables the estimation of fabric consumptions of different trouser models in a speedy way [4]. For cost and pricing purposes, Değirmenci and Çelik designed a computer program that helps calculate the unit costs of knitted fabrics [5]. A software has been developed using the Microsoft Visual Basic 6.0 programming language for clothing garment expense (tshirt models) for knitted garment enterprises. After the waste allowance is added on to the fabric yardage taken

from the system, the total need for the order is determined [7]. Khalilov and Bozkurt have developed a software using Microsoft Access 2003, VBA and SQL programming language, which calculates pants fabric cost for manufacturers that produce denim pants [8]. Vuruskan has studied production parameters in knitted garment manufactures and prepared a computer program that calculates unit cost of 13 different clothes styles (t-shirt, jacket, athlete, skirt, blouse). The system using MySQL as a database computes the product cost in terms of user input data and archive information [9].

It is extremely important that the fabric is used efficiently. Various studies have been done to determine the use of fabric and to reduce losses. Ng, Hui and Leaf aimed to estimate the loss of fabric in the laying process by developing a mathematical model. They separated the fabric losses into two groups, one inside and one outside the marker, and they created a mathematical formula by using the parameters used in the cutting plan and the factors which affect the fabric spreading in manufacturing [10]. Baykal and Göçer have compared the process counts and durations, cutting plan productivity, tape efficiencies and second quality ratios for different fabrics and different models during a garment operation [11].

Computer programs and softwares are widely used in order to find solutions to problems experienced in textile, ready-to-wear and fashion. Artificial Neural Networks, Fuzzy Logic, Genetic Algorithms and a Hybrid Planning Processes have been used to reduce fabric usage, mold development, analyzing and improving faulty fabrics in garment sector [12–17].

CAD/CAM systems provide significant advantages to apparel manufacturers. Hands et al. have experimentally proven in that the CAD system increases the rate of use of fabric usage and shortens the duration of pattern preparation [18, 19].

Antemie, et al have developed a new method and emphasized the improvement on the stability of theoretical estimations regarding material consumption for textile products by adding this new method to computer assisted technical design [20].

This study was carried out to determine the unit fabric consumption of different bathrobe models by using computer programme. The program developed to calculate bathrobe fabric usage is explained. Therefore, first of all, the fabric consumption of bathrobe models is determined practically by the CAD system. In the end, the results of the software are compared with the actual fabric consumptions of the CAD system.

## MATERIAL AND METHOD

### Material

The materials of this research consist of bathrobe models, fabrics, measurement charts, Konsan CAD system and computer programme which we created by using software. A garment manufacturing company which produce t-shirt, skirt, dress, towel, bathrobe and which use quite a lot of different kinds of woven

fabric is selected to take the actual unit fabric consumption of any style.

The selected company has a production capacity of 30,000 units a day. The company exports 95% of its products to European countries. Experimental studies related to the research patterns were made in the Konsan CAD system. The preparation of this research has taken 18 months together with the company. The company's process of calculating fabric consumption has been examined in the pattern department depending on the styles requested by the customers. The information required to determine the fabric consumption of a garment required for pricing has been arranged and classified. Information such as the order number of each bathrobe model, technical specifications, measurement charts, size breakdown, fabric usage rate and fabric width were analyzed. A total of 3042 purchase order, which consist of bathrobe product groups in various models, were included in the research. Software has been developed for the quick calculation of fabric consumption of a new style by using the previous marker plans. In total 3042 marker plans have been used.

### Method

After all the information about the marker plan of a specific style is entered into the computer, the programming language Microsoft Access 2013, Microsoft Excel 2013, Visual Studio 2012, C # (as encoding language), SQL Server 2014 and SQL (Structured Query Language) were used to calculate the fabric consumption in the requested size. In the evaluation of fabric consumption, two different metric values were considered.

- i. Practical metrics obtained using CAD system;
- ii. Theoretical unit obtained by computer software.

The actual fabric consumption and marking efficiency (fabric utilization rate) obtained from marker in the CAD system is taken into consideration for each model. The marker efficiency of the fabric can be seen in the marker layout in CAD system.

After the pattern layouts are made on the CAD screen, the unit fabric consumption for size M is calculated according to the following equation.

$$\text{Unit Fabric Consumption (m)} = L(i) / \sum S(i) \quad (1)$$

where  $L(i)$  is the  $i^{\text{th}}$  marker length, and  $\sum S(i)$  is the  $i^{\text{th}}$  total size in the marker.

Five bathrobe models, the most produced in the company, were examined in the study. These are kimono, shawl collar, single hooded, child and double hooded bathrobes. Some bathrobe models are shown in figure 1.



Fig. 1. Shawl collar, single hooded, child bathrobe models

Table 1

Pattern ID	Pattern no.	Pattern name	Model type	Measurement point	Size breakdown				Size name	Size	Unit Fabric Consumption (m)
					Size_1	Size_2	Size_3	Size_4			
16,00	ZC-1000-	267-1000	Double Hooded Bathrobe	Hem width	140,00	152,00	NULL	NULL	Size_1	M/L	3,05
16,00	ZC-1000-	267-1000	Double Hooded Bathrobe	Hem width	140,00	152,00	NULL	NULL	Size_2	L/XL	3,42
16,00	ZC-1000-	267-1000	Double Hooded Bathrobe	Length	105,00	113,00	NULL	NULL	Size_1	M/L	3,05
16,00	ZC-1000-	267-1000	Double Hooded Bathrobe	Length	105,00	113,00	NULL	NULL	Size_2	L/XL	3,42
16,00	ZC-1000-	267-1000	Double Hooded Bathrobe	Chest 1/2	62,00	68,00	NULL	NULL	Size_1	M/L	3,05
16,00	ZC-1000-	267-1000	Double Hooded Bathrobe	Chest 1/2	62,00	68,00	NULL	NULL	Size_2	L/XL	3,42
16,00	ZC-1000-	267-1000	Double Hooded Bathrobe	Arm length	75,00	79,00	NULL	NULL	Size_1	M/L	3,05
16,00	ZC-1000-	267-1000	Double Hooded Bathrobe	Arm length	75,00	79,00	NULL	NULL	Size_2	L/XL	3,42

### Computer program

In the research, original data of 313 marker plans from 3042 marker plans were recorded in the database of the developed program. The steps followed when developing the software are as follows.

1) The marker plan inputs in the company's database were arranged in Excel format in table 1. As input data; pattern number, pattern name, measurement chart, size breakdown, size name, model definition, fabric type, CAD productivity and actual fabric consumption were kept in a special format prepared during 1.5 years. Then the form was transformed to table 2. Table 2 (target\_table) is an example table in which a portion of the data is contained.

2) The Excel data shown in table 1 was transferred to a database named "unit fabric consumption" created in SQL Server 2014. Figure 2 shows the SQL Server Management Studio database and tables screen.

3) Three pieces "view"s were created to use the code to be written with SQL. "View"; are query interfaces that can be used to create new virtual output tables using relationships between these tables. A sample "view" and columns are shown in figure 3.

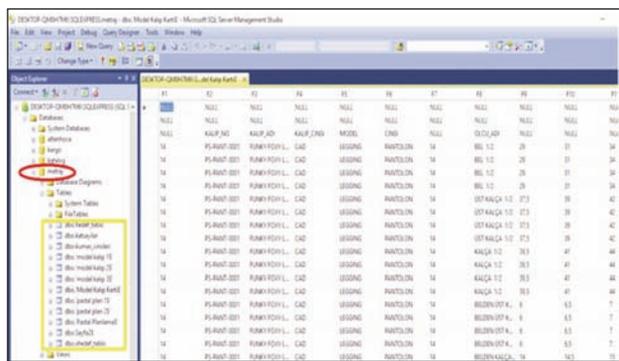


Fig. 2. SQL Server Management Studio database and tables screen

4) After 3 views were created, they were extracted with the following "cursor" written in the SQL language and incomplete or inconsistent data were eliminated. The data that can be used in the estimation were transferred to the file named "target\_table" based on the inputs used in the written program. Cursor is a database system structure that is written in SQL language and enables to process the data by examining line by line. With this process, data of 313 marker plans from 3042 were obtained. Screenshot of a section of the cursor code written in SQL Language is shown in figure 4.

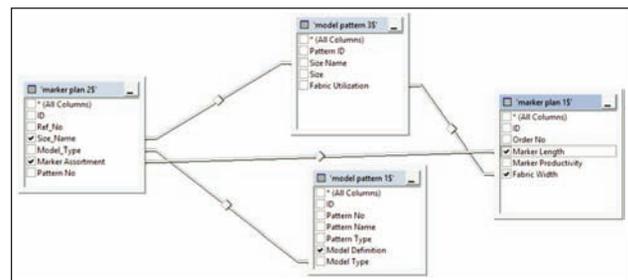


Fig. 3. The sample "View" to be used by SQL written code

```

SQLQueryLog - D:\MSB7M6\kaha (53) - x
DESKTOP-QM87M66\traj - dbc.View_3'

declare @rapson_buys float
declare @sep_eni float
declare @sep_buys float
declare @peraz_eni float
declare @peraz_buys float
declare @biris_metraj_gerceklezen float

open c
fetch next from c into @pastal_uzunlugu,@sorti,@cad_verimlilik,@pastal_eni,@kumas_cinsi,@biris_metraj_gerceklezen
while (@FETCH_STATUS = 0)
begin
select top 1 @boy_uzunlugu = TRY_CONVERT(FLOAT,EBAT_1) from View_4 where OLCU_ADI like 'BOYU' and
[PASTAL_BOVU] = @pastal_uzunlugu and [PASTAL_ASORI] = @sorti and
[PAS_VERDI] = @cad_verimlilik and [KUPAS_ENI] = @pastal_eni and
[KALIP_ADI] = @kumas_cinsi and [BIRIM_METRAJ] = @biris_metraj_gerceklezen

select top 1 @etek_covresi = TRY_CONVERT(FLOAT,EBAT_1) from View_4 where OLCU_ADI like 'BETEK' and
[PASTAL_BOVU] = @pastal_uzunlugu and [PASTAL_ASORI] = @sorti and
[PAS_VERDI] = @cad_verimlilik and [KUPAS_ENI] = @pastal_eni and
[KALIP_ADI] = @kumas_cinsi and [BIRIM_METRAJ] = @biris_metraj_gerceklezen

select top 1 @kol_buys = TRY_CONVERT(FLOAT,EBAT_1) from View_4 where OLCU_ADI like 'KOL BOYU' and
FRUKTAI_BOVU = @pastal_uzunlugu and FRUKTAI_ASORI = @sorti and

```

Fig. 4. Screenshot of a section of the cursor code written in SQL Language

Table 2

Marker No	Marker Length (cm)	Size Breakdown	CAD Fabric Utilization Ratio%	Marker Width (cm)	Model Type	Length (cm)	Hem (cm)	Sleeve Length (cm)	Armhole (cm)	Belt Width (cm)	Belt Length (cm)	Hodd Width (cm)	Hodd Length (cm)	Pocket Width (cm)	Pocket Length (cm)	Moulding Width (cm)	Moulding Length (cm)	Actual Unit Fabric Consumption (m)
669	241	1	88	157	2	60	95	29,5	0	3,5	130	22,5	28,5	14	15	0	0	1,26
670	241	1	88	157	2	66	103	34	0	3,5	140	23,5	29,5	14	15	0	0	1,43
671	241	1	88	157	2	70	109	37,5	0	3,5	150	24	30	15	16	0	0	1,58
672	241	1	88	157	2	73	117	40	0	3,5	150	24,5	30,5	15	16	0	0	1,68
673	241	1	88	157	2	85	126	0	0	0	150	25	31	16	17	0	0	1,8
674	257	2	89,8	157	2	60	95	29,5	0	3,5	130	22,5	28,5	14	15	0	0	1,26
675	257	2	89,8	157	2	66	103	34	0	3,5	140	23,5	29,5	14	15	0	0	1,43
676	257	2	89,8	157	2	70	109	37,5	0	3,5	150	24	30	15	16	0	0	1,58

The screenshot shows a database interface with a 'Tables' list on the left containing 'target table', 'co-efficient', 'model type', and 'pastingerror'. The main window displays the 'co-efficient' table with columns 'co-efficient no.' and 'co-efficient'. The data rows are numbered 115 to 130, with values ranging from 1,77639541 to 0,001356942.

Fig. 5. Screenshot of the database and “coefficient” tables used by the program

```

double hexapla(double x1, double x2, double x3, double x4, double x5, double x6, double x7, double x8, double x9, double x10, double x11, double x12, double x13, double x14, double x15)
{
    string sql = "select katsayi from katsayilar order by katsayino asc";
    OleDbDataAdapter a = new OleDbDataAdapter(sql, bcu);
    DataSet ds = new DataSet();
    a.Fill(ds);
    double sabit = Convert.ToDouble(ds.Tables[0].Rows[0][0]);
    double y = sabit + x1 * Convert.ToDouble(ds.Tables[0].Rows[1][0]) +
        x2 * Convert.ToDouble(ds.Tables[0].Rows[2][0]) +
        x3 * Convert.ToDouble(ds.Tables[0].Rows[3][0]) +
        x4 * Convert.ToDouble(ds.Tables[0].Rows[4][0]) +
        x5 * Convert.ToDouble(ds.Tables[0].Rows[5][0]) +
        x6 * Convert.ToDouble(ds.Tables[0].Rows[6][0]) +
        x7 * Convert.ToDouble(ds.Tables[0].Rows[7][0]) +
        x8 * Convert.ToDouble(ds.Tables[0].Rows[8][0]) +
        x9 * Convert.ToDouble(ds.Tables[0].Rows[9][0]) +
        x10 * Convert.ToDouble(ds.Tables[0].Rows[10][0]) +
        x11 * Convert.ToDouble(ds.Tables[0].Rows[11][0]) +
        x12 * Convert.ToDouble(ds.Tables[0].Rows[12][0]) +
        x13 * Convert.ToDouble(ds.Tables[0].Rows[13][0]) +
        x14 * Convert.ToDouble(ds.Tables[0].Rows[14][0]) +
        x15 * Convert.ToDouble(ds.Tables[0].Rows[15][0]);
    return y;
}

```

Fig. 6. Screenshot of the function which the program uses in estimation

5) Thousands of data were parsed by means of a cursor and the necessary data were retrieved again, then the data were transferred back to in Excel (table 2 – target\_table). For some sections the data were not available due to the bathrobe model.

6) In Excel, a regression analysis was performed to determine the relationship between input data and output data. As a result of the regression analysis, the coefficient relation between output and inputs is determined as shown in figure 5. The coefficient values for the inputs and outputs obtained at the end of the regression analysis are transferred to the previously prepared target table. Formula 2 contains the equation for the regression analysis.

*Predicted fabric consumption (m) = CAD Fabric Utilization Ratio \* Coefficient [2][2] (Column 2 of the 2nd column of the coefficient table) + Marker Width \* Coefficient [3][2] + Model Type \* Coefficient [4][2] + Length \* Coefficient [5][2] + Hem \* Coefficient [6][2] + Sleeve Length \* Coefficient [7][2] + Arm Hole \* Coefficient [8][2] + Belt Width \* Coefficient [9][2] + Belt Length \* Coefficient [10][2] + Hood Width \* Coefficient [11][2] + Hood Length \* Coefficient [12][2] + Pocket Width \* Coefficient [13][2] + Pocket Length*

$$* \text{Coefficient [14][2]} + \text{Moulding Width} * \text{Coefficient [15][2]} + \text{Moulding Length} * \text{Coefficient [16][2]} + \text{Coefficient [1][2]} . \quad (2)$$

7) The data in the “coefficients” table are used to transform the input form to the output form and the estimated value is calculated by the function written in the C # programming language by creating the estimation formula in figure 6.

8) This database in Access is finally delivered with the program written in C # programming language in Visual Studio 2012.

## FINDINGS

Estimation can be achieved on the main screen of figure 6 of the developed program. The data for the bathrobe to be estimated are entered to program in order to make estimations. It was initially tested with real data at 20%, which were not used in testing the program generated with real data. The estimates of the program have been found to be very close to the actual values (98.25%).

For example, when the marker length, size breakdown, estimated CAD efficiency, fabric width and bathrobe measurements for double-hooded bathrobe model shown on figure 7 were entered then the “Calculate” button was pressed, the estimated unit fabric consumption in the seconds is calculated as 1.47 cm. The actual fabric consumption for this



It is a very important achievement to start production by determining the optimal fabric width for garment manufacturers.

- The amount of fabric required according to the order quantity can be determined and contributed to the planning.
- Estimating power of the program written in the research is ( $R^2 > 0.982$ ). The reliability of the system is 98.25%. Therefore, in a short period fabric consumption can be determined with a close approximation to the fact. In fact, fabric consump-

tion that can be determined by a long study can be determined in a few seconds via software.

In this research, a software was developed that calculates the fabric consumption of bathrobe models. Software that calculates the fabric consumption of other types of clothing, such as trousers, dresses, t-shirts and skirts, can be developed in a similar way.

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# The potential of biofilms from moving bed bioreactors to increase the efficiency of textile industry wastewater treatment

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## REZUMAT – ABSTRACT

### Potențialul biofilmelor din bazinele cu biofilm fixat pe suport artificial mobil în creșterea eficienței de epurare a apelor uzate generate de către industria textilă

Procesele din industria textilă produc unele dintre cele mai poluate ape reziduale din lume. Apele reziduale din industria textilă sunt, de asemenea, foarte variabile (variază în funcție de timp și de fabrică) și conțin o mare varietate de poluanți. Acest lucru face ca tratamentul efluenților din industria textilă să fie complex, specific și scump. Numeroase combinații de tehnologii de tratare a apelor reziduale sunt aplicate în prezent în industria textilă, însă metodele care funcționează sunt adesea necorespunzătoare, insuficiente, necorespunzătoare sau nesustenabile. Odată cu evoluția industriei textile, cercetarea din domeniul epurării apelor reziduale trebuie să țină pasul cu cerințe care sunt în continuă creștere. Obiectivul mai extins al epurării apelor reziduale din industria textilă este maximizarea eficienței eliminării poluanților, în timp ce se eliberează efluenți pe care societatea îi consideră acceptabili și siguri din punct de vedere ecologic. În ultimii zece ani s-au făcut mari eforturi pentru a reduce consumul biochimic de oxigen ( $CBO_5$ ) și amoniac ( $NH_4^+$ ) în apele reziduale. Aceste progrese conduc la întrebarea: intensificarea utilizării acestor tehnologii din industria textilă poate să ducă la creșterea eficienței sale? Echipa de cercetare a analizat epurarea apei prin biomineralizare aerobă prin intermediul biofilmelor microbiene imobilizate pe suprafețe solide și situate în reactoare cu biofilm fixat pe suport artificial mobil (MBBRs). Aceste biofilme sunt selectate pentru oxidarea carbonului și amoniacului. Autorii compară potențialul de biotratat cu nămol activ cu performanța bioreactorului de tip MBBR. Rezultatele sunt utilizate pentru a evalua potențialul MBBR ca soluție de reducere a costurilor în instalațiile de epurare a apelor reziduale din industria textilă. Analiza susține că modernizarea unor astfel de stații cu o utilizare mai intensă a biotehnologiei MBBR ar crește sustenabilitatea și atitudinea prietenoasă față de mediu. Autorii abordează, de asemenea, direcțiile de cercetare și rețerele pentru extinderea efectelor MBBR asupra tratării apelor reziduale din industria textilă.

Cuvinte-cheie: biofilme, tratarea apelor reziduale,  $CBO_5$ , amoniac, industria textilă

### The potential of biofilms from moving bed bioreactors to increase the efficiency of textile industry wastewater treatment

Textile industry processes produce some of the most heavily polluted wastewater worldwide. Wastewater from textile industry is also highly variable (it varies with time and among factories) and contains wide diversity of pollutants. This makes the treatment of textile industry effluents, complex, site-specific and expensive. Numerous combinations of wastewater treatment technologies are currently applied in the textile industry, yet methods that work for one emitter are often unsuitable, insufficient, not necessary or unsustainable to another. As textile industry evolves, its water treatment research also has to keep pace with increasing demands. The broader aim of the textile industry wastewater treatment is to maximize the efficiency of pollutant removal, while releasing effluents that society considers as being environmentally acceptable or safe. In the last ten years great strides have been made in the ability to lower the biological oxygen demand (BOD) and ammonium ( $NH_4^+$ ) in wastewater. These advances elicit the question: can intensifying the usage of such technologies in the textile industry also increase its efficiency? The research team analysed water treatment by aerobic biomineralization via microbial biofilms immobilized on solid surfaces and hosted in Moving Bed Bio-Reactors (MBBRs). These biofilms are selected for carbon oxidation and ammonia oxidation. The authors compare the potential of active sludge biotreatment with the performance of MBBRs. The results are used to evaluate the potential of MBBRs as a cost-reducing solution in textile wastewater treatment plants. Our analysis supports that upgrading such stations to more heavily usage of MBBR biotechnology would increase their sustainability and environmental friendliness. The authors also discuss research directions and milestones for expanding the effects of MBBRs on the textile industry wastewater treatment.

Keywords: biofilms, wastewater treatment, BOD, ammonia, textile industry

## INTRODUCTION

The textile industry is one of the largest water polluter worldwide in terms of the number of chemicals produced, in the amount of chemicals released and in the amount of wastewater produced [1]. This industry generates approximately 70 billion tons of wastewater

each year [2]. Waste water produced by this industry is too toxic to be released in nature, and has to be treated. As water treatment plants that are commonly used to treat domestic water waste cannot handle textile industry outflows, textile industry factories use custom made water treatment stations.

The various processes used by the textile industry (staining, printing, bleaching, scouring, defatting, hydrolysis, etc.) produce dissimilar types of pollution. These waste streams vary greatly with regards to the chemicals they contain and their concentration, but may also vary considerably in time even for the same factory. Legislation regarding what is, and what is not, allowed to be discarded in nature by the textile industry varies from one country to another. In this paper are presented the legal limits from three textile industry producers: Germany, China and Romania.

Wastewater treatment plants (WWTP) used in the textile industry are complex and in most cases custom-designed for specific emitters and chemicals. The most important criteria for judging these stations are: chemical specificity, efficiency at removing pollutants and cost. Water treatment technologies have to keep pace with a fast-evolving textile industry (in terms of materials and methods). Fast response in water treatment technologies is the key to balance legal pollutant requirements with the economical sustainability of the textile industry.

The most frequent and abundant pollutants produced by the textile industry include (in no specific order): dyes, sulfide, enzymes, starch, ammonia, aniline, organic carbon, disinfectants, insecticides, NaOH, surfactants, fats, waxes, enzymes, peroxide, metals, salts, solvents, chlorinated compounds, acetate, softeners, urea and formaldehyde. In this paper we focus on advances in the removal of ammonia nitrogen ( $\text{NH}_4^+-\text{N}$ ) and the fraction of organic carbon that can be lowered by aerobic respiration and called Biological Oxygen Demand (BOD). These choices ( $\text{NH}_4^+-\text{N}$  and BOD) came from increasing pressure from the public and from legislation to control emissions that can produce eutrophic pollution or anoxia in ecosystems.

## MBBR UTILIZATION FOR THE TREATMENT OF THE TEXTILE WASTEWATERS

### Biofilm carriers

The Moving Bed Biofilm Reactor (MBBR) process is based on the aerobic biofilm principle and has the advantages of activated sludge and other biofilm systems and in the same time exceeds the disadvantages of activated sludge processes. The biofilm carriers are made from varied materials, but most of them are made from high density polyethylene or varied materials mixtures based on polyethylene. The materials for the realization of biomedica are selected based on several criteria such as: porosity, erosion resistance, size and density (the biofilm carriers must have a close-to-water density). Using a relatively small reactor volume can maintain a high biological activity by utilizing biofilm carriers with a large specific surface area ( $\text{m}^2/\text{m}^3$ ). The biofilm carriers are mixed inside the wastewater tanks with the help of bubbles produced by the diffusers of the aeration system. This type of free biomedica is the most efficient since the clogging is not possible.

There are several models of biofilm carriers worldwide and a few of these are shown in figure 1 [3, 4].



Fig. 1. Biofilm carriers [3, 4]

As shown in figure 1 and figure 2, the biofilm carriers have an internal zone, where the biofilm is created and protected. These internal surfaces provide protected areas and optimal conditions for the bacteria culture to thrive and develop. The biofilm inside each carrier element protects the bacterial cultures against the industrial processes with fluctuations in pollutants' discharging. The free carriers represent a stable place for the microorganisms to grow, compared to the activated sludge process, so less tank volume is needed. Essentially nutrient and dissolved oxygen levels are the only control aspects for the process operation. MBBRs are used to remove biological and chemical oxygen demand (BOD and COD) from wastewater streams. Nitrogen removal is also efficient in MBBRs. Existing activated sludge wastewater treatment plants can be upgraded using biofilm carriers, to achieve higher efficiencies for COD, BOD, nitrogen and phosphorus removal. The MBBR technology provided satisfaction to thousands of both municipal and industrial beneficiaries worldwide.

A new type of biofilm carrier was developed, which evolved from the existing models using the Kaldness process and were modified to obtain a higher strength and a larger surface for biofilm development. In figure 2 is presented the new type of biofilm carrier, developed by some of the authors.

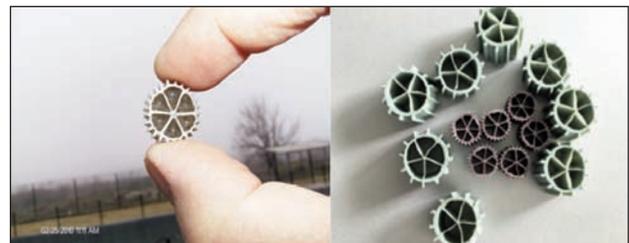


Fig. 2. Biofilm carriers

### Biofilms development and utilization in process engineering

Biofilms are assemblages of microorganisms, encased in a matrix, that function as a cooperative consortium, the biofilm mode of life being a feature common to most microorganisms in natural, medical and engineered systems including those involved in wastewater treatment [5]. Many bacteria can adhere non-specifically to different surfaces but some bacteria adhere best to hydrophobic substrates whereas other

adhere best to hydrophilic substrates [6] or to intermediate materials [7–8]. Interesting, the growing conditions influence the ability of the same bacterium. The chemical nature of the biomedica (biofilm carriers) to be used in a waste water treatment plants should be carefully chosen to promote biofilm formation and activity.

According to Costerton, in the last decade it became clear that bacteria live preferentially in multicellular biofilms in which cells established optimal metabolic interaction for their persistence in the ecosystem [9]. These biofilm communities have developed structures and strategies in response to attacks by chemical and biological antagonists, as well as for the availability of nutrients. The predominance of biofilms in natural and engineered ecosystems is of paramount importance for the processes occurring in those ecosystems, and for understanding them. Chronologically, biofilms were first observed in oligotrophic mountain streams and afterwards in natural aquatic systems of increasing nutrient content, culminating in abattoir effluents, deeply questioning about the biological significance of biofilms. Nowadays it is generally accepted that the bulk of bacterial transformations that occur in the biosphere take place in these sessile microbial communities, which are also important in engineered ecosystems.

The followings are the advantages of bacterial life in biofilms [6, 10]:

a) Increased nutrient availability, as compared with free, planctonic way of life, the chemicals, including macromolecules, being subject of adsorption at different (clean) surfaces immersed into a natural or engineered ecosystem. This process is obvious especially in environments where the nutrient concentrations are very low (open ocean, mountain lakes etc.).

b) The biological diversity of biofilms occurring in natural and engineered ecosystems favors very complex interactions between individual cells, including complex catabolic and anabolic reactions, based on complementary nutritional and physiological associations between bacteria, thus enabling the biofilm with improved capabilities. This aspect is essential for biofilms active in different type of wastewater plants.

c) The dense structure of biofilm and close physical contact between individual cells promote increased genetic exchanges between cells as well as cell-cell signalling processes, called quorum sensing.

d) Protection from harmful factors such as antibiotics, chlorine (disinfectants, in general) and heavy metals, the increased resistance being based on extra parietal structures of bacteria and complex inter-cellular matrix of biofilm, as well as to mechanism(s) occurring at individual level. There is evidence that bacteria, especially those bellow the biofilm-water interface, more closely to the solid substrate, are protected against grazing by protozoa and metazoa, parasitism by bacteriophages or by bacteria, (e.g. *Bdellovibrio*), as well as from predation by amobe.

The followings are the disadvantages of bacterial life in biofilms [6]:

a) As they are fixed (at a given scale time) in biofilm bacteria seems to be more exposed to grazing as compared with free, planctonic bacteria or actively (e.g. flagella etc.) moving bacteria.

b) Due to the complex structure and dimensions of biofilms (thickness from micrometers to millimeters or more) different type of gradients occur. Aerobic microorganisms occur at the biofilm-water interface consuming the molecular oxygen which, at deeper position in the biofilm, become absent, thus creating conditions for anaerobic bacteria, capable of either anaerobic respiration (on nitrate, for example, if present) or fermentation.

For the point of view of the usefulness of biofilms in MBBR, all the above aspects are important, but new points could emerge. For example, grazing by protozoa and metazoa could be useful as the external cells are removed, the remaining cells being closer to the surface; this situation favors an improved exchange of chemicals with the liquid phase, thus maintaining the cells in an active state of growth.

Thus, biological and technical parameters should be optimized together for a robust and efficient biofilm activity in MBBR.

The complex physical structure of the biofilms as well as their biological diversity with respect to strains living there, their metabolism and metabolic interactions, based on a huge genetic diversity make them robust ecosystems. This is why different types of solid materials are added to bioreactors in order to provide attachment surfaces for the biofilm development, with positive effects both on the increase of active biomass and higher rates of pollutants degradation [11]. The advantages of biofilms in different type of wastewater plants configurations are further enhanced by the specific configuration of MBBR, where the biofilm concentration per volume unit is higher compared to the classical biological wastewater treatment. There is the need to monitor the quantity of the cells within the biofilms as well as their metabolic activity [5, 12–13].

The research team focused on few usual methods. The quantity of cells within the biofilms is usually measured by crystal violet assay, a method which do not differentiate between alive cells, active cells or dead cells [13]. However, the method has the advantage that is rapid and cheap, being also widespread in the study of different types of biofilms, not only those important in wastewater treatment. One method to differentiate alive cells from dead cells uses two types of fluorescent markers – one which is impermeable to normal, healthy plasma membrane (e.g. propidium homodimer) and the other one which is permeable to both normal and severely injured plasma membranes (related to dead cells or dying cells, ex. Syber green) [13]. Dead cells are labelled by both markers whereas the living cells are labelled only by Syber green. However, these fluorescent markers can be used with ordinary fluorescent microscopes only for incipient states of biofilm development, where the biofilm is composed of only one

layer of cells (mono-stratified). The life span of biofilm is composed mainly of multilayered biofilm, where is the need to use confocal microscopes [13]. When it comes to metabolic activity (and, indirectly, the amount of living cells) the use of resazurine method reach some popularity [14–18].

In figure 3, it is presented the same microscopic field inspected in either green filter (Syber green fluorescence) or red filter (bromide homodimer I fluorescence), containing cells from disintegrated (ultrasonic treatment in the presence of Tween 20) biofilm from the biofilm carriers developed by the research team and presented in figure 2. Total cells (both alive and dead) are labelled by SG whereas the dead cells are labelled only by HD- the difference consists of alive cells.

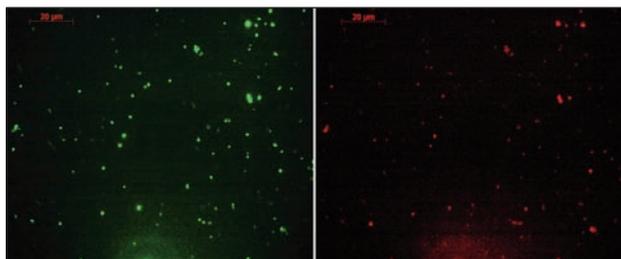


Fig. 3. Fluorescence images of the same microscopic field containing cells chemically detached from the new biomedica: total cell (both alive and dead) were labelled with Syber Green I (green fluorescence) whereas dead cells were labelled with Bromide Homodimer I (red fluorescence)

### NUMERICAL SIMULATIONS FOR MBBR DESIGNING DESIGN AND FUNCTION OF MBBRS

Since a considerable number of parameters is interfering with the treatment efficiency in MBBRs (as shown above), further researches are necessary to design a proper biological treatment stage. For the breathing process of the micro-organisms capable to reduce BOD and  $\text{NH}_4^+\text{-N}$  inside the biological tanks, an aeration system is needed. The needed dissolved

oxygen (DO) quantity is established both from numerical determination. However, with the help of the numerical simulation the DO quantity inside MBBR can be known. The scientific literature states that an important advantage of MBBR utilization instead of activated sludge is the fact that less energy is required [1]. Using numerical simulations, the research team also demonstrated the above statement. MBBRs use less electrical energy than activated sludge process since less air (DO) is needed inside the tanks. The biofilm carriers act as a barrier in front of the air bubbles, thus increasing the retention time.

The research team realized a series of numerical simulation to determine the DO profiles inside a MBBR. The dispersion equation was considered to realize the mathematical model for the determination of the DO profile inside a MBBR [1]:

$$\begin{aligned} \frac{\partial \bar{C}}{\partial t} + \frac{\partial}{\partial x} (\bar{u} \bar{C}) + \frac{\partial}{\partial y} (\bar{v} \bar{C}) + \frac{\partial}{\partial z} (\bar{w} \bar{C}) = \\ = \frac{\partial}{\partial x} \left( \epsilon_x \frac{\partial \bar{C}}{\partial x} \right) + \frac{\partial}{\partial y} \left( \epsilon_y \frac{\partial \bar{C}}{\partial y} \right) + \frac{\partial}{\partial z} \left( \epsilon_z \frac{\partial \bar{C}}{\partial z} \right) + \\ + D_m \left( \frac{\partial^2 \bar{C}}{\partial x^2} + \frac{\partial^2 \bar{C}}{\partial y^2} + \frac{\partial^2 \bar{C}}{\partial z^2} \right) + S(x, y, z, t), \end{aligned} \quad (1)$$

where  $\epsilon_x$ ,  $\epsilon_y$ ,  $\epsilon_z$  are the longitudinal, transversal and vertical dispersion coefficients. Due to the dependence of dispersion coefficients to the flow regime, the simplified form of the above equation was considered:

$$\frac{\partial \bar{C}}{\partial t} + \frac{\partial}{\partial x} (\bar{u} \bar{C}) + \frac{\partial}{\partial y} (\bar{v} \bar{C}) = \frac{\partial}{\partial x} \left( \epsilon_x \frac{\partial \bar{C}}{\partial x} \right) + \frac{\partial}{\partial y} \left( \epsilon_y \frac{\partial \bar{C}}{\partial y} \right) \quad (2)$$

where quantities are averaged over a time period. In figures 4–7 are presented the results obtained from numerical simulations. There were considered several cases: cross section through the bioreactor without and with biofilm carriers in different proportions.

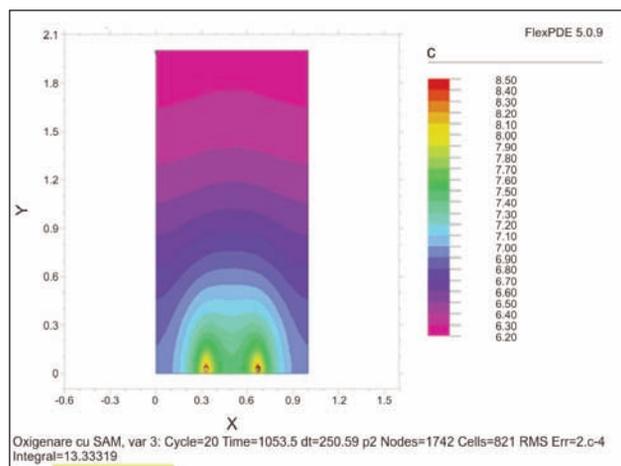


Fig. 4. Dissolved oxygen concentration profiles – cross section

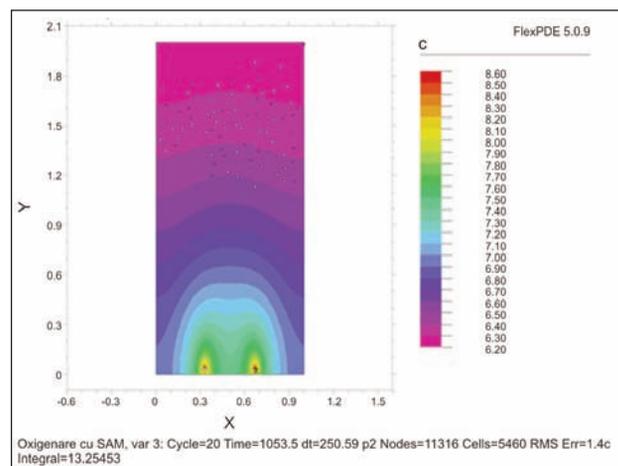


Fig. 5. Dissolved oxygen concentration profiles – cross section

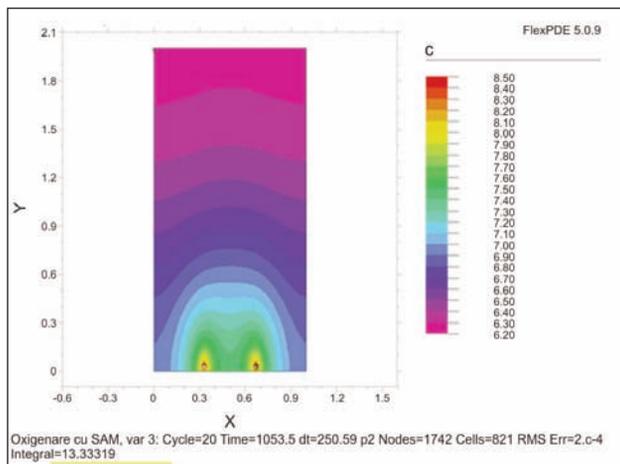


Fig. 6. Dissolved oxygen concentration profiles – cross section

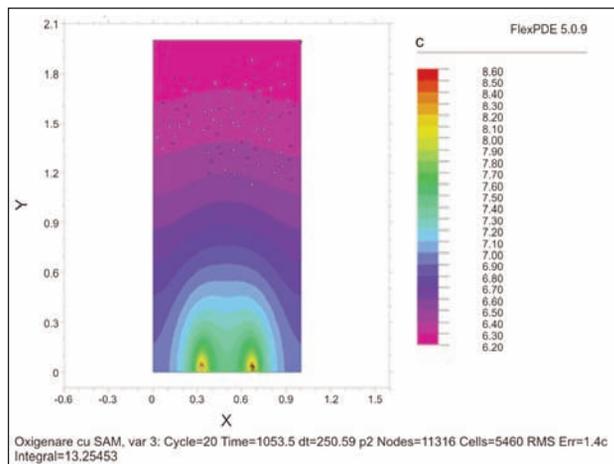


Fig. 7. Dissolved oxygen concentration profiles – cross section

Analysing figures 4–7 it can be easily observed the fact that biofilm carriers help the oxygenation process, resulting air bubbles which rise to the surface meeting in their way the biofilm carriers. Bubbles, due to their interactions with the media, divide and by-pass the biofilm carriers. The contact duration between air and wastewater increases, resulting a better oxygen mass transfer. Also, the increasing quantity of the biofilm carrier inside a MBBR reactor leads to a better mass transfer. In this way, it is necessary to introduce a smaller amount of air which implies a reduced energy consumption.

## RESULTS AND DISCUSSIONS

### Pollutants in textile chemistry wastewater

The textile wastewaters are especially characterized by the presence of dyes. In dyeing, color is applied in the form of solutions and the dye is applied as a thick layer of paste. Also, in the textile dyeing industry, bleaching is an important process and it needs almost 35% of the total water consumed in textiles wet processes. Bleaching it is based on: sodium hypochlorite, hydrogen peroxide and sodium chlorite. The pollutants generated by dyeing and bleaching are important and they lead to a high COD (chemical oxygen demand), TSS (total suspended solids), chlorine etc. High values of COD and BOD<sub>5</sub>, TSS, oil and grease in the effluent causes depletion of DO, which has an adverse effect on the environment. The potential specific pollutants from textile printing and dyeing are presented in table 1 [20].

### Limits for textile industry discharge

The dyeing wastewaters have many complex components with high concentrations of pollutants. According to the high values for BOD and COD, coloration, salt etc. the wastewaters resulting from dyeing cotton with reactive dyes are seriously polluted. The characteristics of discharged wastewaters vary and depend on the type of textile manufactured and the dyes/chemicals used. The effluents contain considerable amounts of agents, including suspended and dissolved solids, BOD, COD, chemicals, odor

Table 1

SPECIFIC POLLUTANTS FROM TEXTILE AND DYEING PROCESSING OPERATIONS [20]	
Process	Compounds
Desizing	Sizes, ammonia, starch, enzymes, waxes
Bleaching	High pH, AOX, sodium silicate or organic stabiliser, H <sub>2</sub> O <sub>2</sub>
Scouring	NaOH, disinfectants residues, surfactants, waxes, fats, pectin, anti-static agents, oils, spent solvents, soaps, enzymes
Mercerizing	High pH, NaOH
Printing	Solvents, urea, metals, colour
Dyeing	Metals, colour, salts, organic processing assistants, surfactants, sulphide, formaldehyde, high/low pH,
Finishing	Waxes, resins, acetate, chlorinated compounds, spent solvents, stearate, softeners

and color causing damage to the human health and environment. Typical characteristics of textile effluent are shown in table 2 [21].

As the wastewater is harmful to the environment and people, there are strict requirements for discharged influents. However, due to the difference in the raw

Table 2

TYPICAL CHARACTERISTICS OF TEXTILE EFFLUENTS [21]	
Parameter	Value
pH	6 – 10
Total dissolved solids [mg/l]	8.000 – 12.000
BOD [mg/l]	80 – 6.000
COD [mg/l]	150 – 12000
Total suspended solids [mg/l]	15 – 8.000
Chlorine [mg/l]	1.000 – 6.000
SO <sub>4</sub> [mg/l]	600 – 1.000
Total Kjeldahl Nitrogen [mg/l]	70 – 80

THE LIMITS OF DISCHARGED CONCENTRATION IN DIFFERENT COUNTRIES [22–23]						
Parameter	GERMANY	CHINA			ROMANIA	
		The limits of discharged concentration	The limits of discharged concentration for new factory	The special limits of discharged concentration	NTPA 002	NTPA 001
COD [mg/l]	160.0	100.0	80.0	60.0	500.0	70.0 (125.0)
BOD [mg/l]	25.0	25.0	20.0	15.0	300.0	20.0 (25.0)
TP [mg/l]	2.0	1.0	0.5.0	0.5	5.0	1.0 (2.0)
TN [mg/l]	20.0	20.0	15.0	12.0	30.0	10.0 (15.0)
NH <sub>3</sub> -H [mg/l]	10.0	15.0	12.0	10.0	NA	NA
TSS [mg/l]	NA	70.0	60.0	20.0	350.0	35.0 (60.0)

materials, products, dyes, technology and equipment, the standards of the wastewater emission have many items. The standards of printing and dyeing vary from a country to another. Through access to the relevant information, the textile industry standards for water pollutants in Germany, China and Romania are presented. It is developed by the national environmental protection department according to the local conditions and environmental protection. In some countries such as China, the limits for the discharged pollutants are different depending on the factory situation. In other countries the limits vary depending on the region. Table 3 presents the maximum discharge limits for Germany, China and Romania [22–23].

### Recommended placement of MBBRs in textile wastewater treatment plants

As the pollutant number is high several treatment stages can be combined for an efficient wastewater treatment. The main treatment stages for the textile wastewater treatment are: physicochemical treatment (equalization and homogenization; floatation; coagulation flocculation sedimentation; chemical oxidation; adsorption; membrane separation process), biological wastewater treatment (activated sludge process; oxidation ditch process; sequencing batch reactor activated sludge process; MBBR; rotating biological contactor), biochemical and physicochemical combination processes and advanced treatment stages (photochemical oxidation; electrochemical oxidation; ultrasonic technology; high energy physical process).

Based on the influent characteristics and all the available treatment process, a general diagram for the textile wastewater treatment (figure 8) is proposed by the authors. The MBBR technology is recommended to be used due to its advantages related to other biological treatment.

### CONCLUSIONS

The textile industry, apart from being an important contributor to the economy of numerous countries, is also a major source of various liquid, solid and gaseous wastes. This kind of industrial activity has a

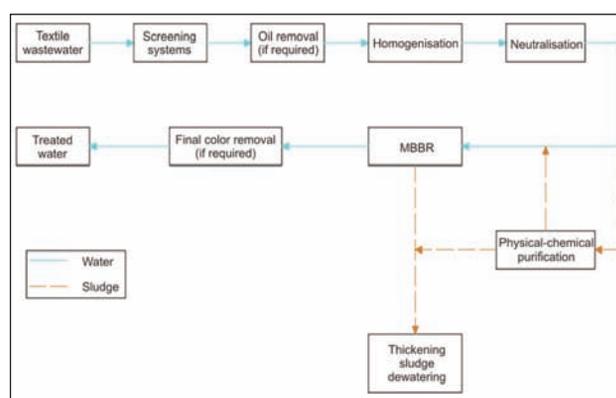


Fig. 8. The simplified diagram of wastewater treatment plant in the textile industry

negative impact on the environment, both in terms of pollutant discharge as well as of water and energy consumption.

In this context researchers are searching for new cost-effective treatment technologies. The authors recommend the MBBR utilization in textile wastewater treatment processes because it meets the requirements of an efficient and cost-effective technology. So far, wastewater treatment plants all over the world are using MBBR treatment stages but the researchers are actual and constantly are seeking ways to increase the efficiency. The researches more and more use new mathematical instruments for modelling and simulation. MBBR has proved the efficiency of reducing especially BOD and ammonia, in small tank volumes.

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# Insulation materials for buildings – a successful research & development collaboration for the Romanian wool fibres manufacturing

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## REZUMAT – ABSTRACT

### Materiale izolatoare pentru construcții – o colaborare de succes în domeniul cercetării și dezvoltării pentru producerea fibrelor de lână din România

În contextul dezvoltării durabile, sectorul textil constituie un pilon puternic al industriei românești, în măsură să contribuie la valorificarea materiilor prime naturale autohtone. Lucrarea prezintă aspecte economice, în baza rezultatelor obținute în cadrul unui proiect finanțat prin programul Sectorial, al Ministerului Cercetării și Inovării, pentru valorificarea fibrelor de lână, românești, în scopul obținerii de materiale termo și fono-izolatoare, pentru construcții. Se evidențiază rezultatele colaborării, din cadrul proiectului, a trei actori importanți, din activitatea de cercetare și mediul economic: Institutul Național de Cercetare-Dezvoltare pentru Textile și Pielărie – INCDTP București – unicul institut de cercetare din domeniul textile-pielărie din țară, compania S.C. MINET S.A. Râmnicu Vâlcea – companie reprezentativă pentru industria neșesutelor din România și Institutul de Cercetare-Dezvoltare pentru Creșterea Ovinelor și Caprinelor – Palas, Constanța, parteneri în cadrul consorțiului coordonat de Institutul Național de Cercetare-Dezvoltare în Construcții, Urbanism și Dezvoltare Teritorială Durabilă – URBAN INCERC București.

Cuvinte-cheie: fibre de lână românești, inovare, eficiență, inițiative antreprenoriale, textile tehnice pentru construcții

### Insulation materials for buildings – a successful research & development collaboration for the Romanian wool fibres manufacturing

Having in view the sustainable development context, the textile sector represents a strong pillar of the Romanian manufacturing industry, which is able to contribute to the valorization of natural indigenous raw materials. The paper presents economic aspects in the base of the results obtained through developing/ implementing a research project financed by the National Sectorial Program, coordinated by the Romanian Ministry of Research and Innovation, aiming to establish strategic solutions for capitalization of Romanian coarse wool fibers. There are emphasized the project's results obtained by the collaboration of three important actors from research activity and economic environment: the National Research and Development Institute for Textiles and Leather – INCDTP Bucharest, the only R&D Institute in Romania, SC MINET SA Company, Râmnicu Vâlcea county – a representative manufacturing company for nonwoven materials and the Research Institute for Sheep and Goats Breeding, Palas, Constanța county, partners in the consortium coordinated by the National Research and Development Institute in Constructions, Urban Planning and Sustainable Spatial Development URBAN-INCERC Bucharest.

Keywords: Romanian wool fibres, innovation, efficiency, business entrepreneurial initiatives, technical textiles for buildings

## INTRODUCTION

Romania's rural development strategy for the coming years is in line with the EU's reform and development context with the Europe 2020 strategy [1]. Following the objectives of the Europe 2020 strategy for a smart, sustainable and inclusive economy, the strategy sets ambitious targets for Member States in the areas of education, innovation, energy/environment, employment and social inclusion and improving competitiveness in general [2].

The National Rural Development Program (NRDP) 2014-2020 [3] contributes to **smart growth** by supporting forms of cooperation between research institutions and farmers and other actors in the rural economy, but also by supporting training, skill acquisition and dissemination of information. The NRDP

also envisages a **sustained growth** that focuses on lowering carbon emissions and supporting environment-friendly farming practices. Last but not least, support for investment in the infrastructure and the rural economy leads to poverty reduction and job creation in rural areas, thus contributing to an **inclusive growth**.

All these objectives will be possible to materialize only under the conditions of efficient utilization of indigenous raw materials, among which wool fibers, a valuable source both for the textile industry and for related sectors, as is the field of ecological constructions.

The wool processing sector in Romania experienced a regression in terms of fiber quality after 1989, caused by a combination of factors:

– uncontrolled crossbreeding,

- low area,
- low quality pastures,
- lack of support for sheep breeders.

As a result, the spin ability limit of the Romanian wool has decreased, as well as the possibility of using it in the textile industry, in the conditions of increasing demand for fine woven and knitted fabrics and knitwear, leading to the closure of many traditional textile companies and the use of imported wool. Under these circumstances, the use of the Romanian wool for related fields such as construction, cosmetics, pharmaceuticals is much more important, as an efficient and viable alternative for recovery and an alternative for revitalization of several economic sectors.

Analysis of the construction materials market shows an increased interest in the use of wool as a thermal insulation material, leading to an economically significant impact, as the construction sector is a major energy consumer within the European Union, accounting for 40% of the total energy consumption and 36% of greenhouse gas emissions [4].

In this context, the Ministry of Research and Innovation in Romania, together with the Ministry of Agriculture, had the initiative to launch a competition for a project entitled “**Research on the Development of Capacity for Transfer and Marketing of Research Results on Integrated Exploitation of Natural Wool Resources. Applicability of Eco-Innovative Products Based on Sheep Wool in the Field of Constructions**”, in September 2017.

A multidisciplinary consortium consisting of representative research centers of industries such as textile – INCDTP Bucharest, mechanics – ICTCM Bucharest, chemical-pharmaceutical research – ICCF Bucharest, sheep breeding – ICPCOC Palas Constanta, SC MINET SA RâmnicuVâlcea, SC IRECSO SA under the coordination of INCD URBAN-INCERC Bucharest ensured the critical mass of specialists in order to achieve the objectives of this project, ongoing in 2018.

The paper presents aspects regarding the textile valorization of the thick Romanian wool varieties in order to produce materials with the role of insulation and sealing, in the field of constructions.

## EXPERIMENTAL WORK

At present, the proportion of “Țurcană” breed sheep is over 70% of total heads, and of the wool volume after shearing, respectively.

The matrix of technological experimentation took into account the following experimental criteria:

- exploiting the technological equipment of the industrial partner SC MINET SA RâmnicuVâlcea; thus two distinct technologies were used in processing: i) consolidation the fibrous material by athermochemical process and ii) mechanical consolidation;
- the use of Țurcana wools in particular; thus the fiber composition variants used were: a) 85% Țurcana wool + 15% heat-activated adhesive fibers, b) 70% Țurcana wool + 30% heat-activated

adhesive fibers – processed, using technology i), and c) 100% Țigaie wool – processed using technology ii), respectively;

- adaptation of technological parameters and processing stages to the characteristics of the blended fibrous material;
- the coverage, according to the adjustment parameters, of the entire range of non-woven structures, possible to be obtained, on the used processing technologies;
- design and fabrication of nonwoven fabrics with different massdensity and thickness, covering a wide range, possible to be used by the constructors in different ways and locations of a building: floor, roof, walls;
- providing improved properties to the materials by applying functional treatments specific to insect/moth protection, flame maintenance and propagation.

The highlighted innovative aspects are the subject of patent application A/10034/2018 of July 30, 2018, entitled “Unconventional textile fabric based on wool, from Romanian breeds, for the isolation of constructions and the process of obtaining thereof”, authored by SC MINET SA, INCDTP, INCD URBAN-INCERC and ICPCOC Palas.

## ASPECTS REGARDING THE ECONOMIC EFFICIENCY

The use of thick and semi-thick wools for the production of non-woven textile materials for the sound and thermal insulation of buildings is a high-potential entrepreneurial area, in the context of rising electricity, thermal and energy prices, and in the context of national and European trends in green building.

These are the arguments for which we analyzed the economic efficiency of capitalizing Romanian wools by simulating a family entrepreneurial business, that is, a minimal investment, possible to be achieved at the level of young Romanian entrepreneurship.

The working hypotheses (input data) we started in this simulation are:

- we are located in a mountainous rural area, where sheep breeding is a basic occupation of most peasant farms;
- the sheep wool is sheared once a year and the raw (greasy) wool obtained is collected and sent to a collection center (already existing in the country) to be forwarded to the wool laundry;
- the wools will be washed either at the wool laundry at SC STOFE SA Buhuși, or in cooperation with the laundries in Turkey;

*\*Observation: INCDTP highlights that an investment to set up a wool laundry is extremely costly, and involves the existence of waste water treatment plants, since it is a water impurifier; considering environmental regulations, we do not consider the possibility of washing small quantities of wool directly into rivers;*

- entrepreneurial initiative starts basically from the processing of washed wools;

– we also believe that the entrepreneur already owns the space and construction to open this micro-enterprise for the processing of washed wools and the production of non-woven textiles for thermal and sound insulation of buildings.

Taking into account these working hypotheses, a simulation of a business plan, based on the technical data obtained in the technological experiments carried out within the project, was conducted by SC MINET SA and INCDTP.

Based on cost categories, the situation is the following:

- raw material costs (lei/kg of washed wool); costs of chemical auxiliaries used in processing, anti-insect treatment products, fire retardance treatment products (lei/kg);
- labor costs: (lei/m<sup>2</sup> of non-woven fabric);
- utilities costs (overheads) (lei/m<sup>2</sup> of non-woven fabric);
- third-party processing: testing raw material and finished products, product research and development, marketing costs, as it is a starting entrepreneurial initiative.

The production prices ranged between 19.27–37.34 lei/m<sup>2</sup>, varying directly in proportion to the specific consumption values g/m<sup>2</sup> and depending on the type of washing of the raw wools used (washing it abroad is more expensive).

It should be stressed that not only the low price aspects must be considered, but first of all the following should be taken into account:

- the technology allows the recovery of Romanian thick and semi-thick wool fibers, a raw material which does not have suitable spin ability characteristics for processing high fineness yarns and which

is currently either burned or exported as raw material or collected in peasant farm conditions;

- the technology may be applied to companies producing non-woven textiles with technical use; currently only three such companies are active in the country, therefore entrepreneurial initiatives in this field are necessary and timely;
- newly created companies will contribute to the use of native resources of thick and semi-thick wools, implicitly in creating new jobs and attracting young people, especially in rural areas, where a depopulation phenomenon is currently occurring;
- the new products will contribute to the development and implementation of the concept of green houses with low construction and operating costs.

## CONCLUSIONS

Under the conditions of increasing Romanian thick and semi-thick wools, their exploitation by producing nonwoven fabrics for efficient constructions, both in terms of construction costs and energy maintenance of buildings is a good opportunity.

The results will be disseminated in debates with representatives of sheep breeders, with the involvement of the Regional Development Agencies in the country and actors in the construction sector, in order to stimulate the regional entrepreneurial initiatives, both in non-woven textiles, and in the green building fields.

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## REZUMAT – ABSTRACT

### Direcții viitoare în repararea țesuturilor folosind biomateriale

Defectele complexe ale țesuturilor moi sunt adesea provocatoare pentru a aborda tehnicile reconstructive chirurgicale convenționale. Deși repararea țesuturilor autologere prezintă încă standardul de aur în chirurgia reconstructivă, există situații speciale când autogrefele nu sunt disponibile. Alogrefele umane rămân o soluție alternativă, dar cu dezavantaje considerabile privind disponibilitatea, reacțiile imunologice și riscul transmiterii bolilor infecțioase. Produsele sintetice utilizate în reconstrucțiile chirurgicale sunt asociate cu rezultate slabe pe termen lung.

Ingenieria tisulară s-a dezvoltat pe baza acestor limitări reconstructive. Recent, matricile de țesuturi (scaffold) au fost introduse ca elemente importante în strategiile chirurgicale de reconstrucție a țesuturilor. A fost utilizat un panou mare de matrice extracelulară, cultivat cu diferite populații de celule, iar rezultate promițătoare obținute au fost raportate în repararea complexă a pierderii tisulare, incluzând oasele, mușchii, nervii, vasele sanguine și defectele cutanate.

Cercetările viitoare sunt obligatorii pentru standardizarea structurilor de bioinginerie tisulară, pentru a obține cel mai bun rezultat în ceea ce privește restaurarea volumului, recuperarea funcției, integrarea vasculară și stabilitatea pe termen lung a reconstrucției.

Dorim să identificăm domeniul de cercetare care poate fi aplicat cu succes în țara noastră, pe baza unei colaborări interdisciplinare între instituțiile medicale, laboratoarele de biologie moleculară și institutele de cercetare tehnică. Considerăm foarte utilă promovarea unei strategii pentru repararea țesuturilor moi și imunomodulare folosind progenitori mezenchimali din diferite surse (măduvă osoasă, țesut adipos, sânge din cordonul ombilical) în sămânțate pe matrici de țesut solide. Această abordare va spori parteneriatul multiinstituțional, permițând dezvoltarea de noi strategii de reconstrucție pentru pacienții cu defecte de țesut, mai accesibile pentru instituțiile medicale locale, la un cost accesibil și în timp util pentru recâștigarea calității vieții.

Cuvinte-cheie: repararea țesuturilor, biomateriale, ingineria tisulară, celulele stem mezenchimale

### Future directions in tissue repair using biomaterials

Complex soft tissue defects are often challenging to approach using conventional surgical reconstructive techniques. Although autologous tissue repair is still the gold standard in reconstructive surgery, there are particular situations when autografts are not available. Human allografts remains an alternative solution, but with considerable drawbacks regarding availability, immunologic reactions and risk of infectious diseases transmission. Synthetic products used in surgical reconstructions are associated with poor long-term outcomes.

Tissue engineering developed based on these reconstructive limitations. Recently, biological scaffolds have been introduced as important players in surgical strategies of tissue reconstruction. A large panel of extracellular matrices cultured with different cell populations has been used, and promising results were reported in complex tissue loss repair including bone, muscles, nerves, blood vessels and skin defects.

Future research is mandatory to standardize the bioengineered structures, in order to get the best outcome regarding volume restoration, function regaining, vascular integration and long term stability of the reconstruction.

We desire to identify the research area which can be successfully applied in our country, based on an interdisciplinary collaboration between medical institutions, molecular biology laboratories and technical research institutes. We consider very useful to promote a strategy for soft tissue repair and immunomodulation using mesenchymal progenitors from different sources (bone marrow, adipose tissue, umbilical cord blood) seeded on solid scaffolds. This approach will increase the multi-institutional partnership, permitting the development of new reconstructive strategies for patients with tissue defects, more accessible for local medical institutions, with an affordable cost and in appropriate timing for regain the quality of life.

Key words: tissue repair, biomaterials, tissue engineering, mesenchymal stem cells

## INTRODUCTION

A biomaterial is represented by a substance or a combination of biologically inert substances that are used for implantation or integration in a living organism in order to improve or replace specific tissue or organ functions [1].

The use of biomaterials and the development of tissue engineering developed due to impossibility of substituting the tissues or organs affected by various pathologies. Human organ or tissue transplantation

remains a standard approach, but there are a series of limitation regarding availability of human donors, immunologic aspects (risk of rejection with subsequent allograft functional impairment or even allograft loss and severe complications associated with long term immunosuppressant therapy) and risk of infectious disease transmission [2].

A specific tissue defect may be approach in different manners, the gold standard being autologous reconstruction due to favorable long-term results in absence of any immunologic response [3].

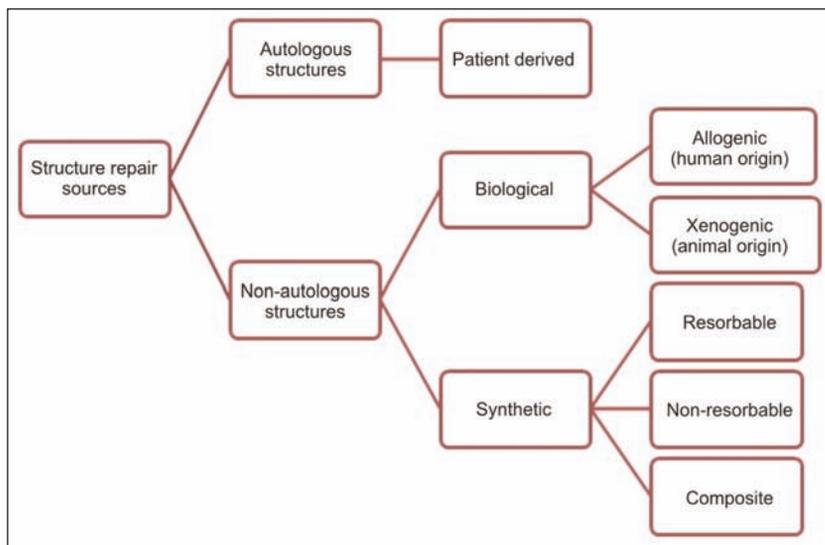


Fig. 1. Reconstructive options

There are situations when patient-derived tissues are not available, due to lack of donor areas, donor site morbidity, or different conditions of the patient that preclude the autologous reconstruction. In these situations other reconstructive options must be chosen, different available possibilities being synthesized in figure 1 [4].

Any structure that is used for tissue reconstruction or replacement has to be very well long-term tolerated by the living organism. The most important requirement for a biomaterial is biocompatibility, ensuring non-cytotoxic effects with favorable properties promoting biofunctionality [5].

Tissue engineering associates elements of biomaterials and cell transplantation in order to substitute affected tissue and also promote structural and functional regeneration. To date, significant progress was noticed in tissue engineering field, resulting in promising therapeutic strategies for organ dysfunction and various tissue loss [6]. Future research is necessary to standardize the bioengineered structures, in order to get the best results regarding volume restoration, function regaining, vascular integration and long term stability of the reconstruction. Three-dimensional designed structures are intensively studied due to their potential to restore more accurate the affected tissue for optimal function [7].

## RECONSTRUCTIVE STRATEGIES USING TISSUE ENGINEERING

In the past, biomaterials were used only for temporary tissue replacing after surgical removal or necrosis. The science of biomaterials evolved and currently, scaffolds are designed similar to natural extracellular matrix in order to support biologic functions, ensuring cellular adhesion, further differentiation and proliferation. Biological scaffolds play an important role for tissue reconstruction [8]. Unlike synthetic materials, biomaterials integrates in the host tissue, release a series of cytokines with anti-inflammatory role, improve healing process and diminish bacterial load [7].

Strict requirements are applied when design a scaffold for in vivo use and it is important to adapt each biomaterial for specific reconstruction, in order to obtain best results regarding durable structural and functional restoration [9].

Key players in regenerative medicine and tissue engineering are scaffolds, cells and cytokines, the essential components of a bio-engineered structure [8].

A large panel of extracellular matrices cultured with different cell populations were used with promising results in complex structural and functional repair including defects of skin, bone, muscles, nerves and blood vessels.

Dermal substitutes represent biomaterials that replace the structure

and functions of the dermal layer of the skin. Both acellular and cellular dermal matrices were introduced in clinical practice, patients with various skin defects benefiting from this kind of therapies. Application of a dermal substitute covered by an autologous skin graft ensures the wound coverage and stimulates the healing process in burns, epidermolysis bullosa, piodermagangrenosum, deep chronic wounds, skin ulcers (cause by diabetes, venous insufficiency, neuropathies, pressure ulcers, autoimmune diseases), pathologic scars surgical replacement [7, 10–13]. Acellular dermal matrices have also other indications, in more extensive reconstructions of craniofacial area, cervical region, thorax, breast, abdominal wall including hernia repair or reinforcement of the muscle flaps [7].

Also composite structures resembling human skin were created, consisting of a superficial stratified layer of human keratinocytes and a deep dermal component represented by a collagen scaffold (for example of bovine collagen gel) and cultured human fibroblasts, with good results for venous and diabetic foot ulcers, epidermolysis bullosa, burns and coverage for donor sites of split-thickness graft [14, 15].

Tissue engineering is also applied for bone regeneration. Most often large bone defects are replaced by bone grafts, autografts being the golden standard. A wide panel of tissue-engineered constructs was introduced for bone defects, the ideal characteristic of this structures being the resemblance with autografts in terms of composition and biologic properties. Strategies as gene therapy, combinations of scaffolds, healing promoting factors, stem cells and three-dimensional printing could be amazing new tools in treating complex bone defects [16].

Cartilage repair is also a difficult procedure. The main research direction consists in use of biological constructs of cartilage biodegradable scaffolds seeded with adequate cells and growth factors to ensure cellular signaling and interaction [17].

Engineering of the tendons and muscles is also extremely challenging, hard to translate in clinical

practice, due to the need for restoring both biological structure and mechanical properties with the new-developed structures [18, 19]. Problems still remain in restoration of complete function of an injured tendon, a standardized combination of biological factors is not yet developed and also there is no optimal method to apply the bioengineered construct in the affected area [18].

Another intensively studied field is the development of tissue-engineered nerve grafts for bridging of nerve gaps following complex peripheral nerve injuries for best regenerative performance [20]. Biomaterials that address vascular reconstruction need to meet complex criteria including the same mechanical properties as recipient vasculature, with stable result, promoting cell growth, production of extracellular matrix and inhibition of thrombus formation [21].

Complex biomedical structures can be adapted for each patient using 3D printing technology. 3D printing shows promising results for complex tissue regeneration including bone, muscles, cartilage vessels and nerves. An indication of this technology is the restoration of cranio-maxillo-facial defects. Also 3D printing may help in organ regeneration, even in challenging micro-architecture like in liver or lymphoid organs [22].

Biomaterial scientists have learned how to mimic the biological systems on different levels. In this direction, nanoscience and nanotechnology will substantially ensure the advance in the field of tissue engineering [23].

Nanotechnologies clearly have influenced the tissue engineering by developing nanomaterials such as carbon nanotubes, nanowires and other inorganic materials. Implanting intelligent nanoscale biosensors within scaffolds will bring more knowledge regarding engineered tissues. Also smart controllable nanorobots could circulate inside the body and repair damaged structures [24].

Another promising direction for the use of biomaterials and nanomedicine is the introduction of smart drug delivery systems with the final goal of improve the therapeutic effects and decrease the side-effects of the substances, resulting in more safer and efficient pharmaceutical agents. Development of performant drug delivery systems is crucial in oncology to ensure a higher tumoricide effect [25, 26].

## **FUTURE DIRECTIONS FOR THE DEVELOPMENT OF CLINICAL APPLICATIONS OF BIOMATERIALS**

We desire to identify the research area which can be successfully applied in our geographic region, based on an interdisciplinary collaboration between medical institutions, molecular biology laboratories and technical research institutes. This approach is based on economic considerations, knowing the limits in implement emerging technologies in developing countries, but having in mind the real benefits of using tissue-engineered systems for tissue regeneration.

We consider very useful to promote a strategy for soft tissue repair and immunomodulation using mesenchymal progenitors from different sources (bone marrow,

adipose tissue, umbilical cord blood) seeded on solid scaffolds.

Human mesenchymal stem cells were isolated for the first time from bone marrow. Further studies demonstrated a broad spectrum of mesenchymal stem cells origin, including adipose tissue, peripheral blood, amniotic membrane, umbilical cord blood, Wharton's jelly. Mesenchymal stem cells express a specific set of markers on their surfaces: CD73+, CD105+, CD90+ and are negative for CD34, CD14, CD45 and HLA-DR [27, 28].

It was demonstrated that mesenchymal stem cells, through cytokine secretion and specific receptors, have an important immunoregulatory role, make them particular from other undifferentiated cells and encourage their utilization as part of future cell replacement treatment and also in transplantation field to promote allograft acceptance and transplant tolerance [27].

Research using embryonic stem cells is controversial due to ethical aspects [29]. Fetal/perinatal originated mesenchymal stem cells (derived from placenta, umbilical cord blood, Wharton jelly, amniotic membranes) are an attractive option for regenerative medicine through their higher proliferative capacity, better differentiation and plasticity, immunomodulatory properties with some genetic features similar of embryonic cells, without any risk of tumorigenicity [28]. Another attractive source of cells for regenerative purpose, recently emerged is human-induced pluripotent stem cells (hiPSCs), with very good properties regarding cell proliferation, cytokine secretion, immunomodulation and ability to modulate the microenvironment through exosomes and secretion of paracrine factors. The autologous iPSC-derived mesenchymal stromal cells could become in the future an unlimited source of regenerative cells, but sustained research is mandatory in this direction [30].

Adult mesenchymal stem cells originate mainly from bone marrow and adipose tissue, both sources ensuring multipotent stem cells, with the ability to differentiate in a variety of tissue lineages including: cartilage, bone, tendon, adipose tissue, muscle and nervous tissue [31].

The use of bone marrow as adult source of mesenchymal stem cells is the most studied in various research studies. Recently, adipose tissue has been more carefully studied as a potential optimal source of mesenchymal progenitors for use in tissue regeneration.

Since their discovery around 15 years ago, adipose-derived stem cells proved their regenerative potential in experimental studies, allowing a safe translation to clinical practice [32–33].

Those cells are obtained from adipose tissue extracted through liposuction or surgically excised in block that is subsequently processed by filtration and centrifugation and enzymatic digested using collagenase [34–35]. Further, adipose progenitor cells are purified and cultured in specific condition [32]. Adipose derived stem cells can be cultured in the presence of various growth factors on porous scaffolds to engineered 3D constructs and ensure an adequate micro-medium

for regenerative purposes. Very good results can be obtained with novel collagen-sericin 3D porous scaffolds (with a spongy structure containing 60% collagen and 40% sericin) cultured with a dipose precursors, as Dinescu and collaborators described in several studies [36–37].

Besides ex-vivo processing of adipose tissue, clinical use of nanofat grafting technique (consisting of fat injection with very thin needles of even 27 gauges) is also promising for its benefic properties demonstrated in skin rejuvenation. Analysis of nanofat samples revealed a large amount of mesenchymal stem cells in their composition [38].

Based on discussed data, we suggest a therapeutic strategy (figure 2) involving adipose tissue progenitor cells, suitable for tissue defects reconstruction or for immunomodulatory properties in various allograft transplantation.

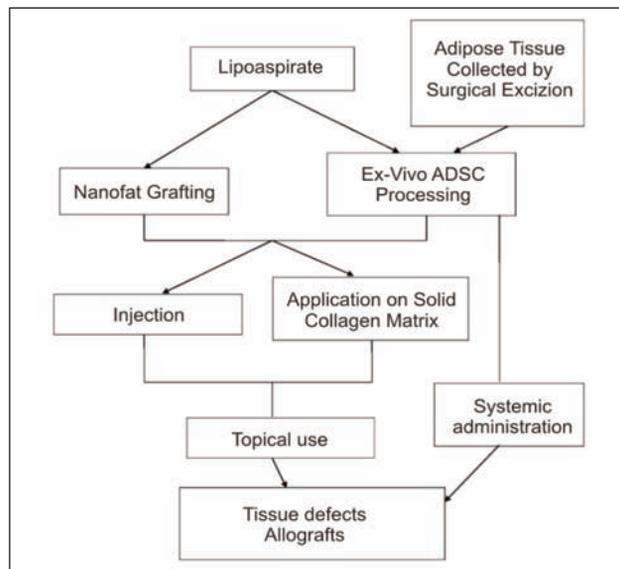


Fig. 2. Therapeutic strategy

This approach will increase the multi-institutional partnership, permitting the development of new reconstructive strategies for patients with tissue defects or need for immune modulation, more accessible for local medical institutions, with an affordable cost and in appropriate timing for regain the quality of life.

Further studies are needed for improve clinical protocols involving bioengineered structures. Stem cells and the creation of microenvironments provide morphogenesis and physical properties. Having, within the patient, a pro-regenerative environment can improve the survival of the engineered graft. Adequate vascularization of any tissue construct is mandatory for its survival and long-term efficacy. In structures with thick scaffolds strategies for stimulate vascularization like angiogenic induction or inclusion of endothelial progenitor cells can be used.

Developing large engineered tissue require a vascularized pedicle to be anastomosed with host vessels. Tridimensional complex scaffolds need a bioreactor model, involving a dynamic system for cell culture [39–40].

## CONCLUSIONS

Important developing in life sciences including stem cell biology, genomics and proteomics contributed to an exponential growth of tissue engineering. Organ and tissue loss led to emerging of therapies that can regenerate tissues and decrease the need of transplantations at least in theory. The desiderate with tissue engineering is to recreate natural healing processes for best structural and functional outcomes. Currently, limitations exist in attempt to develop standardized clinical protocols and also economic considerations. Better-designed bioengineered constructs with affordable technologies may expand the indications in complex tissue defects reconstruction, with minimum morbidity and best results regarding long-term recovery.

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