

ANNALS OF THE UNIVERSITY OF ORADEA

FASCICLE OF TEXTILES, LEATHERWORK

VOLUME 22

No. 1



2021

ISSN 1843 - 813X



ANNALS OF THE UNIVERSITY OF ORADEA

FASCICLE OF TEXTILES, LEATHERWORK

VOLUME 22 No. 1



2021

EDITOR IN CHIEF Liliana INDRIE, University of Oradea, Romania

BOARD OF EDITORS

Sabina GHERGHEL- University of Oradea, Romania Simona TRIPA- University of Oradea, Romania

SCIENTIFIC COMMITTEE:

Muhammad-Ali AFZAL Ionel BARBU Jocelyn BELLEMARE **Maria Angeles BONET Eva BOU-BELDA Raluca BRAD** Nuno BELINO **Galina BASHKOVA Diana COMAN Dana Corina DESELNICU** Pablo DIAZ GARCIA Daniela FARÎMĂ **Carmen GHIŢULEASA** Manfredo GUILIZZONI **Aminoddin HAJI** Josphat IGADWA MWASIAGI Hüseyin Ata KARAVANA Zlatina KAZLACHEVA

Reyhan KESKİN Musa KILIC Steve McNEIL **Elena BADEA** Ahmad MOHD ROZI Mehmet Mete MUTLU Sabina OLARU **Daniel PALET Alexandru POPA** Sengodagounder RAJAMANI Sabrina RAMSAMY-IRANAH Snežana UROŠEVIĆ **Emilia VISILEANU** Hasan Bilgehan YAVUZ Emilija ZDRAVENA Gökhan ZENGİN **Zlatin ZLATEV** Muge YUKSELOGLU

Contact and Editors' Address: Liliana INDRIE UNIVERSITATEA DIN ORADEA, FACULTATEA DE INGINERIE ENERGETICĂ ȘI MANAGEMENT INDUSTRIAL DEPARTAMENTUL: TEXTILE- PIELĂRIE ȘI MANAGEMENT INDUSTRIAL Str. B.St.Delavrancea nr. 4, Oradea, 410058, Romania, Tel.: 00-40-259-408448 E-mail : <u>lindrie@uoradea.ro</u>

Published by

Editura Universității din Oradea Universitatea din Oradea, Str. Universității Nr. 1, 410087, Oradea, Bihor, Romania P- ISSN 1843 – 813X E - ISSN 2457-4880 CD- ISSN 2068 – 1070

Indexed in:

EBSCO-Textile Technology Complete Index Copernicus (ICV 2018: 97,58) Directory of Open Access Journals (DOAJ) Ulrich's Update - Periodicals Directory



STUDY OF RESISTANCE TO SHARP OBJECT PENETRATION AND SPREAD OF THE RESULTING RUPTURE IN WOVEN FABRICS

AMIRSHIRZAD Forough¹, MOUSAZADEGAN Fatemeh², EZAZSHAHABI Nazanin³

^{1, 2,3} Amirkabir University of Technology, Textile Engineering Faculty, 424 Hafez Ave, 15875-4413, Tehran, Iran

E-Mail: amirshirzad@aut.ac.ir, f_mousazadegan@aut.ac.ir, ezazshahabi@aut.ac.ir

Corresponding author: Amirshirzad, Forough, E-mail: amirshirzad@aut.ac.ir

Abstract: Textiles designed to protect the wearer against damages caused by sharp objects have various applications in the workplace and sports. In this study, the resistance of woven fabrics against sharp object penetration was assessed. In this regard, three different fabric samples, including the Twill 2/2, Satin 1/7 weft, and Hopsack 4/4 weave patterns were prepared. Force and energy of penetration by a sharp object were measured in mentioned fabric structures. It was observed that weft density and yarn interlacement are prominent factors that determine the woven fabric resistance against sharp object penetration. Higher weft density leads to greater penetration force and energy since a higher number of yarns resist the sharp object penetration. Moreover, the weave pattern with more firmness restricts the yarn displacement in the fabric structure and consequently provides greater penetration resistance. In this regard, the Hopsack weave pattern, which has higher weft density than Twill fabric, and shorter yarn floats compared to Satin fabric, showed the best resistance among tested samples. In addition to the penetration test, the cut spread test was also performed to evaluate the fabric resistance against the expansion of the cut created in the fabric by sharp object penetration. According to the results, in the cut spread test, the Hopsack fabric provided the greatest resistance. The force and energy of spreading the cut region are much more than creating the cut in the fabric structure since the sharp object should cut the bunched yarn instead of the single yarn.

Key words: Woven Fabric, Weave Pattern, Weft Density, Penetration Force, Sharp Object

1. INTRODUCTION

Resistance to penetration is one of the fundamental properties of fabrics utilized for protection against various types of mechanical impact [1]. Several 2D and 3D textile structures can be used in designing protective textiles. Among them, two-dimensional woven fabric is the most commonly used material for various mechanical impact threats [2]. A study by Nolan et al. [3] revealed that wearing multiple layers of clothing increases the penetration force relative to each clothing layer separately. However, no direct relationship was observed between the penetration force in multi-layer and single-layer clothing. Amirshirzad et al. [4] analyzed the penetration resistance of a three-layer textile structure and its constituted layers. Based on their observation, the penetration force diagram by sharp object displacement of the studied three-layer structure was divided into two regions representing resistance of top layer and both middle and bottom layers. In another study, Amirshirzad et al. [5], studied bending and resistance to sharp object penetration in metal-reinforced fabrics. According to their results, in addition to providing greater penetration



number of metal threads, instead of using coarser metal threads. Cooper et al. investigated the damage morphology caused by kitchen knives, taking into account the effect of fabric extension. Their results showed that the length of the injury is affected by the assailant gender, the fabric structure, and knife properties. In contrast, the fabric extension (0%, 10%) has no significant effect on the injury's length [6].

2.1 Materials

Woven fabrics in three weave structures, including weft-backed Twill $\frac{2}{2}$ Z, Satin $\frac{1}{7}$ weft, and Hopsack $\frac{4}{4}$, were used to study woven fabric's resistance to sharp object penetration and spread the caused cut region. Plain, Twill, and Hopsack are appropriate weave patterns for soft vest applications because of their high in-plane properties. The crimp in the fabric structure acts as an impact absorbing mechanism due to the friction between the yarns and the fibers' slippage. On the other hand, the Satin weave structure is commonly preferred for rigid armor applications due to its low crimp degree. These fabrics were woven through a weft-backed weaving system with a weave ratio of 3:1 using a handloom machine. In this weaving method, the weft density is increased, but the fabric handle properties are not changed. The schematic view of these weave structures is illustrated in Fig. 1. Acrylic yarn with a count of 15/4 Nm was utilized to weave the fabric samples. The physical properties of prepared fabrics are reported in Table 1. The applied sharp object's characteristic is also presented in Table 2.



Fig. 1. The Weave Pattern Design of a) Twill 2/2, b) Satin 1/7, and c) Hopsack 4/4 fabrics

Table 1. Physical Properties of Woven Fabrics						
Weave Pattern	Warp Density (1/cm)	Weft Density (1/cm)	Thickness (mm)	Mass per unit area (g/m ²)		
Twill 2/2	11	10	2.45	612.75		
Satin 1/7	11	16	3.50	940.76		
Hopsack 4/4	11	17	2.98	876.36		

Knife's Shape	Length (mm)	Thickness (mm)	Tip Angle ($^{\circ}$)	Application
	88	1.78	141	Slicing Knife

Table 2. Characteristic of the applied knife



2.2 Test Method

Fabric resistance against sharp object penetration in the vertical and horizontal directions was assessed by modifying Tensile Tester Machine, Instron model 5566. Distinct sample holders replaced the machine's bottom jaw to perform penetration and cut spread tests, and a sharp object was inserted in the machine's upper jaw. Set up of penetration and cut spread tests, the knife, and fabric position at the beginning of the test, and the knife movement direction is depicted in Fig. 2.



Fig. 2. Set up of a) Penetration, and b) Cut-spread tests

The penetration test was conducted as a test method proposed by Sun et al. [7]. According to Fig. 2a, the knife is in contact with the fabric surface at the beginning of the test. As the test starts, the knife moves downward, and penetration force and knife displacement are recorded. The penetration test was conducted at the speed of 100 mm/min, and the penetration process was executed to a depth of 45 mm.

The purpose of performing the cut spread test is to obtain the force required to expand the cut caused by sharp object penetration. As shown in Fig. 2b, the knife is placed in the rupture area caused by vertical knife penetration in the penetration test at the beginning of the test. As the test starts, the blade moves upward at the speed of 100 mm/min. The knife movement along the fabric is set to 30 mm. Three fabric samples' reputations were tested for both penetration and cut spread experiments, and the average of obtained data is reported.

3. RESULTS AND DISCUSSION

3.1. Evaluation of penetration force in fabric samples

Penetration force by knife displacement diagrams of three fabric samples with different weave patterns are shown in Fig. 3. Three stages of indentation, penetration, and perforation can be defined in the knife penetration process into the fabric. In all weave structures, at the beginning of the diagrams, a slight increase in the penetration force is observed with increasing displacement. At first, the knife is not able to penetrate the fabric and move the threads. In fact, this is the indentation step. Gradually, as the knife moves downward, the slope of the graph increases, this area of the diagram can be attributed to the knife involvement with the fabric yarns. Growing the force by increasing the knife displacement eventually leads to the fabric stretching and cutting of the threads.



In this way, the penetration force reaches the maximum value in the diagram and then decreases.



Fig. 3. Force by knife displacement of fabric samples in penetration test

Differences in the penetration force of the Twill, Satin and Hopsack weave patterns; can be elucidated by differences in the yarn interlacing and weft density. The Twill weave pattern has the lowest resistance against knife penetration. In the Twill weave pattern, which has the lowest weft density, fewer yarns resist knife penetration than Satin and Hopsack fabrics, resulting in less penetration force in Twill fabric. On the other hand, Satin and Hopsack weave patterns, with nearly same weft densities were more resistant to the knife penetration.

Taking into account the weave patterns of Satin and Hopsack fabrics shown in Fig. 1, both fabric samples have one interlacing point between warp and weft yarns. It means that, for each weft thread, only at one point the position of weft yarns are changed in relation to warp yarns. For instance, in Hopsack weave pattern, weft yarns are positioned over four adjacent warp yarns, and in the next four warp yarns, weft threads are located under the warp yarns. On the other hand, in Satin weave pattern, in seven adjacent warp yarns, weft thread is placed over warp yarns, and in the eighth warp yarn, weft thread is below the warp yarn. Therefore, Satin and Hopsack fabrics are similar in terms of weft density and yarn interlacement. However, in Satin fabric, weft yarn floats are longer than Hopsack fabric, which allows the threads to move more easily, leading to lower resistance in Satin weave pattern.

In previous studies, it is reported that expanding the cut created in the fabric by sharp object penetration requires less force than penetrating the fabric and creating the cut in the first place [8]. The graphs in Fig. 3 are also a compelling example of this. As illustrated in Fig. 3, as the knife penetrates the fabric and punctures it, the force reaches the maximum value, and after creating the cut in the fabric structure, the force decreases. However, the force needed to expand the cut region in a perpendicular knife movement direction is not explored. In this regard, the cut spread test is performed in the next step, and fabric behavior is assessed.

3.2. Evaluation of cut spread force in fabric samples

Force diagrams by knife displacement of fabric samples are illustrated in Fig. 4. As can be seen from Fig 4, Twill weave pattern has the least resistance to cut spread. In Twill weave pattern, weft density and, consequently, the number of yarns resistant to the cut spreading force is fewer. Therefore, as anticipated, this weave pattern has the lowest resistance to cut spread compared to Satin and Hopsack fabrics. The longer yarn floats in the Satin fabric compared to the Hopsack fabric makes yarns easier to move against the knife, resulting in less force in the cut spread test. Therefore, as the penetration test, maximum force in cut spread test is related to



Hopsack, Satin, and Twill fabrics, respectively. Force and energy of fabric samples in penetration and cut spread tests are reported in Table 3.



Fig. 4. Force by knife displacement of fabric samples in cut spread test

1.04

2 94

4 02

10	uble 5. Force and energy of fabric samples in penetration and cut spread lesis					
			Twill	Satin	Hopsack	
	Penetration Test	Maximum Force (N)	90.54	196.64	221.93	
		Energy (J)	0.84	1.90	2.42	
	Cut Spread Test	Maximum Force (N)	123.24	229.53	349.74	

Energy (J)

Table 3. Force and energy of fabric samples in penetration and cut spread tests

In the cut spread test, the fabrics are in contact with the knife blade and cut by it. The knife blade is much blunter than the knife tip, and it is more difficult to cut the threads with it. Due to this, during the cut spread test, the knife can not easily cut the yarns, and as it moves upward in the fabric, it causes the fabric to shrink. In this case, the knife blade is faced with a bunch of threads instead of a single thread in the fabric structure. As a result, both force and energy values are greater in the cut spread test for all weave patterns than the penetration test.

4. CONCLUSIONS

In the current research work, resistance to sharp object penetration and spread of the resulting rupture in woven fabrics was examined. In this regard, the penetration and cut spread tests were performed on woven fabrics in different weave patterns, including weft-backed Twill $\frac{2}{2}$ Z, Satin $\frac{1}{7}$ weft, and Hopsack $\frac{4}{4}$. The effect of fabric constructional parameters, such as weft density and weave pattern, on fabric resistance to sharp object penetration and spread of the cut caused by sharp object was investigated. In the knife penetration test, the fabric resistance against vertical knife penetration relative to the fabric surface is obtained. In contrast, in the cut spread test, fabric resistance against horizontal knife movement relative to the fabric surface is measured. The results revealed that weft density and weave pattern considerably affect the fabric resistance against creating and spreading the cut caused by a sharp object. The higher the weft density, the higher the penetration resistance and cut expansion resistance since more threads resist the penetration of a sharp object or expand its resulting rupture in higher weft densities.

Furthermore, weave structure of the woven fabrics significantly affect its protective performance against assaults by sharp objects. Woven fabrics with weave structures having more firmness resist better against sharp object penetration. They also have greater resistance to the cut-



spread caused by penetration of sharp objects. Hopsack fabric has the highest force in penetration and cut spread tests due to its higher weft density than Twill fabric and shorter yarn floats compared to Satin fabric. In all fabric samples, the force and energy recorded in the cut-spread test are greater than the penetration test because in the cut spread test, the blunter part of the sharp object cut the fabric that requires more energy. Besides, in the cut spread test, the blade encounters a bunch of yarns since it pulls fabric as it moves downward.

REFERENCES

[1] M. El Messiry, E. Eltahan, "Stab Resistance of Triaxial Woven Fabrics for Soft Body Armor", Journal of Industrial Textiles, vol. 45(5), pp.1062-1082, 2016.

[2] K. Bilisik, "Two-Dimensional (2D) Fabrics and Three-Dimensional (3D) Preforms for Ballistic and Stabbing Protection: A Review", Textile Research Journal, vol. 87(18), pp. 2275-2304, 2017.

[3] G. Nolan, S. Hainsworth, G. Rutty, "Forces Required for a Knife to Penetrate a Variety of Clothing Types", Journal of Forensic Sciences, vol. 58(2), pp. 372-379, 2013.

[4] F. Amirshirzad, F. Mousazadegan, N. Ezazshahabi, "Evaluating the Resistance of Metal Reinforced Multi-layer Textile Structure against Penetration of Sharp Objects", International Journal of Protective Structures, 2020.

[5] F. Amirshirzad, N. Ezazshahabi, F. Mousazadegan, "Study of Bending and Resistance to Sharp Object Penetration in Metal-Reinforced Fabrics", presented at the 21th Int. Conf. FASCICLE OF TEXTILES, LEATHERWORK, Oradea, Romania, 2020.

[6] E.J. Cowper, D.J. Carr, I. Horsfall, S.M, Fergusson, "*The Effect of Fabric and Stabbing Variables on Severance Appearance*", Forensic Science International, vol. 249, pp. 214–224, 2015.

[7] B. Sun, Y. Wang, P. Wang, H. Hu, B. Gu, "Investigation of Puncture Behaviors of Woven Fabrics from Finite Element Analyses and Experimental Tests", Textile Research Journal, vol. 81(10), pp. 992-1007, 2011.

[8] R. Nayak, I. Crouch, S. Kanesalingam, J. Ding, P. Tan, B. Lee, M. Miao, D. Ganga, L. Wang, "*Body Armor for Stab and Spike Protection, Part 1: Scientific Literature Review*", Textile Research Journal, vol. 88(7), pp. 812-832, 2018.



EXPERIMENTAL RESEARCH ON OBTAINING A CHROMATIC PALETTE ON HEMP FABRIC BY COMBINING WELD AND MADDER DYES

BUDEANU Ramona

"Gheorghe Asachi" Technical University of Iasi, Faculty of Industrial Design and Business Management, 67 D. Mangeron Street, Iasi 700050, Romania, ramona.budeanu@academic.tuiasi.ro

Corresponding author: Budeanu Ramona, E-mail: ramona.budeanu@academic.tuiasi.ro

Abstract: In the current world, ecology and sustainability are emerging as a concept. In this context, there is a tendency toward a cleaner industry with better use of energy, replacing synthetic chemicals with raw materials derived from natural and renewable resources, so that they can be preserved in nature. In this regard, the attention of researchers and industry is focused on natural products from nature, such as natural fabrics and natural dyes, which have a remarkable potential. The paper presents the results of an experimental research aimed at developing an ecological process of dyeing hemp fabric and obtaining a chromatic palette with 16 variants by using the natural dyes Weld (Reseda Luteola L.) and Madder (Rubia Tinctorum L.) in different combinations and concentrations. Also, the influence of two types of pre-treatment applied to the samples was analysed, which were applied in order to improve the absorption capacity of the dye, namely scouring with enzyme and scouring and bleaching in one step process. By applying the treatments before the dyeing process and by combining the two natural dyes in different combinations and concentrations, the aim was to obtain a chromatic palette. Wash fastness was evaluated according to ISO 105-C06 standard procedure. The experimental research in this paper is in line with current trends and concepts, offering real solutions in the development of an ecological and sustainable textile finishing process, by using natural hemp fabric and natural dyes in the dyeing process.

Key words: ecological textile products, natural dyes, hemp, sustainability, colours

1. INTRODUCTION

In the current context of globalization, there is a significant worldwide interest in the ecological phenomenon, which point the attention of researchers and industry to the use of materials, processes and technologies in order to ensure the development of organic products. In this context, the ecological phenomenon has a significant importance, as many researchers in various fields of science, and especially in the textile and fashion industry, have turned their attention to products coming from nature [1, 2]. Ecological awareness manifested in all fields, both nationally and internationally, has influenced research, but also the textile industry and the fashion industry.

Hemp is one of the most ecological and versatile natural textile plants [3, 4] and natural dyes considered to be biodegradable, non-allergic and non-toxic, are of remarkable interest.

Against the background of concerns about globalization, natural dyes have become an alternative to synthetic dyes. Natural dyes, obtained from plants, minerals and insects are ecological



and biodegradable, with a minimal impact on the environment, as researchers pay great attention to all areas of application of natural dyes [1, 5, 6].

Natural dyes are not an innovation, but a revival of a rich tradition, and cannot be compared with synthetic dyes only in terms of efficiency for industrial applications. After the current renaissance of dyes coming from nature there is a significant interest in the field of research in relation to everything concerning the pre-treatment process and the process of dyeing with natural dyes. Natural dyes appear to be the most appropriate substitute to the relatively toxic synthetic dyes [2, 3, 4]. They are believed to be safe because of non-toxic, non-carcinogenic and biodegradable nature. Further, natural dyes do not cause pollution and waste water problems.

From literature it is known that Weld and Madder were important sources of colour in textile production from ancient times. The natural dye Madder have been used for dyeing fabrics to the colour red, and the natural dye Weld for yellow colour, and also was usually combined with red dyes to produce different shades of orange.

Weld (Reseda luteola L.) is a perennial plant that produces a yellow dye (luteolin) from its foliage and flowers. This dye is the flavonoid yellow dye source that produces the most stable yellow shades and thus have been widely used for dyeing [7, 8, 9].

Madder (Rubia Tinctorum L.) is a plant anthraquinone red dye contain different anthraquinones, of which the most prominent structures are alizarin and purpurin that are believed to account for the red color [7, 8, 10, 11, 12].

Weld and Madder type dyestuffs belong to the group of mordant dyes which need a pretreatment with a metal salt, the metal salts most in use are alum compounds [12].

Ecological dyeing with natural dyes is a topical issue. Research in this area focuses on obtaining a wide range of colours, tones and shades, on varying the amount of mordant, on the combination between mordants, on the decrease in the working time and on varying temperature.

Recent developments in the process of finishing of textiles with natural dyes are mainly based on the modification of natural fibres by using different agents before treatment and after treatment. They are used to improve colour, speed and functionality characteristics, and are frequently evaluated in numerous studies carried out.

From an ecological point of view, natural dyes are a viable alternative to synthetic dyes and can be used in the development of ecological textiles, by adapting the dyeing methods of the new systems.

2. EXPERIMENTAL RESEARCH

The experimental research in this paper aimed to develop an ecological process in the field of textile finishing. Pre-treatments of fabrics before the dyeing process with natural dyes aimed at applying two types of treatments, namely scouring with enzyme and scouring and bleaching in one step process. The dyeing process involved the combination of natural dyes Weld (Reseda Luteola L.) and Madder (Rubia Tinctorum L.), in different combinations and concentrations of dye. By applying the pre-treatment procedures of fabric samples and combining Weld and Madder dyes in different combinations and concentrations, the aim was to obtain a chromatic palette.

The hemp fabric (hemp 100%, fineness of warp yarn: Nm=10/2 and fineness of weft yarn: Nm=10/1 with specific weight: 276 g/m2) realised in Romania was used.

In this experimental study were used following: enzyme pectinase BioPrep 3000L (Novoyzmes), non-ionic surfactant Triton X-100 (Sigma-Aldrich), NaOH 38°BE, 33%, Hidrogen peroxide 35% and aluminium sulphate Al₂(SO₄)₃ (Acros Organics), Tannex CB and Tanaterge Advance (Tanatex Chemicals B.V.).



Natural dyes Weld and Madder were supplied from Couleurs de Plantes (France).

All experiments were performed with demineralized water and the 5 grams textile material was used as sample.

2.1. The pre-treatments process of samples

Pre-treatments of fabric samples before the dyeing process with natural dyes aimed to applying two types of treatments, namely scouring with enzyme to obtain a ecological textile finishing, and scouring and bleaching in one step process to obtain a high degree of white necessary for dyeing in light colors. By applying both pre-treatments, the aim was to improve the absorption capacity for subsequent dyeing with natural dyes.

2.1.1. Pre-treatment with enzyme

Enzyme pre-treatment is an ecological and sustainable treatment that can thus avoid chemical bleaching and has been applied in order to improve the absorption capacity of the dye.

The experiments were performed in the Zeltex VISTA COLOR equipment, where the samples were introduced, at a temperature of 50° C for 1 h. The experimental study aimed to applying the pre-treatment with 2 ml enzyme pectinase BioPrep 3000L and non-ionic surfactant Triton X-100. The role of the surfactant is to facilitate the absorption capacity of the enzyme, greatly improving the water absorption capacity, thus obtaining a high degree of hydrophilicity necessary for dyeing. The concentration of the buffer solution was constant, at 0.5 M for a pH of 8.0.

After pre-treatment, the samples were washed in cold water and dried in a drying machine for 50 minutes at 50° C.

2.1.2. Pre-treatment scouring and bleaching in one step

In order to improve the absorption capacity of the dye and to obtain a soft touch with hygienic-physiological performance properties, with a high degree of white necessary for dyeing in light colors, the samples were subjected to a process of scouring and bleaching in one step process.

The experiments were performed in the Zeltex VISTA COLOR equipment, where the samples were introduced, at a temperature of 98° C for 1 h. A concentration of 5 ml of H₂O₂ was used, because it offers both an optimal absorption capacity and a high degree of white.

In the pre-treatment scouring and bleaching in one step, the receipe includes: 5 g/L NaOH $38^{\circ}BE$, 33%; 1 ml/L Tannex CB; 5 ml/L H₂O₂; 2 ml/L Tanaterge Advance.

Optimal conditions for this experiment are a temperature of 98° C, a duration of 1 h and 5 ml of H₂O₂. After the bleaching process, the fabrics were washed with water at 90° C, for 1 h, in the Linitester equipment, after which they were washed with cold water and dried in a drying machine, for 1 h at 50° C.

2.1.3. Testing the samples after applying the two pre-treatments

The influence of the two types of finishing treatments applied to the samples was analysed by determining the water absorption capacity of the fabric samples, obtaining values of 1 sec. in the case of bleaching, respectively 2 sec. in case of the enzymatic treatment, which reflect, in both cases, a very good water absorption capacity. The properties of textiles after the application of treatments were determined by measuring the water absorption rate. The measurements were performed on different surfaces of the sample. The water absorption rate was determined by means of the pipetting method (AATCC Test Method 39-1980), which is a method of analysis from this point of view.

Figure 1 presents the untreated sample of hemp fabric, and also the results obtained after the two pre-treatments process applicated.





Fig. 1. Untreated samples; Scouring with enzymes; Scouring and bleaching in one step

2.2. The process of dyeing with natural dyes

Dyeing is a method which imparts beauty to the textile by applying various colours and their shades on to a fabric. In the dyeing process with natural dyes Weld and Madder were used two procedures: mordanting and dyeing procedure.

2.2.1. Mordanting procedure

The mordanting procedure was achieved with $16\% \text{Al}_2(\text{SO}_4)_3$; fabric: liquor ratio 1:30; duration 1 h; temperature 98°C. The mordanting process was carried out in the laboratory, with the Zeltex VISTA COLOR equipment, using 150 ml of demineralized water, 0.8 grams of aluminium sulphate Al₂(SO₄)₃. The 5 grams textile material was treated with mordant for 1 h at 98°C. After the mordanting stage, the fabrics were washed thoroughly with cold water.

2.2.2. Dyeing procedure

The dyeing procedure was achieved in the Zeltex VISTA COLOR equipment, also used in the mordanting procedure, at a temperature of 98°C, for 1 h with following dye concentrations for:

- 1. The combination of Weld and Madder dyes: 5% concentration of dye (0,50 gr. Weld with 0,25 gr. Madder) and 10% concentration of dye (1 gr. Weld with 0,50 gr. Madder)
- 2. The combination of Madder and Weld dyes: 5% concentration of dye (0,50 gr. Madder with 0,25 gr. Weld) and 10% concentration of dye (1 gr. Madder with 0,50 gr. Weld)

After being dyed, the samples were rinsed intensively with hot water at 80°C, followed by rinsing with cold water. Finally, the dyed samples were dried in the drying machine for 50 minutes, at a temperature of 50° C.

2.2.3. Colour Fastness tests

The colour fastness tests was achieved in the Linitester equipment, at the temperature of 40°C, for 30 min, with colour testing detergent: ECE - Color Detergent, according to the Fastness Test ACC. ISO 105-C06 (Gewebe GmbH Test, Germany). The solution was prepared by dissolving 4 grams of detergent and 1 gram of sodium carbonate /1L of water.

3. RESULTS AND DISCUSSION

The experimental research in this paper aimed to obtain a chromatic palette by using and combining Weld and Madder dyes in different combinations and concentrations.

Figure 2 presents the results of dyeing for the combination of Weld and Madder dyes.

Figure 3 presents the results of dyeing for the combination of Madder and Weld dyes.





Fig. 2. The results for combination Weld and Madder (5% and 10% concentration of dye) MADDER - WELD



Fig. 3. The results for combination Madder and Weld (5% and 10% concentration of dye)

The experimental research carried out in this paper demonstrates the obtaining of both an ecological dyeing process, and also a chromatic palette following the application of the two types of pre-treatment and the combination of dyes using different combinations and concentrations.

Absorption time values obtained from enzyme treatments are comparable to those obtained from the scouring and bleaching treatment. Therefore, enzyme treatment is preferable, when the colour variant does not impose light colours and shades because it is an ecological treatment.

From the strict point of view of obtaining a higher degree of whiteness, the use of hydrogen peroxide H_2O_2 is necessary for the possibility of dyeing in light and pastel colours or for obtaining a soft touch, with hygienic-physiological properties of performance regarding the moisture absorption capacity, and the ability to sterilize the textile substrate due to the biocidal effect of eliminating streptococcal pathogens, viruses or bacteria.

The utility of the experimental process used has a special applicative character, by avoiding the classic dyeing using chemical reagents with potential pollutant and by using aluminium sulphate, the volume of wastewater and their degree of loading is considerably reduced. The use of this process has an immediate applicative character, in conditions of industrial processing.

4. CONCLUSION

As it can be seen, this paper is an experimental research that meets the current and prospective requirements of the textile industry on the use of natural dyes extracted from nature in the dyeing process. It can be concluded that through the process of dyeing with natural dyes and the use of natural fabrics, such as hemp, ecological textile products can be made, and by combining the dyes a chromatic palette can be obtained. The sustainable and diverse using of natural resources is significant in the development of environmentally beginning processes and products in the future.



ACKNOWLEDGEMENTS

The author would like to express her content to the staff of the "Research Centre for Design &Technology", Saxion University of Applied Science, namely to Pramod Agrawal, Ger Brinks and Henk Goijer for their support during the research internship in Enschede, the Netherlands, and last but not least for completing this research.

REFERENCES

[1] Q. Khadijah, M. Heba, "Environmental production of fashion colors from natural dyes", International Journal of Physical Sciences, vol.8 (16), 2013. [Online]. Available: https://www.researchgate.net/publication/259192599_Environmental_production_of_fashion_colors _from_natural_dyes.

[2] R. Budeanu, A. Curteza, R. D. Cezar, "Experimental Researches Regarding The Ecological Dyeing with Natural Dyes", Autex Research Journal, Vol. 14, No 4, 2014. ISSN (Online) 2300-0929. [Online]. Available:, DOI: <u>10.2478/aut-2014-0029</u>.

[3] R. Budeanu, An Approach in the Field of the Ecolgical Textile Products from Hemp – Tradition, Research and Innovation, 15th AUTEX World Textile Conference, 2015, Performantica, CD publication, ISBN: 978-606-685-276-0.

[4] R. Budeanu, A. Curteza, "A Sustainable Approach in the Fashion Industry – The use of naturally dyed hemp fabrics", 8th International Textile in The Fashion Industry – The use of naturally dyed hemp fabrics – Magic World of Textiles, 2016, pp. 437-438.

[5] D. Shukla, P.S. Vankar, Natural Dyes for Textiles, Sources, Chemistry and Applications, A volume in Woodhead Publishing Series in Textiles, 2017, Pages 141–166.

[6] S. Verma, G. Gupta, "Natural dyes and its applications: A brief review", vol.4, issue 4, 2017. [Online]. Available: <u>http://ijrar.com/upload_issue/ijrar_issue_490.pdf.</u>

[7] A. Shams-Nateri, A. Hajipour, E. Dehnavi, E. Ekrami, "Colorimetric study on polyamides dyeing with weld and pomegranate peel natural dyes", Clothing Text. Res. J., 32, 2014, 124-135.

[8] H., Willemen, G. J. P. van den Meijdenberg, T.A van Beek, G. C. H. Derksen, "Comparison of madder (Rubia tinctorum L.) and weld (Reseda luteola L.) total extracts and their individual dye compounds with regard to their dyeing behaviour, colour, and stability towards light", 2018. [Online]. Available: <u>https://doi.org/10.1111/cote.12384.</u>

[9] D. A. Peggie, A. N. Hulme, H. McNab, A. Quye, "Towards the identification of characteristics minor components from textiles dyed with weld (Reseda Luteola L.) and those dyed with Mexican cochineal (Dactylopiuscoccus Costa), Microchim Acta 162:371-380, 2008.

[10] Willemen, G. J. P. van den Meijdenberg, T. A. van Beek, G. C. H. Derksen, "Comparison of madder (rubia tinctorum 1.) and weld (reseda luteola 1.) total extracts and their individual dye compounds with regard to their dyeing behaviour, colour, and stability towards light", *Color. Technol.*, 135, 2019, 40-47.

[11] M. R. Shahparvari, S. Safapour, K. Gharanjig, "Study on kinetic behavior and dyeability of woolen yarn with madder and cochineal natural dyes", J. Color Sci. Technol., 10(2016), 195-206.

[12] A. Haji, "Dyeing of cotton fabric with natural dyes improved by mordants and plasma treatment", Prog. Color Colorants Coat. 12, 2019, 191-201.



THE ANALYSIS OF THE SYSTEM "HUMAN–CLOTHING– ENVIRONMENT" WITH APPLICATION IN THE DESIGN OF FUNCTIONAL ADAPTIVE PRODUCTS

FLOREA-BURDUJA Elena^{1,3}, RARU Aliona^{2,3}, FARÎMA Daniela³, IROVAN Marcela¹

¹ Technical University of Moldova, Faculty of Textile and Polygraphy, 4 Sergiu Radautan Street, Chisinau MD-2019, Republic of Moldova

² Technical University of Moldova, Direction of Academic Management and Quality Assurance, 168 Stefan cel Mare Street, Chisinau MD-2004, Republic of Moldova

³ Gheorghe Asachi Technical University of Iasi, Faculty of Industrial Design and Business, 29 Mangeron Street, Iasi 700050, Romania

Corresponding author: Florea-Burduja Elena, E-mail: <u>elena.florea@mctt.utm.md</u>

Abstract: Increasing the life quality for persons with disabilities represents an important problem for the whole society, locally and even globally. This group of people is the most vulnerable and the less protected, as they are limited in movements, actions and independence. Those require constant support, a lot of familial and social care, and a period to adapt, in order to begin a new life. This study presents the results of the research based on the interaction of several elements, that define the system: "persons with amputated inferior limbs-functional clothing-environment", achieving by that a database, that would eventually allow the solving of the main problem: social reintegration of this group. The aim of this study consists in determining the most important factors that describe each element of the above-mentioned system. The interaction of these factors allow us to identify several problems: disability problems, social-economic breaches, psychologic barriers, recovery processes, problems that concern the creation and the exploitation of multifunctional clothing, etc. Functional clothing is designed to ensure the needs for the potential customers with disabilities. Also, following the studies were established the factors that have a negative influence on social integration and personal rehabilitation. These appear as a list of restrictions related to the environment in which the person with locomotor disabilities lives.

Key words: clothes for people with disabilities, adaptive clothing, functional clothing, design, cofort.

1. INTRODUCTION

Everywhere in the world disabled persons achieve weaker results when it comes out about health, modest achievements in education, contribute less in economic life and are generally poorer than totally healthy persons. This is because disabled persons confront barriers, when accessing services, which that are totally normal for healthy persons, such as health, education, employment, transport and access to information [1].

One of tree problems that a disabled face is social integration. To help a disabled integrate in the social community, it is necessary to offer them psychological comfort, which lacks, due to their lower self-confidence. This self-confidence depends a lot on physical and psychological state of the person, and of course: on the way they look.



Clothing meant for disabled shall be ergonomic and shall allow the disabled to perform all their locomotor actions, that will ensure their day to day life. Also, clothes shall wipe out the difference between social needs persons and normal persons, and to ensure psychological comfort. All of these shall help increasing the quality of life of the disabled by giving the opportunities to participate on diverse social events.

The design of adaptive-functional clothing shall correspond to the requirements of the disabled, which is a complex procedure that needs constant research, with knowledge from vast domains. The sophistication of those clothes intended for the disabled are based on two main factors. Firstly, the clothing shall have an esthetic aspect. Clothing shall correspond to the esthetic needs of the disabled and not to highlight their impairments. Clothing shall ensure psychological balance, chromatic harmony and help camouflage the disabilities. Secondly, the clothing shall correspond to the exterior aspect of the disabled, in statics and dynamics, to age, health state, and other factors. Clothing shall be comfortable when dressing up and getting undressed and shall imply minimum activity of the human factor, as less as possible.

2. THE ANALYSIS OF THE SYSTEM "HUMAN – CLOTHING– ENVIRONMENT" CONCERNING THE PROCESS OF ADAPTATION OF THE PERSONS WITH BOTH INFERIOR LIMBS AMPUTATED

Clothing covers more than 80% of the human body, and form together the system "humanclothing-environment". Whiting this system, the product is found in continuous contact with human body and interacts with it. For this reason, products shall satisfy the disabled, offering him psychological and physical comfort [2].

The interaction of the locomotor disabled persons and clothing shall be researched within the ergonomic factor at a psychological level.

In the process of exploitation of the product, the disabled may feel either comfortable either uncomfortable, heat or cold, pressure over some body parts, etc. All those sensations mark an impact over the general state of the disabled and its activity performance. Consequently, when creating and designing adaptive clothing, shall be taken in consideration a more complex anthropometric harmonization of the product of the human body, in conformity with its ergonomic requirements.

With this aim, we will further analyses the system "human-clothing-environment", where, the main role is assigned to the person with both inferior limbs amputated (figure 1).



Fig. 1: Elements of the system "human-clothing-environment"

When we analyses the system "human-clothing-environment", it is necessary to approach every single element of the system. This will allow us to understand better the problem and to establish specific solutions.



The element "person with both inferior amputated limbs" represents the first component of the system, and the most important one. This group reflects the physical state whether is just one limb amputated, or several amputations [3]. Firstly, patients need to adapt to the new physical condition, and the psychological one, by adapting to the feeling of a loss of a limb. They have to adapt to the loss of its function, their new body image and other's acceptance [4].

Most of the disabled that suffered amputations, may be classified into two categories [5,6]. First category includes the young healthy persons, which, suffer from any diseases or tumors, as a result of any accidents. The second category includes the elderly, that suffer from chronic diseases, which lead to other major problems, that eventually require amputation, in order to save the life.

Amputation of the inferior limbs are mainly caused by cardiovascular chronic diseases, diabetes, severe trauma. Most of the studies show that 90% of the amputations strictly refer to the inferior limbs [4, 7 - 10].

The second element of the system concerns the clothing assigned for the disabled. For those persons, right clothing is a way of minimizing the effort lodged by the disabled, to live a normal life. It is necessary to take in count not just the clothing, but also the accessories and other tools. Also, it is necessary to keep in mind the type of the prosthesis, its muffs and racks that ensures movement.

The third element of the system is the environment. Over decades, resorting to such extreme surgery as amputation, represent a well-thought decision, in the favor of the patient and its family. Actually, everything is completely different. As it is important for the state to prioritize the expenses of the state budget, gradually, disabled persons were taken off evidence and surveillance. From this moment, a breakage between disabled patients and medical healthcare service happened, which affected mostly the disabled and their families. All those register an impact over the whole society and a gradual segregation of the disabled.



Fig. 2: The interaction of the elements of the system "human-clothing-environment"

Figure 2 shows us a strong interaction of the elements of this system. The system registers result only when all those micro-elements work perfectly among each other, and are headed on to solve the same common problem- adaptation of the persons with both inferior limbs amputated, throughout adaptive designed products.



3. CONCLUSION

The analysis of the system "human-clothing-environment" allowed us to determine some factors that can be used at the designing some adaptive-functional products. Eventually, those will allow the settlement of a few problems:

- insurance psychological comfort, by wiping out the difference between the healthy persons and the persons with special needs.

- quality life enhancement of the disabled, within socialization, especially study graduation, well payed jobs, etc.

- independence generation at home and in the society.

- removing bans and barriers that obstruct the disabled to participate in different social life activities

acceleration of social adaptation, as effect, attitude and mindset change.

REFERENCES

[1] Raport mondial privind dizabilitate. Publicat de Organizația Mondială a Sănătății în 2011, sub titlul "World Report on Disability,, Ediție tipărită, 2012 ISBN 978-973-0-13597-8 Available:

https://apps.who.int/iris/bitstream/handle/10665/44575/9789730135978rum.pdf;sessionid=BED65E B362B6E00EAA5C343C32126F43?sequence=20

[2] Ш.А., Саидова, И.А., Петросова, Е.Г. Андреева, ОБЗОР СОВРЕМЕННЫХ МЕТОДОВ ПРОЕКТИРОВАНИЯ ЭРГОНОМИЧНОЙ ОДЕЖДЫ // Современные проблемы науки и образования. – 2014. – №4.; Available: http://www. science-education.ru/ru/article/view?id

[3] E. Carolin Horne. (2009). Quality of Life in Patients with Prothetic Legs: A Comparison Study. In: J Proth and Ortho, vol. 21, nr. 3.

[4] Tăbîrță Alisa. Reabilitarea medicală prin tehnici kinetice active a pacienților cu dizabilități prin amputarea membrului inferior: studiu clinico-funcțional. Teză de doctor în științe. Chișinău 2018. Available: http://www.cnaa.md/files/theses/2018/54217/alisa_tabirta_abstract.pdf

[5] Ghidul protezatului. [sursă electronică] // https://www.ortopedica.ro/media/wysiwyg/ download/ghid-protezat.pdf

[6] Ghid pentru pacienții cu amputații de membru inferior [sursă electronică] // Available: https://ortoprofil.ro/wp-content/uploads/2015/12/Ghid-pentru-pacienții-cu-amputații-de-membru-inferior.pdf

[7] Cl. Szabo, Asistența socială în protecția persoanelor cu handicap. Cluj Napoca 2012, Available: http://www.incluziunesociala.ro/upls/89_protectia_sociala_a_pers_cu_handicap.pdf

[8] Antonescu D. M. Patologia aparatului locomotor, vol.1. București: Editura Medicală, 2006.

[9] A., Alvarsson, B., Sandgren, C., Wendel ş.a. A retrospective analysis of amputation rates in diabetic patients: can lower extremity amputations be further prevented. In: Cardio Diabetol, 2012, nr. 2, p. 11-18.

[10] E., Florea-Burduja, A., Raru, M., Irovan, D., Farîma, A., Tăbîrță (2020, Sept.). The role of clothing in various stages of assisting people with amputations (review). Annals of the University of Oradea a Fascicle of Textiles, Leatherwork, [Online]. 21(2), University of Oradea a Fascicle of Textiles, pp. 27-30. Available: http://textile.webhost.uoradea.ro/Annals/Volumes.html



BIOACTIVE HYDROGEL TYPE CARRIER SYSTEMS AIMED FOR TEXTILE WOUND DRESSING

CHIRILĂ Laura¹, ȚIGĂU Andreea¹, CONSTANTINESCU R. Rodica², OLARU Sabina¹, POPESCU Alina¹, STAN Miruna^{3,4}

¹National Research & Development Institute for Textiles and Leather, 16 Lucretiu Patrascanu Street, 030508, Sector 3, Bucharest, Romania

² National Research & Development Institute for Textiles and Leather, Leather and Footwear Research Institute (ICPI) Division, 93 Ion Minulescu Street, 031215, Bucharest, Romania

³ Department of Biochemistry and Molecular Biology, Faculty of Biology, University of Bucharest, 91-95 Spl. Independentei, 050095 Bucharest, Romania, e-mail: miruna.stan@bio.unibuc.ro

⁴ Research Institute of the University of Bucharest–ICUB, University of Bucharest, 91–95 Splaiul Independentei, 050095 Bucharest, Romania

Corresponding author: Ţigău Andreea, E-mail address: andreea.tigau@incdtp.ro

Abstract: Wound healing is a specific biological process related to the general phenomenon of growth and tissue regeneration. Polysaccharide gels are usually biocompatible and show several peculiar physicalchemical properties that make them suitable for a variety of biomedical applications. The hydrogels based on these types of polymers can cool the wound and reduce pain, which is helpful for burns or painful wounds. This paper aims to study the antibacterial activity of a layer-by-layer hydrogel "carrier" system based on sodium alginate and chitosan, which is further functionalized with active principles, like hyaluronic acid, or ZnO nanoparticles, or bacitracin. The synthesis of the hydrogels system was followed by the immobilization on textile woven fabrics made of fibrous blends of different chemical compositions, in order to achieve bioactive wound dressing for application in the treatment of inflammatory skin conditions. The textile materials used to produce the wound dressings have the same weave and warp thread (100% cotton, Nm 50/2), but different weft thread (100% acetate 130 dtex or 100% Lenpur Nm 34/1). For this purpose, several experimental variants of hydrogels were prepared and the treated textile materials were characterized from a physicalchemical and comfort point of view. The antibacterial activity and the biocompatibility of the textile materials functionalized with the hydrogels and active ingredients were also investigated. The textile materials treated with the synthesized hydrogels and subsequent bacitracin addition show an antibacterial effect on both S. aureus and E. coli test strains, while the hydrogels followed by ZnO nanoparticles addition show an antibacterial effect against S. aureus.

Keywords: bioactive textiles, ZnO nanoparticles, bacitracin, hyaluronic acid, antibacterial activity

1. INTRODUCTION

Wound healing is a specific biological process related to the general phenomenon of growth and tissue regeneration [1]. Various biopolymers are used in the production of wound dressings; an ideal skin wound dressing must accomplish the following requirements: provide a physical barrier to prevent further contaminations and injuries, absorb generated metabolites and enhances wound



healing capacity by maintaining a moist environment [2]. In hydrogel-based scaffolds, biopolymers like alginate and chitosan are used due to their biocompatibility, lack of toxicity and relatively low price [3]. The hydrogels can also cool the wound and reduce pain, which is helpful for burns or painful injuries [4]. Polysaccharide gels are biocompatible and show several physical-chemical properties that make them suitable for a variety of biomedical applications [5].

Among wound dressings, the bi-layer composite which consists of a dense outer layer and porous sub-layer has a good ability to promote the healing process and can be synthesised using a variety of biopolymers. Usually, the outer layer is designed for the prevention of bacterial invasion, and possibly to act as a rate-controlling layer for water vapor permeation. Meanwhile, the inner layer is designed for attachment to wound tissue and the drainage of wound exudates [1].

Chitosan is a natural polymer that is widely applied in tissue engineering [3] and present several advantages, such as: low cost, high biocompatibility, biodegradability and ease of chemical modification. Alginate is a linear anionic polysaccharide derived from brown algae or bacteria. The hydrophilicity, excellent biocompatibility, and huge liquid-absorbing capacity make alginate an attractive material for wound dressings [4].

Among various biomaterials for wound management, hyaluronic acid plays a major role in the wound healing and tissue regeneration process [6]. It is a primary component of the extracellular matrix and its molecules strongly bind to water molecules and get heavily hydrated to form a viscous gel. This is the mechanism through which it regulates the viscoelasticity of biofluids and controls tissue hydration [5]. Wound dressings impregnated with antibacterial nanoparticles made of silver, gold, and zinc oxide (ZnO) have been extensively used in the literature. The unique properties of ZnO are to improve epithelialization, to enhance the local defence system and to reduce bacterial infection and inflammation [6].

2. MATERIALS AND METHODS

2.1 Materials

The following chemicals and auxiliaries were required for the development of layer-by-layer hydrogel "carrier" systems: medium molecular weight chitosan (Sigma Aldrich, Germany), sodium alginate (Sigma Aldrich, Germany), vegetable glycerine (99.5% purity) used to give plasticity to the bilayer polymeric structure (SC Herbavit SRL Roumania), hyaluronic acid obtained from Streptococcus equi (Sigma Aldrich, Germany), bacitracin obtained from Bacillus licheniformis, ≥ 65 IU/mg, (Sigma Aldrich, Germany), zinc oxide nanoparticles with a size distribution less than 50nm (Sigma Aldrich, Germany), deionized water served as a solvent for the preparation of the polymeric solution.

As for the textile materials used to produce the wound dressings, two types of fabrics were used, both of them presenting the same weave and warp thread (100% cotton, Nm 50/2), but different weft thread (100% acetate 130 dtex-sample code S_1 or 100% Lenpur Nm 34/1-sample code S_2).

2.2 Hydrogels Synthesis

Sodium alginate is the polymer selected for the first layer of the hydrogel structure. The polymeric solution was synthesized by dissolving a 5% concentration of sodium alginate in distilled water. The obtained solution was maintained under continuous magnetic stirring of 250-300 rpm for one hour until homogenization, followed by the addition of 10% vegetable glycerine, with the role of increasing the plasticity (elasticity) of the polymer matrix and increasing the stability of the structure. The second polymeric layer of the layer-by-layer structure is represented by chitosan. This polymeric solution was achieved by solubilizing 1.5% chitosan in deionized water and subsequently



by adding 1% acetic acid. The solution obtained was maintained under magnetic stirring at 300–400 rpm over a two-hour time interval. The obtained hydrogels were used to perform the functionalization treatments of textile materials (**Table 1**).

2.3 Functionalization Treatments

The first step of the textile materials functionalization consists in the application of the first polymeric layer (hydrogel based on sodium alginate) by the padding method (4 passes through the squeezing rollers, pressure 2,7 bar), followed by drying operation at 50°C for 5 minutes, using a laboratory oven for drying-heat setting operations (ROACHES, UK). The textile material treated in the first step with sodium alginate hydrogel was then treated in the second step with the polymeric solution of chitosan, using the same equipment's and process parameters.

In order to add active principles to the textile surfaces, solutions containing certain amounts of hyaluronic acid, or ZnO nanoparticles, or bacitracin were obtained and subsequently applied on the textile materials by the padding, under the conditions described above. The technological flow can be seen in **Fig. 1**.



Fig. 1. The technological flow used for the functionalization of the textile woven fabrics by adding active ingredients in the multilayer polymer system immobilized on the textile surface

Woven	Fibrous co	omposition		Functionalized	
sample code	Warp	Weft	"Carrier" system	sample code	
S 1	100% cotton, Nm 50/2	100% acetate, 130 dtex	Chitosan–sodium alginate– hyaluronic acid	S ₁ -HA	
S 2	100% cotton, Nm 50/2	100% Lenpur, Nm 34/1	Chitosan–sodium alginate– hyaluronic acid	S ₂ -HA	
S_1	100% cotton, Nm 50/2	100% acetate, 130 dtex	Chitosan–sodium alginate– ZnO nanoparticles	S ₁ -ZnO	
S ₂	100% cotton, Nm 50/2	100% Lenpur, Nm 34/1	Chitosan–sodium alginate– ZnO nanoparticles	S ₂ -ZnO	
S1	100% cotton, Nm 50/2	100% acetate, 130 dtex	Chitosan–sodium alginate– bacitracin	S ₁ -B	
S_2	100% cotton, Nm 50/2	100% Lenpur, Nm 34/1	Chitosan–sodium alginate– bacitracin	S ₂ -B	

Table 1. Experimental variants of textile materials treated with hydrogel type carrier systems



3. CHARACTERIZATION METHODS

3.1. Physical-mechanical and comfort characteristics evaluation

In order to evaluate the influence of the functionalization treatments on the main physicalmechanical and comfort characteristics of textile woven fabrics, they were analysed before and after functionalization from the point of view of: mass (SR EN 12127:2003), hydrophilicity (by drop test) according to STAS 12751/89, water vapor permeability (STAS 9005:1979), air permeability (SR EN ISO 9237:1999).

3.2. Antibacterial activity evaluation

For the testing of antibacterial activity, the diffusion method in agar was used according to the standard SR EN ISO 20645:2005. For the experimental part, cultures of ATCC 6538 strains of Staphylococcus aureus (gram positive) and ATCC 8739 Escherichia coli (gram-negative) in a liquid medium were used. The evaluation method allowed the qualitative assessment of antibacterial activity based on the measurement of the inhibition zone obtained on the tested strains.

3.3. Biocompatibility evaluation

For the biocompatibility tests, the level of nitric oxide (NO) released in the culture environment was determined and the MTT test (based on the reduction of a yellow tetrazolium salt-3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide or MTT, to purple formazan crystals by metabolically active cells) was used to determine cellular viability. To perform these tests, the following preliminary steps were implemented: 1) Cultivation of HaCaT keratinocytes; 2) Exposure of HaCaT keratinocytes to textile material: in 24 well-plated and in inserts for cell culture. All results were calculated with average values of at least three independent experiments (n=3).

4. RESULTS AND DISCUSSIONS

4.1. Physical-mechanical and comfort characteristics evaluation

From the point of view of the influence of the functionalization treatments on the mass characteristic, it was found that regardless of the type of active principle used as final treatment layer (hyaluronic acid, or ZnO nanoparticles, or bacitracin), the multilayer polymer functionalization system as a hole generates an increase of the values of mass characteristics per surface unit, with about 35-40% in the case of woven fabric containing 100% acetate yarns and with about 26% in the case of woven fabric containing 100% Lenpur yarns in the weft direction.

From the comparative analysis of the air permeability values obtained for the treated and untreated textile materials it can be shown that the functionalization treatments cause a significative decreasing in the air permeability characteristic due to the constituent polymeric substances deposited on the surface of the textile support in the form of a semi-permeable film. The same conclusion can be drawn in the case of comparative analysis of the values of water vapor permeability, for which, a reduction between 28-37% of the value of this characteristic occurred in the case of woven fabric containing acetate yarns and 38-42% reduction in the case of fabric containing Lenpur yarns.

In terms of hydrophilicity determined by the drop test, it was found that the textile materials of both series treated with the polymeric system chitosan-alginate sodium-ZnO provide the highest values of hydrophilicity in the series studied. The determined characteristics are presented in **Table 2**.



Sample	Fibrous composition of yarns		Mass	Air permeability	Water vapor permeability	Hydrophilicity, drop test
code	Warp	Weft	(g/m ²)	(l/m²/sec)	(%)	(seconds)
S_1^*	1000/	1000/	136	260.4	42.4	instantaneous
S ₁ -HA		100% 100% cotton, acetate, Nm 50/2 130 dtex	191	23.42	30.4	>300
S ₁ -ZnO	,		188	22.42	26.3	26.95
S ₁ -B	Niii 30/2		184	25.08	27.2	270.58
S_2^*	1000/	1000/	172	386	45.5	instantaneous
S ₂ -HA	100% cotton, Nm 50/2	100%	217	89.66	27.8	>300
S ₂ -ZnO		, <u>1</u> ,	217	95.88	26.5	12.59
S ₂ -B	14111 30/2	1111 34/1	218	82.56	27.8	45.4

Table 2. Mass, air permeability and water vapor permeability of the textile materials functionalized with the layer-by-layer hydrogel type carrier systems

* Control samples: Textile woven fabrics before the application of functionalization treatment

4.2. Antibacterial activity evaluation

Analysing the data obtained for the antibacterial testing (**Table 3**), it can be concluded that in the case of the untreated textile structures, considered control samples, the test strains had a significant growth, the inhibition zones of bacterial growth being absent.

Among the functionalized treatments studied, the variants treated with the chitosan-sodium alginate-bacitracin system, shows antibacterial activity on both tested strains, respectively *E. coli* and *S. aureus*, for which the biggest inhibition zone were highlighted (16 mm). The textile materials treated with the polymeric chitosan-sodium alginate-ZnO nanoparticles polymeric system show low antibacterial activity against on *S. aureus* strain only, with a inhibition zone of 2.5 mm (**Table 4**).

The samples treated with chitosan-sodium alginate-hyaluronic acid system have an insufficient antibacterial effect, exhibited by a significant bacterial growth and the absence of inhibition zones of bacterial growth.

4.3. Biocompatibility evaluation

In the case of keratinocytes grown in the presence of textile materials impregnated with bacitracin, the cell viability was better compared to the results obtained in the case of keratinocytes grown in the presence of textile materials impregnated with hyaluronic acid or zinc oxide nanoparticles (**Fig. 2**).

Sample code	Inhibition zone (mm)		Evaluation	
F	E. coli	S. aureus	E. coli	S. aureus
S_1^*	0	0	Insufficient effect	Insufficient effect
S ₁ -HA	0	0	Insufficient effect	Insufficient effect
S ₁ -ZnO	0	H=2.5 mm	Satisfactory effect	Satisfactory effect
S ₁ -B	H=16 mm	H=16 mm	Satisfactory effect	Satisfactory effect

Table 3. Evaluation of the antibacterial activity for the obtained biomaterials



S_2^*	0	0	Insufficient effect	Insufficient effect
S ₂ -HA	0	0	Insufficient effect	Insufficient effect
S ₂ -ZnO	0	H=2.5 mm	Satisfactory effect	Satisfactory effect
S ₂ -B	H=16 mm	H=16 mm	Satisfactory effect	Satisfactory effect

* Control samples: Textile woven fabrics before the application of functionalization treatment

Table 4. Images of Petri dishes after the exposure to the *E.coli* and *S. aureus* strains of the S₁, S₂ woven fabrics before and after the functionalization







Fig. 2. Graphical representations of: a - Analysis of the cellular viability using the MTT test; b - Level of NO released in the culture environment to observe the pro-inflammatory effect after 5 hours of incubation in a two-dimensional system

Cellular viability values were close to those of the control, with some exceptions, such as bacitracin-treated textiles. Increased levels of nitric oxide were observed in textiles impregnated with bacitracin, in comparison with the other treated samples, suggesting a possible pro-inflammatory potential in the skin (**Fig. 2**).

5. CONCLUSIONS

Layer-by-layer hydrogel carrier systems based on sodium alginate, chitosan and certain bioactive substances were applied on the textile woven fabrics by the padding method to accomplish textile wound dressing for the application in the treatment of inflammatory skin conditions. The functionalized textile materials were characterized from several physical-chemical and comfort point of view. Also, antibacterial activity and biocompatibility were evaluated in order to prove the safety application of the developed wound dressing on the skin. It was found that regardless of the type of active principle used as a final treatment layer (hyaluronic acid, or ZnO nanoparticles, or bacitracin), the multilayer polymer functionalization system as a whole generates an increase of the values of mass characteristics per surface unit up to 40%, and a significative decreasing of air and vapor permeability and of the hydrophilicity. Biocompatibility evaluation demonstrates that the textile material functionalized with the studied hydrogels do not present skin irritation. Taking into consideration the antibacterial activity data, it was proved that the textile samples containing bacitracin show antibacterial effect for both *S. aureus* and *E. coli* test strains, while those that contain ZnO nanoparticles show antibacterial effect against *S. Aureus* only.

To sum up, wound dressings based on hydrogels are innovative materials that exhibit antibacterial activity and biocompatibility and are a suitable solution for the treatment of inflammatory skin conditions.

ACKNOWLEDGEMENT

This work was carried out through the Nucleu Programme, with the support of MEC, project no. 4N/08.02.2019, PN 19 17 03 01, project title: "Multifunctional integrated systems based on nanocomposites and pharmacodynamic therapeutic agents for different skin conditions – BIOPANTEX".



REFERENCES

[1] F. Han, Y. Dong, A. Song, R. Yin and S. Li, "Alginate/chitosan based bi-layer composite membrane as potential sustained-release wound dressing containing ciprofloxacin hydrochloride", Applied Surface Science, vol. 311, pp. 626-634, 2014.

[2] D. E. Rădulescu, D. M. Rădulescu, L. Chirilă and A. Popescu, "*The evaluation of novel wound dressings based on hydrogels*", Annals of the Oradea University Fascicle of Textiles, Leatherwork, pp. 103-108, 2020.

[3] M. T. Khorasani, A. Joorabloo, A. Moghaddam, H. Shamsi and Z. M. Moghadam, "Incorporation of ZnO nanoparticles into heparinised polyvinyl alcohol/chitosan hydrogels for wound dressing application", International Journal of Biological Macromolecules, vol. 114, pp. 1203-1215, 2018.

[4] M. Zhang, X. Zhao, "Alginate hydrogel dressings for advanced wound management", International Journal of Biological Macromolecules, vol. 162, pp. 1414-1428, 2020.

[5] S. Tiwari, P. Bahadur, "*Modified hyaluronic acid based materials for biomedical applications*", International Journal of Biological Macromolecules, vol. 121, pp. 556-571, 2019.

[6] Z. Hadisi, M. Farokhi, H. R. Bakhsheshi-Rad, M. Jahanshahi, S. Hasanpour, E. Pagan and M. Akbari, *"Hyaluronic acid (HA)-based silk fibroin/zinc oxide core-shell electrospun dressing for burn wound management"*, Macromolecular Bioscience, vol. 20, 2020.



DEVELOPMENT OF FUNCTIONAL PRODUCTS FOR PREMATURE BABIES USING THE CAD SYSTEM

DANILA Victoria¹, CURTEZA Antonela², BALAN Stela³

^{1, 2}, Gheorghe Asachi" Technical University of Iasi - Romania, Faculty of Industrial Design and Business Management, Blvd. Mangeron, No. 59A, 700050, Iasi, Romania <u>victoria.danila@mctt.utm.md, antonela.cuteza@tex.tuiasi.ro</u>

³Technical University of Moldova, Faculty of Textile and Poligraphy, avenue Stefan cel Mare si Sfint 168, 2004-MD, Chisinau, Republic of Moldova: E-mail: <u>stela.balan@adm.utm.md</u>

Corresponding author: Victoria Danila, victoria.danila@mctt.utm.md

Abstract: The development and implementation of a new model of sustainable clothing through the automated design system aims to optimize the design process and provide optimal solutions. The paper presents the SMART design concept implemented to obtain functional products for premature babies. The obtained results describe the process that integrates the sustainable design and the way of designing the clothing. The intention is for the model of sustainable clothing to provide wearers with functionally sustainable products and to solve some of the environmental problems in terms of clothing production. The proposed original constructive and technological solutions offer the necessary thermal and psychological comfort to premature babies, anthropometric correspondence both for the child's static positions and for his dynamics, and the product system reduces the dressing-undressing time in medical emergencies.

Key words: prematurity, SMART design, functional clothing, CAD system.

1. INTRODUCTION

The basic criteria for designing clothing for premature babies is that the products are functional and comfortable so that medical staff can undress and dress the child in less than 1 minute to provide routine medical treatments, medical examinations in emergency situations. Clothing must maintain a stable body temperature of 37° C in an environment with air conditioning of 24° C and relative humidity of 62% and be designed to reduce heat loss, allowing certain control areas to be easily opened. as needed, during checks and medical treatment by minimizing the body's exposure. Probes, tubes, wires and other devices used to monitor the patient's health should not be twisted when handling the child or as he / she moves when wearing the clothing. Clothing should allow children to move their limbs easily prematurely. The size of the product and the locking elements must be designed in such a way as to encompass the child's body, not to be too loose or too tight, so as to impede freedom of movement, increase ventilation or heat levels for the premature newborn. In addition, the selected seams should be as simple as possible to reduce the thickness in the assembled areas.

For the elaboration of the clothing products, the dimensional characteristics of the group of wearers, the additions of lightness and the adjusted clothing models were taken into account to allow the movement, growth and application of various threads from medical devices, etc. However, the



garment has minimal processing seams to reduce thickness, which could cause discomfort and increase productivity in the manufacturing industry, obtaining minimal waste and thus opting for the manufacture of sustainable functional products.

2. DEVELOPMENT OF FUNCTIONAL CLOTHING PRODUCTS USING THE CAD SYSTEM

Currently, we can note the importance of developing and using information technologies in the design of children's clothing.

The application of information technology in different stages of design and technology for the development of functional products for premature babies [1] will not only reduce the preparation time for the production of new models, but will also reduce the cost of developing new products, will respond quickly. to the requirements imposed, but most importantly will improve the quality of the production process of clothing for premature babies.

The design of functional assortments of children's clothing aims to meet the requirements [2] of consumers and medical staff who are directly involved in the medical procedures to which these children are subjected. In order to obtain appropriate products, anthropometric data, the degree of child development and advanced methods of designing shoulder-supported and waist-supported products based on the use of computer technology will be taken into account.

Ensuring these requirements is the initial stage in the development of children's clothing. In this sense, creating a database of information assistance for computer-aided clothing design is a task that needs urgent solution. The solution to this problem, in addition to increasing the efficiency of children's clothing [3], ensures an increase in meeting the needs of children in clothing with a high level of comfort and quality.

Based on the above, the paper presents the results of the process of optimizing the design of children's clothing, which consists in researching, creating and implementing scientifically justified input parameters: the results of anthropometric examinations of children's bodies, innovative design methods, rational design - technically adapted technological processing, which can reduce not only design and manufacturing terms, but also most importantly, significantly increase the quality of products that meet the needs of premature babies.

To achieve this goal, the following tasks have been solved:

• Study of the requirements imposed on this type of products for the design of functional clothing for children born prematurely.

• Classification of the range of functional clothing for boys and girls.

• Anthropometric data were taken from children with varying degrees of prematurity.

• A rational system of values has been defined, which allows the unification of dimensional parameters.

• Product lengths are determined based on anthropometric measurements.

• Development of technical documentation for the manufacture of clothing for premature babies.

• Development of the database that includes the basic patterns in the CAD system for the proposed models.

3. EXPERIMENTAL RESEARCH

Application part:

The stages of the SMART Design design process that we applied for the production of the series of sustainable functional products for premature babies are presented in figure 1:





Fig. 1. Product development stages - SMART Design

In order to optimize the process of designing functional clothing for premature babies, we went through the following steps: 1) After analyzing the needs and functional, ergonomic, aesthetic and economic needs of the wearer group, product ideas are generated to meet these requirements. Design requirements for children's clothing include ease of wear, washability, durability and versatility. 2) Based on the initial data, a database is created with basic patterns 3) which later facilitates the elaboration 4) of the appropriate solutions. From the proposed solutions, the model that corresponds to the pre-established requirements is selected 5), after which the basic pattern and the model pattern (BP and MP) are developed with the help of the CAD system (figure. 2). 6) At this stage, the prototype of the product is made, which then passes the verification in accordance with the indicated criteria. 7) The package of technical documentation is sent to the production section and the products are made. At the end there is 8) delivery of the product to the consumer.



Fig. 2. Development model for children with CAD system: a) basic pattern; b) model pattern; c) solutions model; d) final prototype model

At this stage, the size of the wearer and the requirements that the product [4,5] must meet have been taken into account. Subsequently, the calculation of materials necessary for manufacturing was made based on the frameworks generated by the system. This method of optimizing product development process for children is an effective and sustainable.

4. CONCLUSION

From the above we conclude the following:



1. The efficiency of the use of information technologies depends to a large extent on the quality of the adapted constructive solutions and of the technological processes of making clothing products for premature newborns.

2. Based on anthropometric studies on children from various groups of prematurity, the influence of various developmental factors on physical appearance was highlighted.

3. A classification of functional clothing for children was made, differentiated by groups of prematurity, based on the analysis of the requirements of medical procedures, in order to establish the assortment.

4. Statistical processing of anthropometric data was performed for 500 premature babies, a rational system of type-dimensions was developed - for different groups of prematurity, which allows the quantitative reduction of product sizes needed to make children's clothing.

5. Rational design parameters for functional clothing for premature babies have been identified, ensuring a good quality dimensional and aesthetic fit.

6. Development of technical documentation for the manufacture of children's clothing, which is the initial information for the creation of a database of information assistance for the automated design process of children's clothing.

8. Algorithms for designing basic and model patterns for premature baby clothing have been developed and introduced into the CAD system.

9. A database has been developed for the technological design and training subsystem, compatible with CAD design system for automating the process of designing and preparing technical documentation.

10. Testing the results of the works, performed in the form of an experimental verification and the implementation of technical and regulatory documentation developed for the design of functional clothing for premature babies.

All these will definitely contribute to obtaining functional clothing products with a high degree of dimensional and ergonomic correspondence for premature babies.

REFERENCES

[1]. H, Als ,G, Lawhon FH, Duffy, GB, McAnulty, R, Gibes-Grossman, JG. Blickman, Individualized developmental care for the very low-birth-weight preterm infant. Medical and neurofunctional effects. JAMA. 1994 Sep 21;272(11):853–8.

[2]. V., Danila, *Cerințe impuse îmbrăcămintei speciale pentru copiii nou-născuți prematur*. În: Conferința Tehnico-Științifică a Colaboratorilor, Doctoranzilor și Studenților, 16-18 noiembrie, 2017/Univ. Tehn. a Moldovei. – Chișinău: Tehnica-UTM, 2018, 81-84 pp. ISBN 978-9975-45-543-5.Vol. 2. – 529 p.

[3]. V., Danila, A., Curteza, S., Balan, M., Irovan, *Designing comfortable neonatal system clothes for premature babies.* 9th INTERNATIONAL TEXTILE, CLOTHING & DESIGN CONFERENCE – Magic World of Textiles October 07th to 10th 2018, DUBROVNIK, Croatia, pag.179-284. ISSN 1847-7275.

[4]. Danila Victoria, Curteza Antonela. Special care of premature babies and their specific functional clothes. Section 5 Functional textiles and clothing. 7-9 November CORTEP 2018, Iaşi. 17th Romanian Textiles and Leather Conference Cortep 2018-Book of Abstracts/ Manuela Avadanei. -Iasi: Performantica, 2018 ISBN 978-606-685-615-7, p.39.

[5]. DANILA Victoria, ISCHIMJI Ana, Durability product and impact on promoting sustainable production, Conferință științifică internațională "Patrimoniul cultural de ieri – implicații în dezvoltarea societății durabile de mâine" (ediția I), proiect 19.00059.06.20F/ MS, Direcția strategică "Patrimoniul național și dezvoltarea societății"114 pag., ISBN 978-9975-3290-4-0, p.85.



SMART VALORIFICATION OF RECICLABLE TEXTILE WASTE

DOBLE Liliana¹, BÖHM Gabriella², PORUMB Camelia Luminița³

^{1, 2}University of Oradea, Faculty of Energy Engineering and Industrial Management, Department Textiles, Leather and Industrial Management, 410058, Oradea, România, E-Mail: <u>liadoble@yahoo.com</u>

> ³ University of Oradea, Faculty of Art, Departament of Vizual Art, 410058, Oradea, E-mail: <u>cameliaporumb1968@gmail.com</u>

Corresponding author: Doble Liliana, E-mail: liadoble@yahoo.com

Abstract: Clothing belongs to the category of short-term textiles. In the field of clothing, the cycles that shape the moral wear and tear of the garment are followed at shorter and shorter intervals. If in the Middle Ages and even later clothes were bequeathed from one generation to the next, today, fashion cycles are followed from one season to the next. More and more questions are asked whether clothes are worth maintaining to reuse them next season. Can there be "ageless" clothes? Here we refer to clothing items that have a value through materiality, cut line, chromatic etc. For such "worthy" items, maintenance is a necessary process for adequate preservation, also extending the life, both physically-functionally and aesthetically. It is important not to forget that physical wear always leads to loss of both the function and the appearance for which the product was created. This is where the process of recycling both used clothing and waste left over from the manufacture of new products comes into play. Recycling brings many useful benefits both to the environment and to us as beneficiaries. This must be seen as a collective responsibility to ensure a sustainable future. Recycling is fundamental in promoting a prosperous economy, which is the new paradigm of sustainability, capable of reducing environmental implications and helping to create new opportunities, jobs in the textile industry.

Key words: recycling, textile waste, reuse, blazer, design

1. INTRODUCTION

One of the most necessary industries in the world is the textile industry. The textile products for the fashion industry are a big problem for the environment, because of the production process and consumption of resources. One of the methods of fighting pollution and textile waste is that of recycling and reuse [1].

In this paper we want to show that when we recycle, used materials can be transformed into new products, reducing the need to consume the natural resources of their production. By recycling some waste from production or by extracting pieces of other used items, we have been able to preserve both materials that have an important value for us and to use waste, which, in the manufacturing process, would implicitly increase production costs through energy consumption, water, etc.



2. FROM TEXTILE WASTE TO A FASHION PRODUCT

Nowadays we need to transform our economic model from a "pattern" of growth such as "obtaining, manufacturing, using, eliminating" (a linear model that assumes that resources are abundant, available and cheap to eliminate) to a "pattern" that favours the reuse, repair, reconditioning and recycling of existing materials and products, by preserving the value of products and materials for as long as possible [2]. This generates less waste, and, at the same time, decreases the pressure on precious resources and the environment

The circular economy model has recently gained a lot of attention worldwide from scientists, business people and authorities. The importance of the transition towards a more circular economy has also been noted in the European Union. The new regulations provide the enabling framework for the circular economy to flourish [3]. Implementing a circular textile industry model is a challenge that requires time and openness to new approaches. This new model requires the development of new initiatives, and even collaborations, on the use of raw materials, as well as the development of new technologies for the recycling of textile materials and their use in new clothing products [4].

Using a computer graphics program, the sketch was made, (fig.4.) of what we were going to design. The chosen product (fig.6) is a women's blazer, intended for the spring-summer season, which follows the body line, having a special structure and elegance by carefully assembling these fabric waste [5]. As raw material was used, also waste from tailoring(fig.1), pieces recovered from non-compliant products (defects) and defective fabric coupons that had to be disposed of in the shaming operation. Non-compliant products have defects that can't be corrected, thus being products that can't be marketed. In order to recover from the value of their losses, pieces or even entire parts are recovered, for re-using them.



Fig.1. Waste from tailoring



Fig.2. Non-compliant products







Fig.3. Preparation of waste for manufacture

Fig.4. Sketch of the product

In fig.3 are presented the pieces of fabric to be processed for the parts of the product to be made (fig.5). For this action, the pieces were treated umido-thermal, arranged in such a way as to take into account the warp direction of the fabric.

The next step was to cut out some rectangles. A very important step is to match the pieces so that the aesthetic appearance is a pleasant one, from a design point of view.



Fig. 5. Product's elements



Fig. 6. Finished product



Because it was intended to achieve a disordered effect, geometrical, there were pieces that were assembled on a diagonal line. This requires increased attention so as not to bring deformations of the textile surface.

The opportunity to recycle materials opens up new business horizons for small and mediumsized workshops as well as companies already established in the labour market, enriching their production. The use of industrial waste is difficult for large-series producers, who have an advanced degree of uniformity of production, they have specialized production lines, regular staff, delivery times and a high concentration of efforts to produce as much clothing as possible with as little labour costs as possible [6]. This causes a number of materials to be disposed of in the bin, increasing the amount of waste in landfills.

5. CONCLUSIONS

Since the recovery of industrial waste is difficult for large-series producers, collaborations must be established between them and the small business environment, covered by small workshops, various non-profit organisations or school organisations, which have the capacity to reuse them. Following the study carried out in this work, we applied methods of recovery of textile waste, such as the use of defective material coupons, textile waste from the tailoring process or even the reuse of pieces of an existing article.

REFERENCES

[1] A.P. Cobuz, M. Greenley, M. Orban, E.S.Lakatos *Recycling textile materials for environmental sustainability*, A XVII-a Conferință internațională multidisciplinară "Profesorul Dorin PAVEL – fondatorul hidroenergeticii românești", la Sebeș, 2017.

[2] K. Falkenberg, *The recycling concept in the context of the revision of the Waste Framework Directive*. Directive 20008/98/EC.

[3] M. Koszewska *Circular economy - challenges for the textile and clothing industry,* AUTEX Research Journal, DOI: 10.1515/aut-2018-0023.

[4] E.C. Loghin, A:G Topală, M. Avădanei, I. Dulgheriu, S.I. Ionesi, I. Ionescu, *Reutilizarea deșeurilor textile în produse cu valoare adăugată*, Buletinul AGIR nr. 1/2019,

[5] Anon. Breslo - Vinde-ti propriile creații sau Cumpără produse unicat de la cei mai talentați creatorii români. Breslo [online]. Comunitatea Creativă din România – Platformă online ce le permite creatorilor români să se conecteze între ei și cu cei care le apreciază munca. Văzut la data de 10 februarie 2021. Disponibil la adresa https://www.breslo.ro/.

[6] M. Blaga, D. Dan, Contribuții privind valorificarea deșeurilor textile, Buletinul AGIR nr. 1/2017.


NEW PARADIGMS IN DESIGNING THE MEDICAL TEXTILES

ENE Alexandra¹, MIHAI Carmen², SCARLAT Razvan³, GROSU Catalin⁴, HERTZOG Radu - Gabriel⁵, POPESCU Diana⁶

^{1, 2,3,4} The Research-Development National Institute for Textiles and Leather, 16 Lucretiu Patrascanu Street, 030508, Bucharest, Romania, E-mail: <u>office@incdtp.ro</u>

^{5, 6} "Cantacuzino" National Military-Medical Institute for Research and Development, 103 Splaiul Independentei, 050096, Bucharest, Romania, E-mail:<u>office.cantacuzino@mapn.ro</u>

Corresponding author: Grosu, Catalin, E-mail: catalin.grosu@incdtp.ro

Abstract: Foreign bodies penetrating deep into the wound at the time of tissue damage can cause chronic inflammatory responses that delay healing and can sometimes lead to the formation of granuloma or abscess. In general, a product intended for the treatment of gunshot wounds must be hypoallergenic, non-toxic and biocompatible; be able to prevent microbial contamination, to enable the gas exchange and exudate evacuation; to be flexible and adaptable to body contours; to be able to deliver, with the appropriate kinetics, important therapeutic compounds for the healing process and to be adherent. The researches were focused on performing of coupled simulations of the human body anatomy, analyse of the impact of the military weapons with different ballistic performances against human body, mathematical modeling of the electrospinning the textile structures used in particular case, was performed, with the help of a dedicated software, the simulation of the main areas of interest of the human anatomy, respectively: the circulatory, bone and muscular system. The results of the simulations enabled the description of the phenomena that take place on the human body subject to the action of the 5.45 x 39 mm infantry armament at subsonic speeds and the mathematical model allowed the prediction of the control of the mesh permeability and of the neo tissue cellular growth and proliferation.

Key words: software, anatomy and physiology, simulation, modelling, electrospinning.

1. INTRODUCTION

Shot wounds are characterized by an inlet and an outlet and the imaginary line joining the two orifices orients the organs affected by the projectile that passed through the body segment.

Although most wounds could be healed without major events, problems can sometimes occur are associated with failure or prolongation of the healing period. Failure in healing process of a wound within the expected time frame, usually leads to the development of a chronic lesion that cannot be easily treated due to the disruption of the orderly sequence of events associated with the natural healing process [1]. Excessive exudate production can cause, around the wound, maceration of healthy skin tissue and can inhibit the healing process. In addition, exudation from chronic wounds differs from acute wound fluid, with relatively higher levels of more corrosive and destructive tissue proteases. The odor and colour produced by the exudate can also have a negative impact on a patient's overall health and quality of life. Wound healing is a complicated process, which involves the adoption of different strategies, as a function of the types of wounds [2].



Depending on the area of the trauma, the infectious situation and the medical treatment, wound healing can be divided into different categories, including primary, secondary and sub-eschar healing.

2. WEAPONS AND HUMAN BODY INTERACTION

2.1. Coupled simulations of the human body anatomy

The anatomy of the circulatory system is represented by:

- blood vessel system: arteries, capillaries and veins (Fig. 1);

- lymphatic vessel system: capillaries, vessels that collect and conduct lymph and lymphoid organs.

The couplings between these systems are shown sequentially in Fig.1.

The skeletal system (skeleton) consists of 206 connected bones, in most cases through the joints and is divided into: cephalic skeleton, axial (thoracic cavity, sternum, spine) and appendicular (limbs). It was considered that does not present importance the simulation of the thoracic cavity, of the sternum or of the spine as the skeletal system could be simulated together with the muscular system. However, during the simulation process the bone tissue was considered, as the protection of these anatomic parts requires bulletproof protective equipment (helmet, vest), even when it comes to instruction and training, not only in the theater of operations. The images resulting from the simulations are presented in Fig. 2. For the simulation process the associated pathologies for this system (fractures, infection) at impact with weapons (bullet, shrapnel) were considered.



Fig. 1. Circulatory system: a. arthers, b. capilars, c. veins



Fig. 2. Bone tissue: a. periosteal fibrous layer, b. endost, c. bone marrow

The muscular system (with 600 skeletal muscles) is especially important for the field of application, because it is involved in maintaining the basic position and movements. Although the muscles are of three types (skeletal, cardiac, smooth), for simulation, the cardiac muscle was removed, because it was considered that it is not involved in these types of situations (shooting or burns), its role intervening rather after the development of pathologies and its action represents an



effect of interaction. In Figs. 3, 4 and 5 are presented layered (layer 1,3,7), from skeleton to skin, examples of skeletal muscle categories depending on location along with the skeletal system.



Fig. 3. Muscular system – layer 1: a. articularis genius muscle; b. levatores costarum longi muscles; c. adductor brevis muscle.



Fig. 4. Muscular and bone system – layer 3: a. clavicle; b. semispinalis capitis muscle; c. vastus medialis muscle



Fig. 5. Muscular and bone system – layer 7: a. Sartorius muscle; b. sternal head of sternocleidomastoid; c. plantaris muscle; d. soleus muscle; e. latissimus dorsi muscle; f. all muscles



The simulation was performed for infantry troops using assault rifles PA Md. 86 and 63/65/90, cartridges loaded with projectiles 5.45x39 mm and with following ballistic performance: bullet mass between 3.43g (53 gr) - 3.62 g (56 gr) type FMJ, velocity: 880 m/s (2900 ft/s) and energy 1.328 J (979 ft.lbf) - 1.402 J (1.034 ft.lbf).

For simulation, the shooting distance was kept constant, 800 m. Figure 6 highlights the behavior of the human body (male) in two distinct cases, for energies developed of 1,328 J and 1,402 J respectively for impact in the area of the lower and upper limbs (constituted virtually with all anatomical systems).



Fig. 6. Simulation of the bullet of 5.45x39 mm impact for 328 J (a., b. - right leg) and 1,402 J (c., d. - left leg; e. - right hand)

The images obtained (Fig. 6 e. and f.) show the fracture of the left lower limb joint and the penetration of the bone in the case of the right upper limb. Future researches actions will be directed to the simulation of burns caused by artillery or aviation ammunition.

3. ABSORBANT LAYER ACHIEVEMENT

3.1 Electrospinning technological process simulation

The dates obtained as a coupled simulation results evidenced the values of the main parameters that should be considered in order to design and develop medical textiles dedicated to these types of applications, as a function of the energy absorbed at the impact of the bullets and anatomic region is between 1200-1500 joules. Moreover, the pression of the circulatory system against the textile support determine, mainly for the absorbent layer, the values imposed for the porosity, pores dimension and braking resistance.



Worldwide, the most used technology to obtain an absorbent layer for this type of wound is represented by the electrospinning process, because it allows:

a. making fibers with very small diameters (in the range of 20 nm - 20 $\mu m)$ - essential for obtaining the specific functionalities of the textile structural network;

b. increasing the surface areas of the constituent entities.

Recent studies [2,3,4] have shown that, for biomedical applications, the adhesion of fibroblast cells and their migration depends on the fiber diameter.

In the case of usage as a composite absorbent layer (e.g., deep burns due to splinters, shingles, etc.) its hardness decreases proportionally with the decrease of the fiber diameter, because the surface area and implicitly the volume increase with the decrease of the diameter [3,4,5].

Despite the apparent simplicity, the mathematical modeling process is complicated, because many factors influence the fiber diameter and implicitly its final morphological structure, such as:

- solution parameters: viscosity, concentration, molecular weight, surface tension, electrical conductivity, electrical dipole moment, dielectric strength;

- process parameters: feed rate, electric field strength, distance between peak and collector, collector type, composition, temperature, relative humidity and air flow.

Empirical models are known that predict the morphology according to a single parameter of the fluid, for instance: fiber diameter and polymer concentration have a linear relationship [1,4], fiber diameter is proportional to the polymer concentration cube, jet radius is inversely proportional to cubic root of the electrical conductivity of the solution, the jet diameter is related to the feed rate (Q) in the form $D \sim Q \ 0.04$, the fiber diameter has an exponential relationship with the viscosity of the solution (exponent 0.41), the jet diameter and material properties are in relation (1) [1,2,5].

$$d = \sqrt[3]{\gamma \epsilon \frac{Q^2}{I^2} \frac{2}{\pi (2Ln(\aleph-3))}}$$
(1)

where: γ = surface tension, ϵ = dielectric constant, Q = debit, I = the current carried by the fiber, \aleph = nozzel diameter, L = the initial length of the jet.

3.2. The control of the textile mesh permeability in order to control of the cellular growths and proliferation and differentiation of the neotissue

It is well known that nanoporous materials can be optimized by controlling pore size and distribution and a variation of the porosity in the range [75; 92] % leads to the variation of the breaking strength limit from 0.475 MPa to 1.886 MPa [1, 6, 7].

Research conducted so far by the Research-Development National Institute for Textiles and Leather specialists on fibrous materials obtained by electrospinning has led to the conclusion that the fibrous network can be characterized by the variable "average coverage", so the number of fibers that converge at a point in the network is defined by the function of probability:

$$f(x) = \begin{cases} e^{-\lambda} \frac{\lambda^n}{x!}, & x \in N = \{0, 1, 2, \dots, n\} \\ 0, & in rest \end{cases}$$
(2)

(Poisson distribution of parameter $\lambda > 0$ for a random variable X, as the limit of the binomial distribution with parameters n and p, for the particular case $n \to \infty$ and $p \to 0$, with $np \to \lambda$).

The average number of contacts per unit length (in the case of 3D) can be calculated depending on the fiber diameter, the porosity of the material, its thickness, the cross-sectional area of the fiber and the number of layers. Experiments continue to determine the logarithmic expression associated with this number, the shape of the fiber cross section and the pore diameter. Also, the



possibility of using fractal theories and the capillary model to describe the distribution of pore size and their length in the fibrous environment is studied, this being represented as a series of orthogonal fiber cells with random volumes.

Research continues in order to determine the permeability of fibrous layers with a random arrangement (depending on the volume of the fiber, its radius and the Knudsen number), especially for low working pressures.

4. CONCLUSIONS

The simulations performed so far by the Research-Development National Institute for Textiles and Leather specialists have allowed the prediction of the phenomena that take place on the human body subject to the action of the 5.45×39 mm infantry armament and that move with subsonic speeds. The control of the morphology and the mechanical behavior of the nanofibers is still at the beginning and the mathematical models obtained so far predicting these parameters are strictly developed for the field of use of the network.

It is necessary to deepen the specific phenomenology of this process for future modeling simulation routes, especially regarding the evolution of nanoscale structures and parameter control.

ACKNOWLEDGEMENTS

This scientific paper is elaborated within the project entitled "Dispozitiv medical inovativ pentru medicina de urgentă și operațională" (CELLMATRIX), financed by UEFISCDI under the contract 496PED/2020.

REFERENCES

[1] J. G. Penn-Barwell, I. D. Sergeant, P.M. Bennett, C.A. Fries, J.M. Kendrew, M. Midwinter, J. Bishop, R.F. Rickard, K. Porter, T. Rowlands, A. Mountain, A. Kay, D. Mortiboy, T. Stevenson, R.W. Myatt, "*Gun-shot injuries in UK military casualties – Features associated with wound severity, Injury*", in Int. J. Care Injured, 2016, pp.242-246.

[2] S. Thomas, M. Uzun," *Testing dressings and wound management materials*", in: Rajendran S, editor. Advanced Textiles for Wound Care (Second Edition): Woodhead Publishing; 2019. p. 23-54

[3] Z. Ma, W. He, T. Yong, S. Ramakrishna, "*Grafting of gelatin on electrospun poly(caprolactone) nanofibers to improve endothelial cell spreading and proliferation and to control cell Orientation*" in Tissue Eng;11(7-8), Jul – Aug 2005, pp. 1149-1158.

[4] A. L. Yarin, S. Koombhongse, D. H. Reneker, "Bending instability in electrospinning of nanofibers" in J Appl Phys; 89(5), Mar 2011, pp. 3018-3026.

[5] J. Nam, Y. Huang, S. Agarwal, J. Lannutti, *"Improved cellular infiltration in electrospun fiber via engineered porosity"* in Tissue Eng vol. 13, 2007, pp 2249-2257.

[6] J. Doshi, D. H. Reneker, *Electrospinning process and applications of electrospun fibers*. J Electrostatics; 35(2-3), Jul – Aug 2005, pp. 151-160.

[7] C. L. Casper, W. Yang, M. C. Farach-Carson, J. F. Rabolt, *"Coating electrospun collagen and gelatin fibers with perlecan domain I for increased growth factor binding"* in Biomacromolecules 8(4), Apr 2007; 1116-1123.



RECOVERY OF REACTIVE COLORANT WITH HYDROTHALCITE AND REUSE FOR PRINTING

LÓPEZ-RODRÍGUEZ Daniel¹, MICÓ-VICENT Barbara², MONTAVA Ignacio¹, BOU-BELDA Eva^{1*}

¹ Universitat Politècnica de València, Departamento de Ingeniería Textil y Papelera, Plaza Ferrándiz y Carbonell s/n, Alcoi, Spain

² Universitat Politècnica de València, Departamento de Ingeniería, Plaza Ferrándiz y Carbonell s/n, Alcoi, Spain

Coresponding autor: Eva BOU-BELDA, Email: evbobel@upv.es

Abstract: In this work, the adsorption capacity of hydrotalcite on the Reactive Yellow 2 textile dye has been verified. Nanoclays are elements with a high capacity for adsorption of dyes and can be reusable as printing pigments. For this, the Lambert-Beer lines of each dye have been previously made. A dye concentration of 1 g/L and a clay concentration of 3 g/L have been used. Then the dye has been introduced into the clay by stirring for 24 hours in 100 mL of solution of the dye, to later filter it and allow to dry. The adsorption of the dye by the nanoclay has been almost absolute, leaving the initial solution very clean, which are excellent results from the point of view of cleaning wastewater. After drying and collecting the clay, a stamping paste in a substrate of PES/CO 50%/50% was made with the hybrid obtained, using a concentration of 1.5 g/kg and 7.5 g/kg. They were then heat-set at 180°C for 30 seconds. Finally, the samples obtained are analyzed on a Minolta CM-3600d reflection spectrophotometer to assess the color achieved. There is a difference in color when comparing the two samples, as expected, the printed sample with higher concentration of hybrid shows a reference to make an assessment of the strength of the color obtained.

Key words: nanoclay, dye recovery, clay pigment, stamping, printing, reactive dye recovery.

1. INTRODUCTION

There are more and more environmental alerts that ask us to intervene to end all the adverse effects caused by pollution. The textile industry stands out notably for the amount of effluent discharges of both organic and non-biodegradable inorganic products that consequently produce high bioaccumulations. Within the textile industry, chemistry is the one that has the greatest environmental impact and is the one with the most chemical activity on the planet [1]. One of the most common discharges is that of colorants, of which we can find concentrations of about 50-1000 ppm, although in some cases they may be lower [2]. One of the recovery methods that are being widely used by researchers are nanoclays since they have a great adsorption capacity, are cheap, reusable and have many possibilities for reuse and recovery of adsorbed dyes.

The natural base of this mineral can be natural or synthetic and in all cases it shows hydrophilic character. Many of these synthetic nanoclays have been improved in the laboratory to increase their adsorptive characteristics [3]. The aim of this study is to confirm the adsorption



capacity of nanoclays, as has been demonstrated in previous works [4]–[9] and also demonstrate experimentally how the hybrid obtained after adsorption can be used to carry out pigment printing on PES/CO polyester cotton textiles. The properties of hydrotalcite make it a very adsorbent element and it also establishes very strong binding forces, allowing the colorant to fix in the clay. In addition, it has been shown that the color resistance to external agents is much higher when it has previously been fixed in a clay, achieving better fastness values.

2. MATERIALS AND METHODS

Reactive Yellow 2 was used as a colorant for this study. As clay we used calcinated hydrotalcite (HC) which was prepared according to Dos Santos R.M.M. [10]. Cotton and polyester plain fabric with 100 g/m² was used to perform the printing process.

Dilutions of each of Reactive Yellow 2 were prepared to obtain the Lambert-Beer line [11]. With this line we can know the concentration of dye after adsorbion for the clay. Table 1 shows the equation of the lines and the regression (R).

Colorant	Equation	\mathbb{R}^2
Reactive Yellow 2	y = 5,8593x + 0,0428	0.9806

The aim is to obtain a clay with a high color intensity for the subsequent realization of the printing paste, for which the adsorption phase is made in the following form. 1000 mL of concentration solution 1 g·L⁻¹ were taken by the dye. We introduce 3 g·L⁻¹ of the clay were introduced and mixture was put under stirring [12], two hours at maximum stirring and then it went to lower speed 600 r.p.m. The solution was then filtered with the clay for 24 hours to separate the clay-dye hybrid from the rest of the solution and measure with the spectrophotometer to calculate the concentration of dye that has not been adsorbed by the clay [13,14]. The hybrid obtained was measured in a Jasco V-670, double beam spectrophotometer between 190-2700 nm and the color differences were calculated. The residual dye solution that has not been adsorbed was also measured with the spectrometer Zuzi model 4251/50 to know the amount of dye that has not been adsorbed.

For the printing procedure, Lutexal CSN was used as a synthetic thickener, STK / 100 center resin and Luprintol SE fixative. Two printing tests were carried out with the hybrid described above, following the following recipes shown in table 2.

m 11	•	D • .•	•
Table	2.	Printing	recipe

Sample n°	Lutexal CSN	Resin STK / 100	Luprintol SE	clay-dye hybrid
1	30 g/Kg	10 g/Kg	10 g/Kg	1,5 g/Kg
2	30 g/Kg	10 g/Kg	10 g/Kg	7,5 g/Kg

The printing was carried out on a PES / CO fabric 50% Cotton and 50% Polyester with 4 passes of a scraper, it was dryed for 15 minutes at 60 °C and then heat-set at 180 °C for 30 seconds on a pressure plate. The samples obtained are analyzed on a Minolta CM-3600d reflection spectrophotometer to assess the color achieved. In the spectrophotometer, 3 measurements were made of each sample and the average was obtained in the maximum reflectance peaks.



3. RESULTS AND DISCUSSSION

The results in Table 3 show how after the clay action the dye concentration have gone from 3 $g \cdot L^{-1}$ to values between 3.08 · 10⁻⁴ and 3.21 · 10⁻⁴ g · L⁻¹. Thus, a good adsorption behavior of the nanoclay is observed as expected and the obtaining of a functional hybrid for the printing phase.

	Sample ref.	HC conc. g·L ⁻¹	Initial conc g·L ⁻¹	Final conc g·L ⁻¹	% absorption
Reactive	1	3	1	$3.08 \cdot 10^{-4}$	99.85
Yellow 2	2	3	1	$3.21 \cdot 10^{-4}$	99.36

Table 3. Difference i	n concentration a	ifter HC absorption
-----------------------	-------------------	---------------------

The stamping carried out according to the method described in the previous section has been a complete success. It can be seen how it has been possible to carry out a printing with the hybrid and the printing paste, leaving the colored fabric in the area of application.

To have a quantitative assessment of the color obtained, the reflectance has been measured and the Kubelka-Munk theory (K/S) has been taken as a reference, which defines two parameters that explain the interaction phenomena of light with matter K: absorption coefficient and S: diffusion coefficient [15]. The results obtained in the visible range of 400-700 nm and under the illuminant D65 show that the maximum reflectance peak is at 400 nm. The reflection results and the calculated mean color strength is expressed in Table 4. Regarding the colorimetric parameters, the L^* a* b* values and the color difference ΔE_{ab}^* are shown in Table 4.

Sample	L*	a*	b*	K/S (400 nm)	$\Delta E_{ab}*$
White	88.6925	-0.5089	2.2863	0.0942	-
Sample 1	87.7489 ± 0.2842	-2.4045 ± 0.1752	10.0666 ± 0.7453	0.1926 ± 0.061	8.0689 ± 0.7287
Sample 2	87.4025 ± 0.2561	-4.5439 ± 0.2137	23.9254 ± 2.9868	0.4552 ± 0.0712	22.0520 ± 2.9829

4. CONCLUSION

In view of the results, we can conclude that the results and interpretations provided in other studies are reproduced in our trials. The HC shows a great capacity for absorption and fixation of the dye within its structure. Furthermore, it has been shown that the obtained hybrid can be used successfully as a textile printing pigment. The samples obtained show good coatings, use and intensity of color, fulfilling the intended objective.

REFERENCES

[1] M. Wakkel, B. Khiari, and F. Zagrouba, "Textile wastewater treatment by agroindustrial waste: Equilibrium modelling, thermodynamics and mass transfer mechanisms of cationic dyes adsorption onto low-cost lignocellulosic adsorbent," J. Taiwan Inst. Chem. Eng., vol. 96, pp. 439-452, Mar. 2019, doi: 10.1016/J.JTICE.2018.12.014.

[2] M. L. Mathew, A. Gopalakrishnan, C. T. Aravindakumar, and U. K. Aravind, "Low cost multilayered green fiber for the treatment of textile industry waste water," J. Hazard. Mater., vol.



365, pp. 297–305, Mar. 2019, doi: 10.1016/J.JHAZMAT.2018.11.014.

[3] F. M. Micó Vicent, Barbara; Martinez Verdu and J. E. Gilabert Perez, "Optimización de la síntesis de nanopigmentos de origen natural para biopolímeros mediante el uso del diseño de experimentos."

[4] M. Ogawa, R. Takee, Y. Okabe, and Y. Seki, "Bio-geo hybrid pigment; clayanthocyanin complex which changes color depending on the atmosphere," *Dye. Pigment.*, vol. 139, pp. 561–565, Apr. 2017, doi: 10.1016/J.DYEPIG.2016.12.054.

[5] F. López Arbeloa, V. Martínez Martínez, J. Bañuelos Prieto, and I. López Arbeloa, "Adsorption of Rhodamine 3B dye on Saponite colloidal particles in aqueous suspensions," *Langmuir*, vol. 18, no. 7, pp. 2658–2664, 2002, doi: 10.1021/la0113163.

[6] M. I. Carretero, M. Pozo, C. Sánchez, F. J. García, J. A. Medina, and J. M. Bernabé, "Comparison of saponite and montmorillonite behaviour during static and stirring maturation with seawater for pelotherapy," *Appl. Clay Sci.*, vol. 36, no. 1–3, pp. 161–173, Apr. 2007, doi: 10.1016/J.CLAY.2006.05.010.

[7] D. Guillermin *et al.*, "New pigments based on carminic acid and smectites: A molecular investigation," *Dye. Pigment.*, vol. 160, pp. 971–982, Jan. 2019, doi: 10.1016/J.DYEPIG.2018.07.021.

[8] E. M. Moujahid, R. Lahkale, H. Ouassif, F. Z. Bouragba, and W. Elhatimi, "New organic dye/anionic clay hybrid pigments: Preparation, optical properties and structural stability," *Dye. Pigment.*, vol. 162, pp. 998–1004, Mar. 2019, doi: 10.1016/J.DYEPIG.2018.11.021.

[9] J.-Z. Yi and L.-M. Zhang, "Removal of methylene blue dye from aqueous solution by adsorption onto sodium humate/polyacrylamide/clay hybrid hydrogels," *Bioresour. Technol.*, vol. 99, no. 7, pp. 2182–2186, May 2008, doi: 10.1016/J.BIORTECH.2007.05.028.

[10] R. M. M. dos Santos *et al.*, "Removal of Acid Green 68:1 from aqueous solutions by calcined and uncalcined layered double hydroxides," *Appl. Clay Sci.*, vol. 80–81, pp. 189–195, 2013, doi: 10.1016/j.clay.2013.04.006.

[11] M. M. F. Silva, M. M. Oliveira, M. C. Avelino, M. G. Fonseca, R. K. S. Almeida, and E. C. Silva Filho, "Adsorption of an industrial anionic dye by modified-KSF-montmorillonite: Evaluation of the kinetic, thermodynamic and equilibrium data," *Chem. Eng. J.*, vol. 203, pp. 259–268, 2012, doi: 10.1016/j.cej.2012.07.009.

[12] Silva, M. M., Oliveira, M. M., Avelino, M. C., Fonseca, M. G., Almeida, R. K., & Silva Filho, E. C. (2012). Adsorption of an industrial anionic dye by modified-KSF- montmorillonite: Evaluation of the kinetic, thermodynamic and equilibrium data. Chemical engineering journal, 203, 259-268.

[13] B. Micó-Vicent, FM Martínez-Verdú "Method for optimising the synthesis of hybrid Nanopigments" (2017) WO2017072382A1.

[14] H. Fischer, L.F. Batenburg, "Coloring Pigment", (2001) patent WO0104216.

[15] H. G. Volz, "Industrial Color Testing Fundamentals and Techniques J. Am. Chem. Soc. 1995, 117, 9618," *J. Am. Chem. Soc.*, vol. 118, no. 43, p. 10678, 1996.



SORPTION OF TEXTILE DYES ON SORBENT MADE FROM COTTON TEXTILE WASTE - EQUILIBRIUM ADSORPTION

MICIC Aleksandra¹, RISTIC Ivanka², DJORDJEVIC Suzana³, RISTIC Nebojsa⁴, DJORDJEVIC Dragan⁵

^{1,5} University of Nis, Faculty of Technology, 16000, Leskovac, Serbia, E-Mail: micic.aleksandra86@gmail.com

^{2,3,4} Academy of Vocational Studies Southern Serbia, Department of Technological Art Studies Leskovac, 16000, Leskovac, Serbia, E-Mail: <u>szn971@yahoo.com</u>

Corresponding author: Djordjevic Dragan, E-mail: drdrag64@yahoo.com

Abstract: The article analyzes the equilibrium adsorption and thermodynamics of the process of removing a disperse dye from water using a sorbent based on cotton textiles waste. Sorbent is made from cotton knitted fabric waste after cutting, during the procedure of clothes production. The loss after washing of raw cotton waste, before transformation into adsorbent, is about 9%. The yield of sorbent made from washed raw cotton waste is just under 40%. The characterization of the new sorbent showed that there is porosity in the structure of the particles and that the chemical composition is dominated by carbon. Equilibrium adsorption was verified by three models, Freundlich and Temkin. The Freundlich equilibrium isotherm showed the best result, i.e. the fitting curve is closest to the experimental points. Since the staining takes place according to the mechanism predicted by the Freundlich model, a monolayer of adsorbed dye molecules is formed in the initial phase of adsorption. At the same time, more layers of adsorbed dye molecules are formed at non-specific sites on the sorbent through self-association of adsorbed dye molecules. The newly-adsorbed dye molecules can bind to the previously adsorbed molecules within the sorbent. The heat of adsorption, determined from Temkin's model, has values that assume that an exothermic process takes place during the adsorption of the dye on the sorbent.

Key words: cotton knitted fabric, cutting, sorbent, disperse dye, equilibrium isotherms, thermodynamics.

1. INTRODUCTION

Disperse dyes are generally non-ionic materials with limited dissolubility in water that have a substantiality to the hydrophobic fiber, for example, nylon, polyester, etc. [1, 2].

These are mostly substituted compounds of the azo, anthraquinone or diphenylamine compounds, which as being non-ionic, do not contain a solubilizing groups, and therefore, the surface active agents are used which finely distribute dye particles in aqueous dispersion. Since disperse dyes are frequently used in dyeing and since they are insoluble in water, special attention should be paid to the potential environmental problem after dyeing, when wastewater is necessary to be disposed of [3].

One method for removal of organic matters – dyes from aqueous solution is the use of solid sorbents. Properties of such substances, which make them useful, are the following: high porosity and specific surface area, as well as suitable places within for the adsorption of the dye molecules.



The most well-known solid sorbents are activated carbons, which are most frequently used in separation and purification processes. Lately, researches have been reinforced to find alternative inexpensive sorbent to replace expensive activated carbons. Industrial agro-cellulosic materials are potential sources for the preparation of cheap sorbents to remove dyeable and undyeable organic substances of the water [4, 5].

This paper deals with the preparation of the inexpensive sorbent from cotton cellulose waste and model adsorption of textile disperse dye on this sorbent from aqueous solution. Textile waste has emerged from the process of cutting in the process of clothing production. The aim is to use well generated textile material waste, transform it into a cheap sorbent and use it for decolorization of water. With the help of equilibrium models of adsorption as well as thermodynamics of the process, the manuscript shows the method of dye binding, the influence of temperature and the feasibility of the process in general.

2. EXPERIMENTAL PART

2.1 Textile waste processing

Textile waste is derived from cotton knitted fabric for the manufacture of women underwear. It is a by-product of cutting out stacked parts of cotton knitted fabric. Cotton waste, which occurs during this cutting process of one set of cutting layers, in a professional workshop, is about 5 kg.

Sorbent, used for adsorption of disperse dye, is a material obtained by chemical and physical modification of cotton cellulose waste. After collecting, the waste was washed, dried and cut into the finest parts. Then, it was treated with a solution of H_2SO_4 (Tehnohemija, Serbia) (ratio 1:5) for 48h at room temperature. Thereafter, heating at 700°C followed for 1.5 h. Then, cooling and fragmentation were performed, rinsing with distilled water and, finally, the neutralization of aqueous solution of sodium bicarbonate (Tehnohemija, Serbia). Final drying (100°C) and grinding were the last stages in the preparation of the inexpensive sorbent from cotton waste.

2.2 Adsorption process

The test of adsorption model was carried out in glass vessels in which sorbent was suspended in a solution of disperse dye (the adsorbate). Glass vessels were placed on a shaker (120 rpm, circular motion) at a temperature of 20, 40 and 60°C and kept for some time. The amount of sorbent was 1 g, in all cases, while the solution in a constant amount of 0.1 dm³ contained a disperse dye concentration in the range of $10-100 \text{ mg/dm}^3$. Processing time, with continuous stirring, was 60 min, the pH of the dye solution was 3, in all cases.

Used dispersed dye belongs to the group of monoazo dyes with two nitro and one acetamide group; there are thiophene and diethylaniline in the strucuture, as well. The sign of the dye is C.I. Disperse Green 9 (DG9), it is not dissolved in water and it is used for dyeing polyester textile material at high temperature and pressure.

2.3 Methods

Determining the absorption of dye solution was done is in a spectrophotometer UV-VIS (*Cary 100 UV-VIS Conc, Varian*) at the maximum of the spectrum, i.e. at 680 nm.

SEM and EDS measurements were performed on TESCAN MIRA3 microscope (The Czech Republic).



The *Freundlich* adsorption isotherm is of empirical character and describes the adsorption on energy heterogeneous surface where the adsorbed molecules interact. This model well describes the multilayer adsorption [5].

The *Freundlich* model is shown by the following equation:

$$\ln(q_{e}) = \ln(K_{F}) + \frac{1}{n_{F}} \cdot \ln(C_{e})$$
(1)

where: $K_F - (mg/g) \cdot (dm^3/mg)^{(1/n)}$ and n_F - constants typical for the proposed system: a sorbent, adsorbate and the solvent; $q_e - (mg/g)$ amount of adsorbed dye (adsorbate) per unit mass of sorbent at equilibrium time; $C_e - (mg/dm^3)$ final (equilibrium) concentration of the dye solution;

The *Temkin* isotherm [6] is represented by the following equation:

 $q_e = B_T \cdot \ln(K_T) + B_T \cdot \ln(C_e)$ (2) where: $K_T - (dm^3/mg)$ and $B_T - Temkin$ constants, the first of which represents the adsorbate - sorbent interactions, and the second is related to the heat of adsorption b_T - (J/mol);

 B_T and b_T are connected as follows:

$$B_T = \frac{R \cdot T}{b_T} \tag{3}$$

where: $R - (8,314 \text{ kJ/mol} \cdot \text{K})$ is a universal gas constant;

3. RESULTS AND DISCUSSION

Since the sorbent is made from cotton textiles waste after cutting out the stacked cutting parts, it is interesting to clarify something about the generated waste. It is calculated the degree of loss from cutting the parts for producing cotton underwear in 3 different widths, whose resulting loss–waste is used to make a sorbent for the adsorption of disperse dye. All cutting images show the expected loss which occurs at fitting of cutting parts. It was found that cutting textile parts with a width of 152 cm causes the least loss of material (14.18%), due to the suitable combination of cut parts compared to other widths (17.57% for 145 cm and 19.54% for 140 cm).

The characterization of the adsorbent was done with the aim of introducing the basic fusionmorphological properties as very important factors for the successful performance of the adsorption process.

The loss after washing of raw cotton waste, before transformation into adsorbent, is about 9% in relation to the pre-washing sample of waste. The yield of the sorption material made from waste cotton textiles is about 40%.

The used powdered sorbent is granular material with a heterogeneous structure of the particles, with indented shape and forms. Inside the particles, there are cracks, cavities and channels, which are the basis of the porosity of the material. Micrograph of Figure 1 shows the appearance of adsorbent particles with a magnification of $\times 200$.

The EDS system enables a quick assessment of the primary composition of the sample. The following chemical elements were detected: C (49.46%), O (48.20%) and Na (2.34%). According to the EDS analysis, carbon is the most abundant, as expected, while the greater presence of oxygen is related to metal oxides (Na), since this metal has been detected, as well as the fact that reaction of adsorbent with the oxygen from the air is possible.





Fig. 1. SEM micrograph of the applied sorbent

The diagram of Figure 2 represents a graphical interpretation of the *Freundlich* linear isotherms for the adsorption of the disperse dye on the sorbent, for a constant quantity of a sorbent (1 g) and the temperature range of $20-60^{\circ}$ C.

On the basis of this diagram and fitting straight lines according to the *Freundlich* model, the suitability of this model to describe the adsorption of dye applied for the sorbent from textiles waste has been evaluated. Since the fitting lines pass very closely to the experimental points, the validity of the *Freunlich* model is verified.

According to the diagram in Figure 2, the fitting curve at 60° C is located in the top part of the diagram in the topmost position, which shows that the adsorption capacity is the greatest at the highest temperature. Slightly lower position has the fitting curve at 40° C and much lower position was observed at temperatures more than 20° C.

If it is assumed that the dyeing is performed by a mechanism intended by the *Freundlich* model, in the initial phase of adsorption, a monolayer from the adsorbed dye molecule is formed, after which immediately follows the formation of multilayer from adsorbed dye molecules at nonspecific sites in the sorbent via self-association. The newly-adsorbed dye molecules can bind to the previously adsorbed molecules within the sorbent. There are also aggregated dye molecules from the solution, which can also be adsorbed. Dye aggregation within the sorbent and thus the formation of a multilayer of adsorbed dye molecules seems likely given the fact that relatively long and flat disperse dye molecules tend to self-combine in solution via π - π interaction (noncovalent interaction between aromatic rings) between adjacent dye molecules [5].

The coefficients of determination for this isotherm are in the range of 0.970–0.983, which means that *Freundlich* model can be used for a good enough description of DG9 dye adsorption to the sorbent.

 K_F , one of the *Freundlich* constants, is used as a relative measure of adsorption capacity. A higher value ($K_F = 0.18-0.55$, depending on the temperature) indicates a higher adsorption capacity.

Other *Freundlich* constant, n, is an empirical parameter that changes with the degree of heterogeneity, indicating the degree of non-linearity between the receiving capacity of the dye and



the concentration of non-adsorbed dye, and relates to the distribution of bound ions on the surface of the sorbent. In general, 1/n<1 shows that the adsorbate is sufficiently adsorbed on the adsorbent, the adsorption capacity increases, new positions for adsorption appear and the higher the value of *n*, the stronger the adsorption intensity. The results confirm that n>1, i.e. 1.23-1.38>1, or 1/n<1, i.e. 1/(1.23-1.38)<1.



Fig. 2. Graphic representation of the Freundlich model

Figure 3 provides a graphical dependence of the variables from the *Temkin* isothermal equations for temperature range $20-60^{\circ}$ C. According to the appearance of curves after the fitting, it is noticeable a little weaker coverage of experimental points on the diagram, and, consequently, a somewhat worse result in comparison to the *Freundlich* model.



Fig. 3. Graphic representation of the Temkin model

The coefficient of determination, R^2 , obtained from the *Tempkin* model (0.960–0.964), has smaller values in relation to the previous isotherm which partly diminish the applicability of the *Tempkin* model for the specific case of adsorption.

Values of the *Temkin* constant B_T , associated with the heat of dye adsorption on a sorbent, is in the range 1.48–1.99, while the constant K_T has a values 0,27–0,63 dm³/mg. Heat of adsorption, b_T ,



determined from the *Temkin* model, has values from $-1.24 \div -1.87$ kJ/mol, which implies the exothermic process during adsorption of DG9 onto the sorbent.

This adsorption energy will be the sum of all individual adsorptions that occur at different places on the sorbent surface. In addition to the structure of the dye and the substrate, the value of the heat of adsorption is greatly influenced by the presence of an additive or impurity which directly impacts the ability of interaction and binding, both by physical or chemical bonds. Since the heat of adsorption is small and negative, the interaction of dye and sorbent is mainly accompanied by reversible physical adsorption which are supported by weak electrostatic forces (dipolar bonds, *Van der Waals* forces or hydrogen bonds) [7].

4. CONCLUSIONS

By the process of thermochemical conversion, the collected cotton waste from the cutting of cutting deposits was, with the help of sulfuric acid, as an activating agent, converted into a powder adsorbent. The sorbent has a porous structure, and the primary composition is dominated by carbon. Thus produced inexpensive sorbent was used to study the adsorption of disperse dye to the sorbent using several significant models for equilibrium adsorption.

The *Freundlich* equilibrium model best describes the process of adsorption of the disperse dye onto the sorbent from cotton textiles waste after cutting. From the *Temkin* model, the calculated heat of adsorption is small and negative, which means that the interaction of dye and sorbent is mainly accompanied by reversible physical adsorption supported by weak electrostatic forces.

Characterizing other, similar in nature, solid waste materials, can help to elucidate the adsorbate–sorbent interactions, which leads to the optimization and greater efficiency of the adsorption process, as an environmentally friendly procedure.

REFERENCES

[1] A. M. Al-Etaibi, M. A. El-Apasery, "Dyeing Performance of Disperse Dyes on Polyester Fabrics Using Eco-Friendly Carrier and Their Antioxidant and Anticancer Activities," Int. J. Environ. Res., Public Health, vol. 16, pp. 4603, 2019.

[2] L. Yan, L. Hou, S. Sun, P. Wu, "Dynamic Diffusion of Disperse Dye in a Polyethylene Terephthalate Film from an Infrared Spectroscopic Perspective," Ind. Eng. Chem. Res., vol. 59, pp. 7398–7404, 2020.

[3] S. Zhao, Z. Gao, G. Jiang, J. Wang, X. Miao, A. Wan, "Effect of the dyeing process on thermal and dyeing properties of poly(butylene terephthalate) fibers," Text. Res. J., vol. 91, pp. 580-588, 2021.

[4] K-W. Jung, B. H. Choi, M-J. Hwang, J-W. Choi, S.-H. Lee, J.-S. Chang, K.-H. Ahn, "Adsorptive removal of anionic azo dye from aqueous solution using activated carbon derived from extracted coffee residues," J. Cleaner Prod., vol. 166, pp. 360-368, 2017.

[5] M. Li, J. Lu, X. Li, M. Ge, Y. Li, "Removal of disperse dye from alcoholysis products of waste PET fabrics by nitric acid-modified activated carbon as an adsorbent: Kinetic and thermodynamic studies," Text. Res. J., vol. 90, 2058–2069, 2020.

[6] P. Pankaj, R. Sharma, S. K. Verma, "Equilibrium, kinetic and mechanism studies on adsorption of textile disperse dye from aqueous solution onto TiO_2 in the presence of ultrasound and rare earth ions," Materials Today: Proceedings, Article in press, 2021.

[7] S. Puttamat, V. Pavarajarn, "Adsorption Study for Removal of Mn (II) Ion in Aqueous Solution by Hydrated Ferric (III) Oxides," Int. J. Chem. Eng., vol. 7, pp. 239-240, Aug. 2016.



LABORATORY STUDIES ON NATURAL FABRICS TEXTILE PRINTING WITH SPIRULINA PLATENSIS SOURCED PHYCOCYANIN

MOLDOVAN Simona¹, FRANCO Esther¹, PASCUAL Javier¹, FERRANDIZ Marcela¹, BONET-ARACIL Marilés², GISBERT-PAYÁ Jaime², BOU-BELDA Eva²

¹Aitex, Technological Research Institute on Textile. Plaza Emilio Sala nº 1, 03801. Alcoy, Spain

²Universitat Politècnica de València, Textile and Paper Department, Plaza Ferrándiz y Carbonell s/n, 03801, Alcoy, Spain.

Corresponding author: Bonet-Aracil, M. Plaza Ferrándiz y Carbonell s/n, 03801, Alcoy, Spain. E-mail: <u>maboar@txp.upv.es</u>

Abstract: Naturally sourced elements represent a sustainable alternative solution for the textile finishing industry, in the context of intense use of contamined, fossil-based raw materials. This study focuses on the exploration of an alternative naturally sourced blue colorant matter solution, by validating the possibility of the employment of Spirulina platensis sourced phycocyanin in natural fabrics coloration through the pigment printing technique. The experiments involved the laboratory-scale exploratory studies of the application of this blue colorant matter on cotton and wool textile substrates, using commercial synthetic printing mother paste. The influence of five different mordants(Cream of tartar, Alum, Tartaric acid, Tanic acid and Aluminum triformate), though pre-mordanting treatment and the printing mother paste were analyzed by measuring the chromatic coordinates and calculating the color strength (K/S), together with the measurement of VIS absorbance spectrum. The color characterization revealed good compatibility of wool -phycocyanin – synthetic paste. Results on cotton were not as good as expected. They conclusions evidence reduced affinity between the fibres and the phycocyanin. In terms of color improvements, Tannic acid revealed the most promising results, for both cotton and wool experiments. The validation of the finishing process was obtained through a fair behavior in laundering fastness, showing low response when analyzed against light degrading external factors.

Key words: phycocyanin, blue, microalgae, wool, cotton, pigment printing

1. INTRODUCTION

The negative environmental impacts, complemented with hazardous health issues, as toxicity, carcinogenic character, allergies producers, of the synthetic dyes have lead to search forsustainable solutions [1][2][3]. Various experiments of textile printing on natural fibers, using natural vegetal colorant matter, have been performed. Due to the fact that natural dyes do not present fiber affinity, as their synthetic alternatives, various mordants were employed for this purpose, as aluminum, stannous chloride, magnesium sul phate, tannic acid, myrobalan, etc.[4][3][5].

One emerging sector able to providea sustainable solution to several industries is represented bymicroalgae and macroalgae exploitation, owning developed cultivation technologies [6], and various commercial application sectors [7].Similar to plants, algae produce highly bioactive compounds, primary and secondary metabolites, as lipids, proteins, carotenoids, phycobiliproteins, phenolic compounds, with valuable prospects for industrial applications [8].The blue-green



microalga *Spirulina platensis* is a source of phycocyanin, a naturally occurring blue colorant matter, representing a secondary metabolite. Phycocyanin is a water-soluble protein, pertaining to the phycobiliproteins family, characterized by covalent attachments of open chain tetrapyrroles. This protein may reach up to 20% of the weight of the cell protein, being used already in various industries as food, cosmetics, the therapeutic agent in oxidative diseases treatment, and biomedical research as a fluorescent marker, etc.[9]. Nevertheless, the potential of this colorant matter is not mentioned in the research destined to the textile industry, but very few applications of natural colorants are considered for pigment printing [10]. The aim of the present work is to evaluate the viability of the textile application of a algal sourced natural alternative for the blue color, phycocyanin, on natural fibers of cotton and wool, through the printingfinishing process. Synthetic commercial printing paste was tested for process efficiency identification.

2. MATERIALS AND METHODS

2.1 Materials

Commercial undyed, bleached 200 g/m² cotton fabrics, EMPA221 (supplied by Intexter UPC, Spain) and bleached wool fabric (supplied by James Heal, England), both complying with ISO 105-F01 were used as printing textile substrates. 5 mordants were applied for the study of their influence in color uptake: Cream of tartar (6% w.o.f) and Alum (10% w.o.f) (supplied Gran velada), Tartaric acid (6% w.o.f), Tannic acid (2% w.o.f) and Aluminum triformate (10% w.o.f) (supplied Sigma Aldrich). Phycocyanin-rich liquid extract, obtained from *Spirulina platensis*, (procured from Banco Español de Algas (BEA), Spain) was employed as a natural dye in all the experiments.

2.2 Methods

2.2.1 Phycocyanin quantification

The phycocyanin-rich liquid solution was subjected to UV VIS spectral absorbance (UV, Thermoscientific Evolution 60S) and the amount of mg/ml of phycocyanin in solution was obtained based on the equation established by Bennett and Bogorad,(1973) [11]with extinction coefficients defined by Bryant et al (1979)[12].

2.2.2Pretreatment of fabrics and pigment printing

Wool bleaching involved the treatment of the fabric for 60 minutes at 55° C, in a Rb=1/15 solution of decalcified water containing 2g/L of Pyrophosphate tetrasodium (Sigma Aldrich), 10 -20 ml/L of Hydrogen peroxide 30% (Fisher Scientific), 2g/L of nonionic Clarite detergent (Huntsman), adjusting the pH at 8,5-9 with Ammonia 25% (PanReac). The fabric is further rinsed and dried at ambient temperature. The bleached wool and the cotton were subjected to a mordant solution, according to the concentrations defined in section 2.1., for 45 minutes at a temperature of 85°C.

A conventional commercial printing stock paste was prepared with the following ingredients, supplied from Color Center, Spain: Color center BC (binder),Color center MC-LF (fixer), Color center H35 (thickener), with 2% dye concentration proportional to the printing paste weight.The binder and fixer were mixed through intense blending, together with the corresponding quantity of water, and the phycocyanin rich solution. Further , the thickener was gradually added, forming the printing paste.The curing step was performed during 3 minutes at 150°C, and the drying step involved ventilation at 40°C, in a laboratory-type drying oven (by Memmert, Germany).Conventional manual printing screen and scraper were used for the application of the colored printing paste onthe fabrics. The scraper was passed over the fabric with three repetitions, applying uniform pressure.



2.2.3 Printed fabrics characterization and analysis

The chromatic coordinates of the printed samples were measured with Datacolor DC 650 (Datacolor, Spain) according to the indications defined in standard UNE-EN ISO 105-J01:2000.

The reflectance spectrum was determined via measurements with UV-VIS spectrophotometer Lambda 950 (Perkin Elmer, Spain). Color relative dye uptake, K/S was calculated with the Kubelka-Munk equation, given in Equation 1.

$$K/S = ((1 - R^2))/2R$$

(1)

where K is the absorption coefficient, S is the scattering coefficient and R reflectance at maximum wavelength [13].

Color fastnessfor laundering and light degradation was determined following the European standardized protocols. As UNE-EN ISO 105-C06:2010. (laundering fastness), involving the immersion of the tested fabric into a canister containing 150 mL of water and 0,6 gr of standardized detergent, together with 10 steel balls, during a washing cycle of 45 minutes at 25°C into a Gyrowash apparatus (James Heal, UK). And UNE-EN ISO B02:2014(light fastness) where samples are pretreated by spraying with water, before the subjection to Xenon arc fading lamp for 16 hours.

3. RESULTS AND DISCUSSIONS

Mild blue tones were obtained through the printing process involving phycocyanin as colorant matter embedded in synthetic printing paste.

In order to analyze the influence of mordants on the resulting blueish printed cotton, it can be identified, as per ΔE values, in Table 1, that a slight total color difference, is facilitated by the use of mordants, in comparison with the printed non-mordanted cotton fabric. The most accentuated color difference, is obtained by the use of Tannic acid, most probably due to the additional influence of the intrinsic natural color of the auxiliary. Nevertheless, this is followed by Alum, justified by a bonding creation between the textile substrate and colorant. The bluer shade is conferred by the Tannic acid (Δb), followed by less intense bluer shades, attributed to the use of Alum, Cream of tartar, Tartaric acid, and Aluminium triformiate.

		Color coordinates and color strength							
Sample	L	a	B	ΔL	Δa	Δb	ΔΕ	K/S	
No mordant	89,56	-1,12	2,42	-	-	-	-	37,27	
Cream of tartar	89,47	-1,28	2,68	0,09	0,16	-0,26	0,32	36,10	
Alum	91,31	-0,7	2,93	-1,75	-0,42	-0,51	1,87	34,85	
Tartaric acid	90,53	-1,03	2,66	-0,97	-0,09	-0,24	1,00	36,57	
Aluminium Triformate	90,11	-0,51	2,61	-0,55	-0,61	-0,19	0,84	35,42	
Tannic acid	88,64	-0,37	4,03	0,92	-0,75	-1,61	2,00	23,70	

 Table 1:Color coordinates of phycocyanin printed cotton fabrics with synthetic paste

On the other hand, the color characterization assumptions are supported by the analysis of VIS spectrum, in terms of color absorbance, as presented in Figure 1. Therefore, the VIS spectrum, reveals color difference between the analyzed fabrics, and confirms the slight influence of the used



mordants in final color intensity, thus positive impact in color absorbance in this finishing process employed.



Fig. 1. VIS spectrum of phycocyanin printed cotton (synthetic paste), and influence of various mordants

The color analysis of the wool printed fabrics focuses on the differences between the nonmordanted printed fabric and the ones treated with various mordants. According to **Error! Reference source not found.**Table 2, significant color difference is observed with respect to the non-mordanted fabric, to all the applied mordants. The general conclusion is that the color lightness difference (ΔL) shows darker colors with the use of Tannic acid, followed by far by the rest of mordants. In the same time, all finished wool textiles show a yellower (Δb) tone, when compared with the non-mordanted one. Considering the whole sets of experiments, wool printing process revealed more intense blue shades, confirming higher affinity between wool, phycocyanin and printing paste.

Somula	Color coordinates and color strength								
Sample	L	а	b	ΔL	Δa	Δb	ΔΕ	K/S	
No mordant	82,01	-2,98	10,55	-	-	-	-	23,98	
Cream of tartar	82,59	-7,7	2,83	-0,58	4,72	7,72	9,07	28,06	
Alum	82,05	-5,62	2,73	-0,04	2,64	7,82	8,25	27,82	
Aluminium Triformate	81,15	-7,95	2,69	0,86	4,97	7,86	9,34	28,16	
Tartaric acid	81,5	-6,98	2,82	0,51	4	7,73	8,72	27,59	
Tannic acid	74,06	-3,43	6,68	7,95	0,45	3,87	8,85	26,14	

Table2. Color coordinates of phycocyanin printed wool fabrics with synthetic paste

The similarity in color shades between all the printed wool fabrics, prepared in this study, is clearly reflected in Fig 2., with the exception of the case where Tannic acid is employed, justified by higher color absorbance and also the intrinsic color shade of the mordant.



Fig. 2. VIS spectrum of phycocyanin printed wool (synthetic paste), and influence of various mordants



A comparative analysis, in terms of influence over color strength, of pre-mordanting of cotton andwool textile substrates has been performed on the printed fabrics. The K/S values in cotton and wool dyeing were calculated for determining the influence of the pre-mordanting process. In the case of the cotton experiments, it can be observed slightly higher values for the not mordanted reference case, which indicates not significant benefits in pre-mordanting the fabric substrate when pigment printing with phycocyanin. Nevertheless, in the case of wool applications, overall the results are confirming to the expected output of the printing process and application of pre-mordanting of wool textile substrates is beneficial with respect to improved color strength values.

Laundering and light fastness tests were performed on the cotton and wool fabrics (Table 3), revealing fair behavior in terms of discharge, at laundering tests and very poor behavior against light agents. Improvements in light fastness, in printing of cotton, were not observed, due to the lack of effect of the pre-mordanting treatment.

Cotton Samples			Light				
Cotton Samples	Wool	Acrilic	Polyester	Poliamide	Cotton	Acetate	fastness
No mordant	4-5	4-5	4-5	4-5	4-5	4-5	3
Cream of tartar	4-5	4-5	4-5	4-5	4-5	4-5	1
Alum	4-5	4-5	4-5	4-5	4-5	4-5	1
Tartaric acid	4-5	4-5	4-5	4-5	4-5	4-5	1
Aluminium Triformate	4-5	4-5	4-5	4-5	4-5	4-5	1
Tannic acid	4-5	4-5	4-5	4-5	4-5	4-5	1
Wool samples							
No mordant	4-5	4-5	4-5	4-5	4-5	4-5	1
Cream of tartar	4-5	4-5	4-5	4-5	4-5	4-5	1
Alum	4-5	4-5	4-5	4-5	4-5	4-5	1
Tartaric acid	4-5	4-5	4-5	4-5	4-5	4-5	1
Tannic acid	4-5	4-5	4-5	4-5	4-5	4-5	1

Table3. Fastness results of mordanted cotton printed with synthetic paste

On the other hand, wool light fastness did not show improvements, with the application of various mordants. This could be explained also by the liquid state of the phycocyanin extract, generating the need for experimenting with powder phycocyanin.

5. CONCLUSIONS

Laboratory experiments based on printing of natural fibers, as cotton and wool, with algae sourced blue colorant material, showed good manipulation properties, in the exploratory experimental set of application with synthetic printing paste.Light blue shades of printed fabrics were obtained, in all experimental cases, and this is due to the nature of the phycocyanin extract, tailored liquid extract, revealing the need of further experimenting with commercial phycocyanin.Color strength, K/S, values indicated no benefits when cotton fabrics were pre-mordanted, and revealing the contrary conclusions for the wool case, with validated improvements when the textile substrates were treated.The individual analysis of each experimental case revealed slight influence of the used mordants in final color intensity, when referring to cotton fabrics printed with synthetic printing paste. These finished fabrics showed fair behavior in terms of discharge, in



laundering tests.Printed wool with synthetic printing paste, definitely revealed more intense blue colors, highlighting a better compatibility between the fabric, printing paste and phycocyanin. Even, mordant application presented differences when used Cream of tartar and Alum, and specifically the Tannic acid. The fair behavior at laundering fastness appears, and poor behavior against light agents.

The results of the experiments developed in this study, represent the first step in demonstrating the behavioral characteristics of this sustainable colorant matter, validating the possibility of use of this chromoprotein in the textile industry. Considering the liquid state of the phycocyanin extract, and the tailored production, it is fair to assume the need of a more general experimenting with commercial phycocyanin, in powder state.

ACKNOWLEDGEMENTS

The authors acknowledge the support of this study to research projects "GREENCOLOR" reference IMDEEA/2017/37 from the Spanish Valencian Institute of Competitive companies, for the project entitled Study on the application of natural dyes for dyeing and printing textiles.

"SEACOLORS". grant number LIFE13 ENV/ES/000445, entitled Demonstration of new natural dyes from algae as substitution of synthetic dyes actually used by textile industries", within the LIFE 2013 "Environment 300 Policy and Governance project application".

REFERENCES

[1]. M. Shahid, Shahid-Ul-Islam, and F. Mohammad, "Recent advancements in natural dye applications: A review," J. Clean. Prod., vol. 53, pp. 310–331, 2013.

[2]. E. Commission, "The EU Blue Economy Report 2019," Luxembourg, 2019.

[3]. S. N. Chattopadhyay, N. C. Pan, and A. Khan, "*Printing of jute fabric with natural dyes extracted from manjistha, annatto and ratanjot*," Indian J. Fibre Text. Res., vol. 43, no. 3, pp. 352–356, 2018.

[4]. M. D. Teli, J. Sheikh, and P. Shastrakar, "*Exploratory Investigation of Chitosan as Mordant for Eco-Friendly Antibacterial Printing of Cotton with Natural Dyes*," J. Text., vol. 2013, pp. 1–6, 2013.

[5]. M. Rekaby, A. A. Salem, and S. H. Nassar, "*Eco-friendly printing of natural fabrics using natural dyes from alkanet and rhubarb*," J. Text. Inst., vol. 100, no. 6, pp. 486–495, 2009.

[6]. R. Slade and A. Bauen, "Micro-algae cultivation for biofuels: Cost, energy balance, environmental impacts and future prospects," Biomass and Bioenergy, vol. 53, pp. 29–38, 2013.

[7]. S. Mobin and F. Alam, "Some Promising Microalgal Species for Commercial Applications: A review," in Energy Procedia, 2017, vol. 110, pp. 510–517.

[8]. M. Rico, A. G. González, M. Santana-Casiano, M. González-Dávila, N. Pérez-Almeida, and M. S. de Tangil, "*Production of primary and secondary metabolites using algae*," in Prospects and Challenges in Algal Biotechnology, Springer Singapore, 2017, pp. 311–326.

[9]. F. S. Antelo, J. A. V. Costa, and S. J. Kalil, "Thermal degradation kinetics of the phycocyanin from Spirulina platensis," Biochem. Eng. J., vol. 41, no. 1, pp. 43–47, 2008.

[10]. S. Moldovan, M. Ferrandiz, E. Franco, E. Mira, L. Capablanca, and M. Bonet, "*Printing of cotton with eco-friendly, red algal pigment from Gracilaria sp.*," IOP Conf. Ser. Mater. Sci. Eng., vol. 254, no. 19, 2017.

[11]. A. Bennett and L. Bogorad, "Complementary chromatic adaptation in a filamentous bluegreen alga," J. Cell Biol., vol. 58, no. 2, pp. 419–435, 1973.

[12]. D. A. Bryant, G. Guglielmi, N. T. de Marsac, A. M. Castets, and G. Cohen-Bazire, "*The structure of cyanobacterial phycobilisomes: a model*," Arch. Microbiol., vol. 123, no. 2, pp. 113–127, 1979.

[13]. Kubelka and Munk, "The Kubelka-Munk theory of reflectance." 1931.



OPTIMIZATION OF PROCESS CONDITIONS OF SILK FABRIC DYEING WITH GALINSOGA PARVIFLORA LEAF EXTRACT FOR ANTIBACTERIAL APPLICATION.

MUSINGUZI Alex^{1, 2}, MWASIAGI, J. Igadwa¹, NIBIKORA Ildephonse² NZILA Charles¹

¹Moi University, Faculty of Engineering, Department of Manufacturing, Industrial and Textile Engineering, P.O. Box 3900-30100, Eldoret, Kenya.

² Busitema University, Faculty of Engineering, Department of Polymer, Textile and Industrialisation Engineering, P.O. Box 236, Tororo, Uganda.

Corresponding author: Musinguzi, Alex, E-mail: musinguzialexb1@gmail.com

Abstract: Silk being one of the organic natural fibers, is susceptible to microorganisms attack thus leading to loss of physical aesthetic and mechanical properties. The present study was focused on optimizing the dyeing process parameters (Extract concentration and dyeing temperature) of dyestuff extracts from the Galinsoga Parviflora plant and analyzing the antibacterial activity of the dyed silk fabric. The Pad-dry method was used for the application of herbal dye extract onto selected silk fabric and the AATCC 100:2019 test method was used in assessing the treated fabric quantitatively against Staphylococcus Aureus and Pseudomonas Aeruginosa bacterial strains. Dyeing conditions obtained using Central Composite Design (CCD) indicated that dye concentration and temperature of 39.14 percent and 70°C respectively could be deemed as optimum. Also, the Analysis of Variance (ANOVA) results showed that extract concentration has a statistically significant effect on bacterial count whereas the effect of temperature was not so much influential. Silk fabric dyed with optimized values demonstrated a 99.33% and 99.15% reduction in the bacterial count against Staphylococcus Aureus and Pseudomonas Aeruginosa bacterial strains respectively. Also, its fastness properties to light, washing, and Rubbing ranged from very good to excellent (4-5 to 5). Thus, in general, this research confirmed that Galinsoga Parviflora plant leaves, abundantly available in most parts of Uganda can be used as an antibacterial finish on silk fabric for improved bacterial resistance.

Keywords: Antibacterial activity, Central Composite Design, Silk fabrics, leaf extract, Fastness properties.

1. INTRODUCTION

Textile fibers and fabrics are well known to be excellent media for the generation and propagation of various micro-organisms which cause adverse fiber properties that include; unpleasant odor, staining, and health-related infections to the users [1]. In a bid to improve the functional ability of the clothing materials, several antibacterial agents can be applied [2],[3]. Recently, there has been an increase in the use of natural-based medicinal plants as antibacterial agents due to their eco-friendly nature and non-allergic properties [4],[5]. Galinsoga Parviflora is a well-known traditional plant belonging to the Asteraceae family, good at treating wounds and beetle bites, and grows on fertile soils which are uncultivated. It also contains several phytochemical and pharmacological properties which help it to resist microbes [6]. Apart from antibacterial activities, medicinal plant extracts can



also be used to provide several colours to the fabrics although their colourfastness properties are relatively low but improve significantly when applied using a cross-linking agent [7]. However to effectively achieve the desired satisfaction of these natural dyes, dye application methods vary which can be optimized using different techniques [8]. Therefore, the present study was aimed at extracting the dye from *Galinsoga Parviflora* plant leaves and optimize dyeing parameters using Central Composite Design, a model fitting tool of Surface Response Methodology on silk fabrics for antibacterial applications.

2. MATERIALS AND METHODS

2.1 Materials

Pre-treated woven silk fabric was bought from Kawanda Silk Research Center, Kampala-Uganda. Mature plant leaves of *Galinsoga Parviflora* were collected from the wild in Biharwe, Mbarara District – Uganda. Sodium Hydroxide, Distilled water, Pestal, and a motor, Digital measuring balance, and Whatman No. 1 filter papers all were bought from INDO Kenya Enterprises, Eldoret-Kenya.

2.2 Methods

2.2.1 Preparation of medicinal dye extract

The dried plant leaves of *Galinsoga Parviflora* were grounded into a moderately coarse powder, weighed, and subjected to an aqueous extraction process keeping material to liquor ratio at 15:250 w/v [9]. After 8 hours on a shaker, the extract was filtered and stored at 4° C degree in an airtight container.

2.2.2 Microorganisms and culture condition

ATCC Gram-positive and Gram-negative pathogenic bacteria viz. *Staphylococcus aureus* and *Pseudomonas aueruginosa* were recovered from the storage media following the Standard Operating Procedures and maintained on Muller Hinton (MH) medium.

2.2.3 Dyeing of the selected fabrics and testing for their antibacterial efficacy

After dye extraction, silk fabric samples were dipped in the bath with material to liquor ratio of 1:40, 3g/l concentration of Alum at 70°C for 30 minutes. Thereafter, the excess dyes were removed by padding mangles and dried under the shade [10]. To optimize the dyeing process, extract concentration and dyeing temperature were done at 15, 25, and 35%, and 60, 70, and 80°C respectively. These values were considered basing on the literature survey and preliminary trials. The dyed fabric samples were tested for bacterial efficacy against selected bacterial strains. The combination that yielded the lowest bacterial count was selected as the optimum condition.

2.2.4 Response Surface Methodology

Basing on the bacterial resistance properties demonstrated by dyed silk fabric samples, dyeing conditions for *Galinsoga Parviflora* plant extract were optimized using Central Composite Design (CCD) of Response Surface Methodology [11]. The experimental variables were extract concentration and dyeing temperature. Their coded and actual levels are stated in Table 1.



Table 1. Experimental variables and their levels							
Variables	Levels						
	-α	-1	0	1	α		
Concentration (%)	10.86	15	25	35	39.14		
Temperature (⁰ C)	55.86	60	70	80	84.4		

Then the response variable was the bacterial count values obtained from testing dyed silk fabrics against selected bacterial strains. All the design and analysis of experiments were performed using the Design Expert 7.0 software package [12].

2.2.5 Statistical Analysis

With the help of ANOVA, the statistical significance of the regression coefficients and adequacy of the developed model was checked. Response surface plots were drawn to analyze the interaction among the different independent process factors and their effect on the bacterial count.

2.2.6 Assessment of Antibacterial resistance and fastness properties of dyed fabric with optmized values.

As per AATCC Test Method-100:2019, dyed and undyed (Control), silk fabrics were tested for antibacterial activity against Gram-positive and Gram-negative bacterial strains. After inoculation and incubation, the microbial inhibition was calculated as a percentage reduction in the number of Colony Forming Units (CFU) with respect to untreated control samples following the formula below;

R = [(B-A)/B]*100. Where; R- Percentage reduction in Microbial colonies, A – CFU/ml for the treated fabric samples after 24hrs incubation, B – CFU/ml for the untreated fabric samples after 24hrs incubation under the same conditions.

For fastness properties, the dyed silk fabrics were analyzed following the ISO standard methods viz ISO 105-X12:2016, ISO 105-E04:2013, ISO 105-B02:2014, and ISO 105-C10:2006 for rubbing, perspiration, light, and wash fastness respectively.

3. RESULTS AND DISCUSSION

3.1. Response Surface Methodology

The bacterial count values of silk fabric samples dyed with *Galinsoga Parviflora* extracts at different dyeing conditions are illustrated in Table 2. An increase in extract concentration and dyeing temperature results in a decrease in bacterial count. However, the reduction in bacterial count majorly depended on the bacterial strains used and the concentration applied. This may have been attributed to the differences in the bacterial structures [3].

Table 2. Bacterial counts at varying dyeing parameters					
No.of Runs	Conc. Of Extract (%)	Temp. (^O C)	Bacterial Count (CFU/ml) Silk fabric		
			Staphylococcus aureus	Pseudomonas Aeruginosa	
1	25	70	$5.20 imes 10^4$	9.60×10^{4}	
2	25	70	$5.36 imes10^4$	$9.20 imes 10^4$	
3	15	60	$9.52 imes 10^4$	$2.35 imes 10^5$	
4	35	80	9.60×10^{3}	$3.68 imes 10^4$	
5	10.86	70	$1.81 imes 10^5$	3.12×10^5	
6	39.14	70	00	$4.80 imes 10^3$	
7	25	84.14	$4.16 imes 10^4$	$8.16 imes10^4$	
		61			



8	25	70	$4.96 imes 10^4$	$9.76 imes10^4$
9	25	55.86	$6.24 imes10^4$	$1.00 imes 10^5$
10	25	70	$5.12 imes 10^4$	$9.36 imes 10^4$
11	25	70	$5.04 imes10^4$	$9.68 imes 10^4$
12	15	80	$8.24 imes10^4$	$2.08 imes 10^5$
13	35	60	$1.12 imes 10^4$	$6.24 imes 10^4$

3.2 Analysis of Variance (ANOVA)

The ANOVA results for bacterial count values of treated silk against *Staphylococcus aureus* and *Pseudomonas Aeruginosa* are illustrated in Table 3. It can be seen that both models (' Y_{ss} ' & ' Y_{sp} ') are significant having p-values = 0.001 and 0.000. Also, the effect of extract concentration on bacterial count is statistically significant with a p-value of 0.000 on both models. Therefore, it can be deduced that a change in extract concentration has a significant effect on dyeing silk fabrics for bacterial resistance whereas, the dyeing temperature did not significantly affect the bacterial count of the dyed fabrics.

Table 3. ANOVA for the bacterial count at different dyeing variables on silk

		Staphylococcus aureus		Pseudomonas Aeruginosa	
Source	DF	F-Value	P-Value	F-Value	P-Value
Model	5	14.85	0.001	87.40	0.000
А	1	67.88	0.000	390.23	0.000
В	1	0.77	0.410	3.91	0.088
A^2	1	4.43	0.073	42.32	0.000
\mathbf{B}^2	1	0.57	0.474	0.01	0.913
A*B	1	0.10	0.761	0.00	0.978
Error	7				
Lack-of-Fit	3				
Pure Error	4				
Total	12				

The second-order regression equations (1 & 2) were generated to model the relationship between bacterial count (Response ' Y_{ss} ' & ' Y_{sp} ') and dyeing variables of extract concentration (A) and Temperature (B) for silk fabric samples. The R² and adjusted R² were 91.39% and 85.23% respectively for the Y_{ss} model and then for the Y_{sp} model, R² and adjusted R² were 98.42% and 97.30% respectively. This implies that 91.39% and 98.42% variations in the data sets can be explained by the models. For the unseen data sets, the adjusted R² for each model is 85.23% and 97.30% respectively. The interaction effects of the dyeing variables are represented on the three-dimensional graphs, response surface plots in Fig. 1.

$$Y_{ss} = 106932 - 14176 A + 5864 B + 141.2 A^2 - 50.8 B^2 + 28.0 AB$$
(1)

$$Y_{sp} = 654883 - 27081 \text{ A} - 1865 \text{ B} + 344.0 \text{ A}^2 + 6.0 \text{ B}^2 + 2.0 \text{ AB}$$
(2)

Whereby;

Y_{ss} & Y_{sp} are bacterial count of dyed silk fabric against *Staphyloccocus aureus* and *Pseudomonas Aeruginosa* bacterial strains



Fig.1. Effect of extract Concentration and dyeing temperature on Bacterial count for dyed silk.

As can be seen in Fig. 1, extract concentration demonstrated a vivid influence on bacterial count as opposed to dyeing temperature. The optimized values were found to be at 39.14% and 70° C. Also, it was noted that at lower extract concentrations and temperatures, there was a slight decrease in bacterial count. This could have been due to the low concentration gradient of the extract and the inability of the fibres in the structure of the fabrics to swell [8]. Then at very high concentrations and temperatures, the bacterial count values increase still due to dyed fabric saturation and decrease in dye molecule stability [13].

Table 4. Ar	ntibacterial effica	icy of dyed silk fabric	T	able 5. Fa	istness prop	oerties	of dye	d silk fa	abric
	Reduction in	Bacterial Count (%)		Plant	Light	Was	h	Rub	fastness
Plant extract	Si	lk fabric		extract	fastness	fastn	ess		
	Staphylococcus	Pseudomonas							
	aureus	Aeruginosa				CC	CS	Dry	Wet
GP	99.33	99.15		GP	5	4-5	5	5	4-5

3.3 Assessment of Antibacterial resistance and fastness properties of dyed fabrics

GP- Galinsoga Parviflora, CC-Colour Change, CS-Colour Staining, 4- Good and 5- Excellent.

From the results in Table 4, dyed silk fabric using optimized values demonstrated a bacterial count reduction percentage of 99.33 and 99.15 against *Staphylococcus aureus* and *Pseudomonas Aeruginosa* bacterial strains respectively. This high percentage reduction in bacterial count may have been attributed to the presence of more functional groups on silk fabric structure and therefore the ability of more of these medicinal extracted dyes to form covalent bonds with it [3]. It was further revealed that due to differences in chemical compositions of their cell walls, the percentage reduction was much higher in the case of *Staphylococcus* compared to *Pseudomonas* bacterial strain. Then the results of colourfastness properties presented in Table 5 ranged from very good to excellent thus confirming the presence of good covalent binding linkages between the dye and the fibres in the silk fabric structure [14].

4. CONCLUSIONS

To achieve the dyed fabric quality for antibacterial applications, optimization of dyeing conditions is vital. The study revealed that dyeing parameters have a significant effect on the



antibacterial properties of dyed silk fabrics. Optimized dyeing conditions were; extract concentration 39.14% and dyeing temperature 70°C. The dyed silk fabrics with the optimized condition showed high bacterial count reduction against both bacterial strains (99.33% and 99.15%) and overall very good to excellent fastness properties (4-5 to 5) thus confirming that *Galinsoga Parviflora* plant extract can be a potential source of eco-friendly natural dye with remarkable antibacterial potency.

ACKNOWLEDGEMENTS

The Authors acknowledge the financial support by ACEII-PTRE at Moi University in collaboration with Busitema University.

REFERENCES

[1] R. Rathinamoorthy and G. Thilagavathi, "Optimisation of process conditions of cotton fabric treatment with Terminalia chebula extract for antibacterial application," *Indian J. Fibre Text. Res.*, vol. 38, no. September, pp. 293–303, 2013.

[2] P Ganesan and K.J. Vishnu Vardhini, "Herbal treated microbial resistant fabrics for healthcare textiles," *Indian J. Nat. Prod. Resour.*, vol. 6, no. 3, pp. 227–230, 2015.

[3] E. M. E.-K. and R. S. R. E.-M. N.F.Ali, "The antimicrobial activity of pretreated silk fabrics dyed with natural dye," *Int. J. Curr. Microbiol. Appl. Sci.*, vol. 4, no. 6, pp. 1166–1173, 2015.

[4] A. C. M. Manyim Scolastica, Kiprop Ambrose K, Mwasiagi Josphat Igadwa, "Optimization of Extraction Conditions of Natural Dye from Euclea Divinorium using Response Surface Methodology," *Ann. Univ. Oradea Fascicle Text. Leatherwork*, pp. 47–52, 2020.

[5] A. Ketema and A. Worku, "Antibacterial Finishing of Cotton Fabric Using Stinging Nettle (Urtica dioica L .) Plant Leaf Extract," *J. Chem.*, vol. 2020, 2020.

[6] M. Y. Samar Ali, Sara Zameer, "Ethnobotanical, phytochemical and pharmacological properties of Galinsoga parviflora : a review," *Trop. J. Pharm. Res.*, vol. 3, no. 6, pp. 274–279, 2017.

[7] Y. Yin, L. Fei, and C. Wang, "Optimization of Natural Dye Extracted from Phytolaccaceae Berries and Its Mordant Dyeing Properties on Natural Silk Fabric," *J. Nat. Fibers*, pp. 1–11, 2017.

[8] S. M. and S. Kanakarajan, "Extraction, Optimization and Dyeing of Silk Yarn using Natural dye from Cosmos sp," *Int. J. Dev. Res.*, vol. 07, pp. 13865–13871, 2017.

[9] A. K. D. and G. D. K.P.Singh, "Evaluation of Antibacterial Activities of Chenopodium Album L .," *Int. J. Appl. Biol. Pharm. Technol.*, vol. 2, no. 3, pp. 398–401, 2011.

[10] V. N. Swamy, K. N. N. Gowda, and R. Sudhakar, "Dyeing of silk using Madhuca longifolia as natural dye source," *Indian J. Fiber Text. Res.*, vol. 40, no. December, pp. 419–424, 2015.

[11] U. H. Shaukat Ali, Sobia Jabeen, Tanveer Hussain, Sodia Noor, "Optimization of extraction condition of natural dye from pomegranate peels using response surface methodology," *Int. J. Eng. Sci. Res. Technol.*, vol. 5, no. 7, pp. 542–548, 2016.

[12] S. Noor, S. Ali, and T. Hussain, "Optimization of Dye Extraction Conditions from (Camellia sinensis) Green Tea Leaves Using Response Surface Methodology," *Asian J. Chem.*, vol. 27, no. July, pp. 4111–4114, 2015.

[13] S. Ali, T. Hussain, and R. Nawaz, "Optimization of alkaline extraction of natural dye from Henna leaves and its dyeing on cotton by exhaust method," *J. Clean. Prod.*, vol. 17, pp. 61–66, 2009.

[14] T. B. T. and L. H. Gopalakrishnan, "Environmental Friendly Dyeing of Silk Fabric with Natural Dye Extracted from Cassia singueana Plant," *J. Text. Sci. Eng.*, pp. 1–6, 2018.



VIRTUAL PROTOTYPING OF PROTECTIVE CLOTHING FOR OVERSIZED SUBJECTS

OLARU Sabina¹, POPESCU Georgeta¹, TOMA Doina¹, BADEA Ionela¹, GROSU Cristina^{1,2}

¹National Research and Development Institute for Textiles and Leather, 16 Lucretiu Patrascanu Street, 030508, Bucharest, Romania

² Gheorghe Asachi Technical University of Iasi, Faculty of Industrial Design and Business Management, 29 Mangeron Boulevard, 700050, Iasi, Romania

Corresponding author: Olaru, Sabina, E-Mail: sabina.olaru@incdtp.ro

Abstract: The paper presents the virtual prototyping of protective clothing for oversized female and male subjects, highlighting the importance of personalization and its competitive advantages. Customisation of protective clothing for oversized subjects offered the possibility to individualise the products to each wearer with different conformation and specific work activities. Customized protective clothing involves the dimensional and conformational aspects of the body, respectively the product size as well as the quality-linked functionality criterion, aspects regarding its wearability and protection tested in accredited laboratories, the effects over the individual comfort. The research implementation involved 3D body scanning for analysis and determination of anthropometric measurements and conformation, 3D CAD technology for automatic rapid design of patterns in Made to measure system, modeling and simulation of product in the virtual environment on customized mannequin, highlighting the body-product correspondence. New technologies in the context of Industry 4.0 are the seeds for disruptive innovation in the clothing industry, by increasing the digital capacity and flexibility to satisfy the customer requests and implement more dedicated services.

Key words: virtual, design, protective clothing, simulation, oversize

1. INTRODUCTION

The textile and clothing industry is a traditional and important part of the European manufacturing industry, having great impact on the economy and social well-being of numerous regions of EU.

In 2019, the entire EU-27 T&C industry represented a turnover of \notin 162 billion. According to Euratex, 160.000 textile & clothing companies, of which 99.8% are SMEs employed 1.5 million people. To keep up with the evolution of the textile-clothing industry, which follows the three strategic directions established by the European Platform for the Future of Textiles and Clothing, the textile industry must increase its competitiveness and benefit from a qualified human resource, adapted and ready to implement the new technologies to obtain smart, advanced, multifunctional, personalized textile products for both T&C and related industries [1, 2]. Recently, the European Commission launched the new Industrial Strategy for a globally competitive, green and digital Europe that will help deliver on three key priorities: maintaining European industry's global competitiveness and a level playing field, at home and globally, making Europe climate-neutral by



2050 and shaping Europe's digital future [3].

Design and creativity, quality fashion products, technical goods of high added-value, smart and technical textiles have been identified as major competitive advantages of the EU T&C industry. Technical textiles represent a key sector regarding the impact on economic growth, sustainable development and employment. The textile, clothing and fashion industry is in a continuous movement, characterized by an increased mobility of production factors, rapid delocalization, fragmentation and higher specialization of the activities in the value chains of products and services. The turbulent business environment and COVID 19 pandemic are forcing textile and clothing companies to adapt their production lines and increase their efficiency In order to face these challenges, companies must improve their internal organization and create a network/cluster of cooperations with external organizations [4-6].

Protective clothing creates barriers against external factors during the professional activity of workers and professionals. The properties of this type of clothing must be balanced in order to assure the level of protection, reduced weight and comfort during usage without impact on work performance.

The current paper presents the virtual prototyping of protective clothing for oversized female and male subjects, highlighting the importance of personalization and its competitive advantages, from the idea to the prototype. Customized protective clothing involves the dimensional and conformational aspects of the body, respectively the product size as well as the quality-linked functionality criterion, aspects regarding its wearability and protection.

2. 3D BODY SCANNING AND MORPHOLOGICAL ANALYSIS OF THE OVERSIZED SUBJECTS

In the study, the subjects were scanned using the body scanner 3D VITUS XXL and the measurement protocols and virtual bodies/parameterized virtual mannequins were generated (fig. 1). The anthropometric data was the basis for designing the personalized patterns in Made-to-Measure system.

The selected female subject had the following main body dimensions extracted from the measurement protocol:

- Body height (Ic) 172 cm;
- Bust circumference (Pb) 129 cm;
- Waist circumference (Pt) 122 cm;
- Hip circumference (Ps) 120 cm.

These values reveal that the bust circumference is 1 cm larger than the superior limit value of 128 cm mentioned in the standard SR 13545 - Clothing. Women's Body Measurement and Garment Sizes [7]. The maximum standard circumference of the bust has a value of 128 cm.

The selected male subject has the following main body dimensions extracted from the measurement protocol:

- Body height (Ic) 190.5 cm;
- Bust circumference (Pb) 143 cm;
- Waist circumference (Pt) 143 cm;
- Hip circumference (Ps) 166 cm.

The data extracted from the anthropometric standard reveal that the body dimensions are outside the standard SR 13544 - Clothing. Men's Body Measurement and Garment Sizes [8]. In this standard, the maximum chest circumference is 132 cm, and the maximum waist circumference is 128 cm.



(Top) Body Head/Neck Torso Shoulder Breast	// Bust Back Wai	st <u>Hip Arm Log</u>	(Top) Body Head / Neck Torso Shoulder Breast / Bust Back Waist Hip Arm
*	0010 Body height	168.2 cm (66.20 in)	0010 Body height 190 5 cm (75 0
	0020 Head height	23.6 cm (9.31 in)	0020 Head height 24 8 cm (9 75
<u> </u>	0030 Nock height	144.5 cm (56.90 in)	0030 Neck height 165.8 cm (65.2
	0040 Distance neck to hip	52.0 cm (20.46 in)	0040 67.6 cm (28.63
*	0050 Distance neck-knee	95.2 cm (37.47 in)	Distance neck to hip 0050 113.5 cm (44.6
*	0060 Distance waist-knee	56.0 cm (22.05 in)	Distance nock-knee
*	0085 Distance waistband-knee	56.0 cm (22.07 in)	Distance waist-knoe 66.6 cm (26.23
Ŕ	0070 Waistband height	104.9 cm (41.29 in)	Distance waistband, 63.9 cm (25.14 knee

Fig. 1. Measurement protocol for studied subjects

3. CUSTOMISATION OF THE SELECTED PROTECTIVE CLOTHING

In the study, different types of protective clothing were analysed and selected for female and male subjects and then were customized by the innovative technological design and production solutions. The Overalls suit with chest and waist pants for women has been developed according to the technical specification.

The protective clothing consists in the blouse, chest trousers and waist support pants. The overalls suit with chest pants and waist pants is worn by the operative personnel in the repair, maintenance and inspection activities of water pipes. The protective clothing for women was manufactured from fabric 35% PES, 64% TENCEL and 1% AS (antistatic), in blue colour with the weight of 205 g/m².

The model of protective clothing selected for the male subject is the Unique costume with two trousers for the Jardarmerie (service uniform), developed according to the technical specification. The unique costume consists of a blouse and two trousers and is worn by the military units as service uniform. The costume was manufactured from fabric containing 86% cotton mixed with 14% polyester, ripstop, dark blue color with the weight of 229 g/m².

Physical-mechanical and physical-chemical characteristics of the fabric were determined in the accredited laboratories (table 1) and the data were used for textile material characterization in the 3D simulation.

Characteristic	Values obtained in the accredited laboratories	Reference document		
Weight (g/m^2)	229	SR EN 12127:2003		
Weave	Diagonal 2/1 with ripstop	SR 6431:2012		
	form 2 filaments			
Warp density (threads/10 cm)	480	SR EN 1049-2/2000		
Weft density (threads/10 cm)	260	SR EN 1049-2/2000		
Tensile strength warp (N)	1426	SR EN ISO 13934-2013		
Tensile strength weft (N)	690	SR EN ISO 13934-2013		
Tear strength warp (N)	83.8	SR EN ISO 13937- 1:2001/AC:2006		
Tear strength weft (N)	97.8	SR EN ISO 13937- 1:2001/AC:2006		

Table 1. Physical-mechanical and physical-chemical characteristics of fabric used for male EIP (selection)



Colour	perspiration acid	4-5	SR EN ISO 1 05-E 04:2013
fastness	alkaline	4-5	
to	rubbing dry	4	SR EN ISO 105-X12:2003
	wet	4	

The pattern design for the selected protective clothing was implemented using Gemini Pattern Editor's Made-to-Measure module.

The fitting of customised patterns, designed according to individual body dimensions, was accomplished by modelling 2D/3D patterns and simulating protective clothing on the parameterized mannequin, using Optitex PDS software for visualization and modelling. The virtual prototyping system involves transferring and fitting protective clothing to virtual mannequin with various shapes and postures. In this sense, the protective clothing must be treated as elastic models and their deformation is controlled by the laws of dynamics [9,10].

The patterns designed in 2D were transposed in 3D environment in order to develop the virtual prototype of customized protective clothing. The following methodological steps were applied:

• dimensioning the virtual mannequin to the anthropometric dimensions obtained from 3D body scanning;

• adding stitching lines and guide points to the 2D patterns (fig. 2 for female subject and fig. 3 for male subject);

• introducing the data regarding the textile materials used for the manufacturing of protective clothing (fibrous composition, weight, drape, shrinkage etc.);

• 3D simulation of the clothing on the virtual mannequins (fig. 4 for female subject and fig. 5 for male subject);

• checking and modifying the 2D patterns to ensure the optimal fitting.



Fig. 2. The 2D patterns of the customized Overalls sit with seam lines and required characteristics of the textile material





Fig. 3. The 2D patterns of the customized protective clothing, with seam lines and required characteristics of the textile material



Fig. 4. Virtual try-on and verification of the customized protective clothing for women



Fig. 5. Virtual try-on and verification of the customized protective clothing for men

For checking the body-product correspondence, the Tension Map function was applied, which renders the degree of ease/adjustment of the products on the bodies (fig. 4 and 5). For the female subject, the software reveals the information that the jacket corresponds dimensionally. Also, the chest and waist trousers fit on the waist line and are slightly wide on the hips line. The degree of ease is justified by the semi-rigid silhouette of the protective clothing. This information is useful for the pattern designer which could return to 2D patterns and introduce necessary corrections.

For the male subject, the jacket product corresponds dimensionally too. Also, the trouser fits on the waist line and is slightly wide on the hips line. The case is similar with the protective clothing for female subject.



4. CONCLUSIONS

Customisation of protective clothing for oversized subjects offered the possibility to individualise the products to each wearer with different conformation and specific work activities. The research implementation allowed the direct involvement of the clothing manufacturing companies in the design of the customised protective clothing. The industrial partners participation represent also their first contact with the 3D body scanning for analysis and determination of anthropometric measurements, 3D CAD technology for automatic rapid design of patterns and simulation software for virtual prototyping. These new technologies in the context of Industry 4.0 are the seeds for disruptive innovation in the clothing industry, by increasing the digital capacity and flexibility to satisfy the customer requests and implement more dedicated services.

ACKNOWLEDGEMENTS

This work was supported by a grant of the Romanian Ministry of Research and Innovation, CCCDI - UEFISCDI, project number PN-III-P2-2.1-CI-2017-0046 and project number PN-III-P2-2.1-CI-2018-0912, within PNCDI III. Also, this work was carried out through the Nucleu Programme, with the support of MCID, project no. 4N/08.02.2019, PN 19 17 02 01, project title: "Advanced multifunctional logistics, communications and protection systems to improve the safety, operability and efficiency of emergency workers – SiMaLogPro".

REFERENCES

[1] EURATEX, "Key Figures 2019", [Online]. Available: https://euratex.eu/wp-content/uploads/EURATEX-Facts-Key-Figures-2020-LQ.pdf

[2] EURATEX, "Annual Report 2018", The European Apparel and Textile Confederation, 2018, [Online]. Available: https://euratex.eu/wp-content/uploads/2019/05/Euratex-annual-report-2018-LR.pdf

[3] European Commission, "Making Europe's businesses future-ready: A new industrial strategy for a globally competitive, green and digital Europe", 2020, [Online]. Available: https://ec.europa.eu/growth/content/making-europes-businesses-future-ready-new-industrial-strategy-globally-competitive-green-0_en

[4] G. Zulch, H. I. Koruca and M. Borkircher, "Simulation-supported change process for product customization – A case study in a garment company", in Computers in Industry, no. 62, pp. 568–577, 2011

[5] H-S. Park, "From Automation to Autonomy - a New Trend for Smart Manufacturing", in DAAAM International Scientific Book, vol. 3, pp. 075-110, 2013

[6] L. Tudor, "*Change in textile and clothing industry*", in Industria Textila, vol. 69, no. 1, pp. 37–44, 2018

[7] SR 13545:2010. Clothing. Women's Body Measurement and Garment Sizes

[8] SR 13544:2010. Clothing. Men's Body Measurement and Garment Sizes

[9] L. Indrie, J. Ilieva And Z. Zlatev, "Implementation of Curved Shapes in Textile Design", in Annals of the University of Oradea Fascicle of Textiles, Leatherwork, vol. 2, pp. 31-40, 2020

[10] A. Cupar, Z. Stjepanovič, S. Olaru, G. Popescu, A. Săliștean and A. Rudolf, "CASP methodology applied in adapted garments for adults and teenagers with spine deformity", in Industria Textila, vol. 70, no. 5, pp. 435-446, 2019



BAMBOO FIBER ANTIBACTERIAL EFFECT (A REVIEW)

RARU Aliona^{1,3}, FLOREA-BURDUJA Elena^{2,3}, IROVAN Marcela¹, FARÎMA Daniela³

¹ Technical University of Moldova, Direction of Academic Management and Quality Assurance, 168 Stefan cel Mare Street, Chisinau MD-2000, Republic of Moldova

² Technical University of Moldova, Faculty of Textile and Polygraphy, 4 Sergiu Radautan Street, Chisinau MD-2000, Republic of Moldova

³ Gheorghe Asachi Technical University of Iasi, Faculty of Industrial Design and Business, 29 Mangeron Street, Iasi 700050, Romania

Corresponding author: Raru Aliona, E-mail: aliona.raru@adm.utm.md

Abstract: This article analyzes the current known information and data on the bamboo plant natural properties: UV protection properties, natural resistance to pests and fungal infestations due to an antimicrobial agent known as 'bamboo kun'. Bamboo has a bacterial rate of up to 99.8%. The bamboo fibers air permeability is 20% higher than that of cotton, and the absorption capacity is 60% higher. After processing, the bamboo fiber contains no harmful chemicals (as specified in the Okeo-Tex Standard 100 - global testing and accreditation system for harmful substances screening in consumer fabrics). The Fabrics Verification Association of Japan confirms that even after 50 industrial washes the bamboo fiber samples keep their properties. Tests conducted by this association show that about 70% of the bacteria with which the bamboo fiber sample was infested were destroyed. Other studies on bamboo / cotton, cotton and viscose. Currently bamboo fibers are used in the manufacture of underwear, socks, bed linen, towels, and bathrobes. The information got as a result of the present study allows to realize the research hypothesis, a step that will be preceded by the own experimental research carrying out in order to determine new directions of bamboo fibers use in the medical field.

Key words: Bamboo, fiber, antimicrobial effect, multifunctional clothing, medical clothing.

1. INTRODUCTION

Daily, man comes into contact with numerous viruses, fungi and bacteria, which settle on his skin and clothing. In textiles, microorganisms multiply rapidly, under the action of heat and moisture. Although most of them are non-pathogenic, there are some that present a real danger to the body.

Developing new multifunctional clothing problem is current for the textile and medical field researchers. An increased interest is presented by the clothing having the monitoring role of the wearer's health as well as the clothing that has any diseases improving properties [1-4]. In this sense, an important role belongs to the multifunctional fabrics.

Around the world, antimicrobial textiles currently find an increasing spectrum of use: from improving daily hygiene conditions to prophylaxis, improvement and treatment of some diseases. In



hospitals it is recommended to use underwear and light clothing made of such materials, possessing bactericidal properties. To prevent complications that may occur in various diseases due to the "unhealthy" properties of clothing, it is recommended to use antimicrobial materials [1].

The bacteriostatic agent possesses various mechanisms of action on microbes: stagnation of the process of osmosis and diffusion of nutrients for microbes, slowing down the processes of multiplication of microbes, killing microbes [2-4]. Some antimicrobial agents are extremely irritating and toxic, which leads to the need to conduct studies that allow the creation of new types of antimicrobial materials [3, 4].

2. EXAMPLES OF GLOBAL STUDIES IN BAMBOO PROPERTIES

What is important for antimicrobial textiles is the determination of the requirements imposed on them: the use of bacteriostatic substances, which do not pose a danger to the health of the wearer, in sufficient quantities for killing microbes; increased action of the bactericidal agent and a fairly wide field of use for the treatment of diseases with the preservation of properties for the duration of wearing; the ability to preserve antimicrobial properties throughout operation, after several washes and / or sterilizations by different methods [1, 4].

In the specialized literature, from the group of antimicrobial and antiseptic fibers are mentioned those based on polyvinyl alcohol (PVA). A representative fiber in this regard is BIOKRIL, with a special and long-lasting antimicrobial efficacy, due to its structure that allows the antimicrobial agent to continuously diffuse to the surface of the fiber. Another acrylic fiber is also COURTEX, in which a number of metal salts of Ag and Zn are inserted, which are fixed to the support, by complex chemical bonds [1, 4]. These fibers have bactericidal properties. Another fiber with antimicrobial properties is also the fiber Microsafe Celanesse. Fiber Amicor has included in its structure antibacterial agents, for the elimination of unpleasant odors because of sweating. The fibers covered with Silver X-Static know various fields of use in medicine, due to the bactericidal properties they possess [1-4].

There are opinions that state that bamboo fiber naturally possesses antibacterial, hypoallergenic and deodorizing properties. Bamboo is the plant that has the fastest growing pace in the world. It grows on average by 10 cm per day, but there are species that grow by a millimeter every two minutes [5, 6]. The growth cycle of bamboo is fast and lasts from three to four months, the plants become mature in three to seven years [5, 6]. Although some species bloom every year, most of them flower in frequently, at intervals between 40 and 60 years and even up to 120 years [6]. Once a bamboo has blossomed, the seeds will fall on the ground, and the plant will die. In the textile form, bamboo retains many of the properties that the plant has [6]. Bamboo absorbs water very well, being able to take up to three times its weight in the water. The absorbency allows removing moisture from the skin so that it can evaporate [7]. Bamboo captures warm air in its transverse fibers to keep warm in case of low temperatures. The air permeability of bamboo fibers is 20% higher than that of cotton, and the absorption capacity is 60% higher, which provides increased comfort in the case of high temperatures [7]. After processing, bamboo fiber does not contain harmful chemicals (as specified in the standard Okeo-Tex 100 - global Testing and accreditation system for screening of harmful substances in consumer textiles) [8]. The fiber is naturally smooth and round, without chemical treatment, which means that it does not have sharp branches that irritate the skin. Bamboo also has many antibacterial qualities, which the bamboo fabric is able to retain, even after several washes [9, 10]. This helps to reduce bacteria that develop in clothes and can cause unpleasant odors. The bamboo plant has protective properties against UV rays and a natural resistance to pests and fungal infestations due to an anti-microbial agent known as "bamboo Kun", which prevents harmful materials from growing on the plant [7-11]. It is considered that bamboo


naturally possesses hypoallergenic, antibacterial, air freshener properties, has the ability to preserve temperature and attract moisture [8-10]. The bamboo plant is extremely durable because it grows naturally without the need to use pesticides or fertilizers and is completely biodegradable, so this eliminates the problem of disposal [8-11]. The processes of transformation of bamboo into fabrics can occur mechanically and chemically. The mechanical process is similar to other loose fibers: the fibers are extracted by maceration, which can be traditional or by means of natural enzymes, to break the wooden walls of the plant, after which the extracted fibers are combed and cleaned before spinning [8, 10]. All this increases production costs, risking the positioning of the eventual finished product out of the market. Bamboo can also be used as a raw material for viscose [5-14].



Fig. 1. Bamboo – advantages and disadvantages

Bamboo is the multifunctional fiber that revolutionized the textile industry, which leads to the need to conduct studies that would confirm or not the assumptions about its multiple advantages.

A group of researchers from Portugal conducted studies of 3D textile structures of different composition. Bamboo, cotton, polyester and bioactive polyester in proportions from 14 to 72%. 72% antimicrobial fiber spacing, such as bamboo, was not active against the tested micro-organisms, while flattened knitted fabrics made from the same yarn were effective against E. coli, P. aeruginosa and T. rubrum. Textiles subjected to testing are recommended for use as lining material for footwear [4].

In another study, bamboo raw materials obtained from the Anhui Taiping test center of the international bamboo and rattan center were tested. The outer and inner layers of bamboo were removed, and then cut into blocks of 10 mm. All the blocks were washed with ultrasound with deionized water and alcohol for 30 minutes at room temperature. To remove the impurity inside the bamboo structure, all samples were vacuum impregnated under water several times and then dried for 24 hours at 60 °C in vacuum. The obtained samples were subjected to antibacterial testing by the



bacteriostatic circle method, with the purpose of observing the growth inhibition properties against Gram-negative bacteria (E. coli) and Gram-positive bacteria (B. subtilis) [9].

Studies investigating the antimicrobial functionality of bamboo fiber were conducted on samples obtained from bleached bamboo material. The samples were incubated at 37 0C for 18 to 24 hours. A clear area of inhibition was observed for both tested microorganisms: E. coli and S. aureus [12].

Natural bamboo fibers produced from bamboo Neosinocalamus affinis have been subject to investigation with the aim of determining the natural bacterial rate and its influence factors. The results of antibacterial tests showed that natural bamboo fiber does not have natural antibacterial properties compared to other textile fibers [8, 10]. The linear relationship between moisture and bacteriostatic rate suggests that hygroscopicity may be a factor influencing the antibacterial performance of the fiber. A method of extraction could improve the antibacterial property of natural bamboo fiber against bacteria; therefore, extracts have influence on this [11, 14].

3. RESULTS AND DISCUSSION

Antimicrobial properties of bamboo fiber are proven by scientific studies conducted by several teams of researchers. The results from such studies are shown in table 1 and figure 2 [5-14].

Team of authors- conducted studies	Studies	Obtained results
Lixia Xi, Daochun Quinn, Xin An and Ge Wang – Resistance of natural Bamboo fiber to microorganisms and factor that my affect such resistance [10]	This study investigates antimicrobial proprieties of natural bamboo fiber compared to cotton, jute fiber, flax fiber, ramie fiber. The bamboo fibers tested were obtained by extraction using different solvents: cold water, hot water, ethanol, benzene, benzene/ethanol, 1%NaOH. The bacteria studied were Escherichia coli and Staphylococus aureus, and Candida albicans.	The results showed that natural bamboo fiber was not effective against E. coli, S. aureus, and C. albicanus. By comparison, the bacteriostatic rate of ramie against S. Aureus was over 90%, and that of regenerated bamboo fiber was 75,8%. Jute and flax had bacteriostatic rates against C. albicans of 48% and 8,7% respectively. The method of extraction of bamboo fiber with hot water proved to be the most effective against E. Coli, with a value of 70% proximity; the use of the 1% NaOH solution achieved an efficiency of more than 67%; at the same time it was proved that the antimicrobial properties of bamboo fiber do not have resistance against C. Albicanus.
T. Afrin,T. Tsuzuki, R.K. Kanwar and X. Wang – The origin of the antibacterial property of bamboo [11]	The origin of antibacterial activity of plant extracts from Australian-grown bamboo (Phyllostachys pubescens) is investigated. Bamboo extracts were made using water, dimethyl sulphoxide (DMSO) and dioxin.	It was found that the extract made in 20% DMSO aqueous solution showed weak antibacterial activity, whereas the extract made using 90% dioxin aqueous solution exhibited strong antibacterial activity, even after 20 times dilution. The results

 Table 1. Antimicrobial properties of bamboo fiber



	Gram-negative bacterium,	indicate that antibacterial agents of
	Escherichia coli (E. coli) -	P. pubescence are located in lignin,
	ATCC 25922 was used as test	not in hemicellulose or other water-
	organism.	soluble chemical components.
C. Javakarbnu –	For antibacterial testing was	The tests showed a durability of
Antimicrobial properties of	adopted the parallel streak	antimicrobial properties up to 10
bamboo fabric finished with	method	washing cycles and an inhibition
	against microorganisms E. coli	zone of 35 mm against S. aureus and
Vempadam Bark [12]	and S. aureus.	34 mm against E. coli.
		Bamboo has very good
	We analyzed the antibacterial	antimicrobial properties, followed
Textile verification Association of	and antifungal behavior of	by the combined fabric 50/50%
Japan –	bamboo fiber socks, compared to	bamboo / cotton, cotton and viscose.
Studies on bamboo fiber and	100% cotton, 100% viscose and	The results also showed that there
antibacterial effect [13]	50/50% bamboo/cotton socks.	was no antimicrobial activity level
		for cotton samples.
	For antibacterial testing was	•
Junyi Zhang, Bo Zhang, Xiufang	adopted the bacteriostatic circle	
Chen, Bingbinng Mi, Penglian	method. The results were	
Wei, Benhua Fei and Xindong Mu	estimated by their growth	It can be observed an inhibition
-	inhibitory properties against	zone 2,15 cm for Gram-negative
Antimicrobial bamboo materials	Gram-negative bacteria (E. coli)	bacteria (E. coli) and an inhibition
functionalized with ZnO and	and Gram-positive bacteria (B.	zone 2,5 cm for Gram-positive
Graphene Oxide Nanocomposites	subtilis). The antibacterial	bacteria (B. subtilis).
[14]	performance was estimated by	
	the size of the inhibition zone.	



Fig. 2. The properties of bamboo fiber

4. CONCLUSIONS

In the study, we can say that the bamboo fiber exhibits a number of advantages. At present bamboo has various fields of use: in construction, in papermaking, in the manufacture of dishes, the



newest bamboo is used in the textile industry. The properties of bamboo fiber are influenced by the method of fiber extraction and the method of manufacturing textiles: fabric or knit, the type of knit. Some studies confirm the antimicrobial properties of bamboo fiber, at the same time the results of other studies demonstrate antibacterial inactivity of natural bamboo fiber. Antibacterial agents of bamboo are located in lignin [9, 11]. The information obtained from this study is insufficient to confirm or not the antimicrobial properties of bamboo, as the results obtained differ from one study to another. In order to materialize the research hypothesis, it is necessary to carry out own experimental research in order to determine new areas of use of bamboo fibers in the medical field.

REFERENCES

[1] V. Goldade, N. Vinidiktova, "Antimicrobial fibers for textile clothing and medicine: current state", International Scientific Journal Theoretical and Applied Science, 2017, pp 178-194

[2] A. Raru, E. Florea-Burduja, M. Irovan, D. Farîma (2020, May) General aspects concerning multifunctional fiber textiles, INTERNATIONAL SCIENTIFIC CONFERENCE "Innovative solutions for sustainable development of textiles and leather industry" Oradea, Romania. P- ISSN 1843 – 813X, E - ISSN 2457-4880, CD- ISSN 2068 – 1070, [Online]. 21 (1). pp. 109-112. Available: <u>http://textile.webhost.uoradea.ro/Annals/Volumes.html</u>

[3] D. Farima, G. Broasca, "Textile supuse tratamentelor antimicrobiene", Buletinul Agir, 2010, p 5

[4] G. Broasca, D. Farima, "Tratamente antimicrobiene pe suprafețe textile", Leather and Footwear Journal, 2010, pp 61-66

[5] J. Piouceau, G. Bois, F. Panfili" Effects of High Nutrient Supply on the Growth of Seven Bamboo Species", Internatioal of Phytoremediation, [Online]. 16(10), 2014, pp. 1042-1057. Available: <u>https://www.researchgate.net/publication/258521582</u>

[6] https://lewisbamboo.com/pages/how-bamboo-grows

[7] G. Soares, A. Magalhaes, A. Vasconcelos, S. Ponto, J. Gomes, Comfort and antimicrobial properties of developed bamboo, polyester and cotton knitted spacer fabrics (Portugal: University of Porto), 2017

[8] L. Xia XI, "The Antibacterial Performance of Natural Bamboo Fiber and Its Influencing Factors", International Convention of Society of Wood Science and Technology, 2012, p 8

[9] M. Fanny, Antibacterial activity of bamboo extract against pathogenic bacteria (Taiwan: Catholic University), 2018

[10] L. Xia Xi, D. Quinn, G. Wang, Resistance of natural Bamboo fiber to microorganisms and factor that my affect such resistance, 2013, p 6501-6509

[11] T. Afrin, T. Tsuzuki, R. K. Kanwar, X. Wang, "The origin of the antibacterial property of bamboo", The Journal of Textile Institute, 2012, pp 844-849

[12] C. Javakarbanu, "Antimicrobial properties of bamboo of fabric finished with Vempadam Bark", Journal of Scientific & Engineering Research, 2018, pp 666-677

[13] C. Gomathi, M. Manoharan, "Antimicrobial behaviour of socks from bamboo fibres", [Online]. Available: <u>https://indiantextilejournal.com/</u>

[14] J. Zhang, B. Zhang, X. Chen, M. Bingbing, W. Penglian, F. Benhua, M. Xindong, Antimicrobial Bamboo Materials Functionalized with ZnO and Graphene Oxide Nanocomposites, 2017, p 239



CHARACTERIZATION OF SOME TEXTILE ARTIFACTS FROM PLOPIS WOODEN CHURCH – PART OF UNESCO WORLD HERITAGE SITES

SECAREANU Lucia-Oana¹, SANDULACHE Irina¹, MITRAN Elena-Cornelia^{1,2}, LITE Mihaela-Cristina^{1,2}, IORDACHE Ovidiu¹, PERDUM Elena¹

¹ The National Research and Development Institute for textiles and Leather (INCDTP), Materials Research and Investigation Department, 16 Lucretiu Patrascanu, 030508 Bucharest, Romania, E-mail: <u>office@incdtp.ro</u>

² University Politehnica of Bucharest,

Faculty of Applied Chemistry and Materials Science, 1-7 Gheorghe Polizu, 011061 Bucharest, Romania, E-mail: <u>secretariat@chimie.upb.ro</u>

Corresponding author: Sandulache, Irina, E-mail: irina.sandulache@incdtp.ro

Abstract: Churches are often similar to art museums, having numerous collections of frescoes, icons, liturgical books, old furniture and various textile pieces, some of them even from the 15th to the 19th century timeframe. Due to their great historical significance, some of these churches became part of UNESCO World Heritage Sites. The Plopis Wooden Church from Sisesti village (Maramures county, Romania) is an example of such sites.

Generally, the textiles found in churches have a role in retaining the humidy from the environment and preventing proper ventilation, contributing to an increase in the moisture content of wood, which can lead to the development of mold. With that in mind, the regular shaking and airing of fabrics, carpets, and any other textile found inside churches, especially wooden ones, is indicated. Consequently, the identification of the fibrous composition of historic textiles and the processes involved in their aging are essential because long-term conservation of such textiles is influenced by these factors. This information facilitates the development of adequate conservation strategies. The chemical composition of fibers greatly impacts their properties. The main requirement for a technique to be suitable for characterizing historic textiles is that the said technique is non-destructive, or micro-destructive, at most. Thus, for the present study, the assessment of a texile fragment – 'Adam and Eve' fabric from Plopis Wooden Church, that is believed to be roughly the same age as the church – was performed via scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS).

Key words: textiles, heritage, fibers, SEM, EDS.

1. INTRODUCTION

The 'Saint Archangels' Plopis Wooden Church from Sisesti village (Maramures county), Romania, was built during 1796 and 1798 [1]. The church is part of UNESCO World Heritage Sites since 1990. The Plopis church was built from oak wood, with many unique features. The church has had many repairs which were meant to preserve it, and the last major restoration was carried out at the beginning of the 1990s. The passing of time had an impact especially upon the frescoes that were painted in 1811, which nowadays are present only in the southern part of the nave [2].



The comprehension of the function, context, and role of a historically valuable object are essential information that may impact its conservation process [3]. Moreover, for textiles in this category, the identification of the fibrous composition and aging processes and mechanisms are important for their long-time conservation.

2. MATERIALS AND METHODS

2.1. Materials

The textile fabric has its origin in the 'Saint Archangels' Plopis Wooden Church. More specifically, it was taken from the western part of the nave, in the area where the choir is situated. The fabric is a long and friable strip of approximately 150 cm, with a film-like layer on one of its sides. The role the fabric had within the Church was not yet identified.

For a precise assessment of the fabric and due to its heterogeneity, two different areas of interest were selected (Fig. 1).



Fig. 1. Photographs of the entire textile fabric (a) and the areas from which the two samples were selected $(b - area \ 1 \ and \ c - area \ 2)$

2.2. Methods

A scanning electron microscope (Quanta 200, FEI) was used in conjunction with an energydispersive X-ray spectroscopy detector (Element, EDAX-AMETEK). The SEM analysis was carried out at accelerating voltages between 10 - 15 kV. The EDS analysis was performed in mapping mode at 15 kV accelerating voltage and 2000× magnification, for both samples.

For both analyzes, the samples were placed on aluminum specimen stubs covered with conductive adhesive carbon tape. The samples did not require additional metal coating and were used as they were.

3. RESULTS AND DISCUSSION

3.1. SEM results

All SEM results were obtained as longitudinal views of the samples placed horizontally, without tilting.





Fig. 2. SEM micrographs of sample 1 at $100 \times (a)$, $400 \times (b)$, and $2000 \times$ magnification (c)

The first sample has been selected from an area which presented both the textile fabric, as well as a film that was present at its surface. The SEM micrographs from Fig. 2 (a, b) show the fibers twisting into a thread and the film on its surface. The mean diameter of the threads is approx. 610 μ m, whilst the diameters of individual fibers range between 10 and 20 μ m, corresponding to linen fibers, according to literature [4]. The fibers have a linen/ hemp appearance (the distinction between these two types of fiber cannot be made via SEM) [5]. The film partially covering the surface of the threads has a granular appearance, as in Fig. 2 (c).



Fig. 3. SEM micrographs of sample 2 at $100 \times (a)$, $400 \times (b)$, and $2000 \times$ magnification (c)

The second sample has been selected to emphasize the textile fibers, not the surface film. The similarity of the fibers'appearance to linen/ hemp fibers was observed in more detail in longitudinal view, especially the smooth surface pattern with bamboo-like cross-marking nodes. The high degradation state of the sample was noticed as well, with longitudinal splits of the fibers and the scuffing of the cross-marking nodes.





3.2. EDS results



Fig. 4. EDS map and spectrum for sample 1



Fig. 5. EDS elemental quantification for sample 1 (wt%)

The area that was mapped is the same as in Fig. 2 (c). The high carbon and oxygen content indicate that the film covering the fabric may be some type of natural polysaccharide-based adhesive [6]. The other elements are, most likely, impurities (oxides, salts), due to their insignificant proportions (< 1%), when compared to carbon and oxygen.





Energy (keV)

Fig. 6. EDS map and spectrum for sample 2



Fig. 7. EDS elemental quantification for sample 2 (wt%)

The area that was mapped is the same as in Fig. 3 (c). Similar to sample 1, the analysis revealed the presence of multiple elements, besides the expected carbon and oxygen inherent to the fibers, which are mostly composed of cellulose (72.5-81%) and, in a lesser proportion, of hemicellulose (14.5-20%) [7]. The many other chemical elements may be present on the sample due to finishing treatments that may have been applied to the textile fabric (i.e., mordanting, dyeing) [8]. Additionally, impurities may also influence the chemical composition.

4. CONCLUSIONS

Following the analyzes that were performed on the samples, the cellulosic nature of the fibers was identified, even though the exact type of fiber could not be determined due to the similar microscopic appearance of linen and hemp. The advanced state of degradation of the fabric was



highlighted and, while it was certainly caused by the old age of the textile artifact, it may also have been accelerated by the salts and oxides that were applied during the finishing processes.

ACKNOWLEDGEMENTS

This work was supported by a grant of the Romanian Ministry of Research and Innovation, CCCDI-UEFISCDI, project number PN-III-P1-1.2-PCCDI-2017-0878/55PCCDI, within PNCDI III.

REFERENCES

[1] B. Tit. (1911). "Date istorice despre protopopiatele, parochiile și mănăstirile române din Maramureș din timpurile vechi până în anul 1911". Gherla.

[2] M. Muresan. (2015). "Biserica de lemn din Plopiş "Sfinții Arhangheli" – monument UNESCO". Available: http://turismmaramures.ro/biserica-de-lemn-din-plopis-sfintii-arhangheli-monument-unesco/

[3] National Park Service's Museum Handbook. (2002). "Part I, Appendix K: Curatorial Care of Textile Objects".

[4] S. M. Yukseloglu, S. Canoglu. (2017). "*Natural fibre composites: a review on flax fibres*". Annals of University of Oradea, Fascicle of Textiles, Leatherwork, 18 (1), 137–142.

[5] Textile School. (2018). "*Microscopic appearance of Fibers*". Available: https://www.textileschool.com/330/microscopic-appearance-of-fibres/

[6] C. V. Horie. (2010). "Materials for Conservation: Organic Consolidants, Adhesives and Coatings". 2nd ed., Routledge, 221–228.

[7] L. Yan, B. Kasal, L. Huang. (2016). "A review of recent research on the use of cellulosic fibres, their fibre fabric reinforced cementitious, geo-polymer and polymer composites in civil engineering". Composites Part B: Engineering, 92, 94–132.

[8] J. N. Chakraborty. (2014). "Fundamentals and Practices in Colouration of Textiles. Chapter 20 - Dyeing with natural dyes". Woodhead Publishing India, 233–261.



COMPARATIVE STUDY BETWEEN POLYAMIDE/ELASTANE AND COTTON/POLYAMIDE/ELASTANE SOCKS

SZABO Monica¹, DOCHIA Mihaela², GAVRILAŞ Simona³

¹ "Aurel Vlaicu" University of Arad, Faculty of Engineering, Department of Automation, Industrial, Textile and Transportation Engineering, Postal address, 310330, 2-4 Elena Dragoi Street, Arad, Romania, E-Mail: <u>szabomonica@yahoo.com</u>

² "Aurel Vlaicu" University of Arad, Research Development Innovation in Technical and Natural Science Institute, Postal address, 310330, 2-4 Elena Dragoi Street, Arad, Romania, E-Mail: <u>dochiamihaela@yahoo.com</u>

³ "Aurel Vlaicu" University of Arad, Faculty of Food Engineering, Tourism and Environmental Protection, Department of Technical and Natural Sciences, Postal address, 310330, 2-4 Elena Dragoi Street, Arad, Romania, E-Mail: simona2213@yahoo.com

Corresponding author: Dochia, Mihaela, E-mail: dochiamihaela@yahoo.com

Abstract: The paper presents a comparative study of the behaviour of 97% polyamide/3% elastane socks, named functional socks, and of 70% cotton/27% polyamide/3% elastane socks, called classic socks, both type used for moderate-intensity physical activity. Nowadays, the question always arises about what socks to wear: classic 100% cotton socks, cotton/polyamide/elastane socks, or polyamide/elastane socks. The wearing comfort of the socks is the determining factor in choosing the optimal variant. The functional socks are made of 100% synthetic yarns and are appreciated due to the properties of absorption and rapid transfer of moisture from the body to the environment. The classic socks are made of cotton or cotton yarns mixed with synthetic yarns, giving better wearing comfort compared to functional socks through the high moisture absorption capacity. But due to the small capacity to transfer moisture from the body to the outside environment, wearing for a long time can produce discomfort as a result of the humid environment that is created. The two types of socks were evaluated through a series of analyses as follows: weight per piece, optical microscopy, hygroscopicity, hydrophilicity, elasticity in the direction of stitches' rows and degree of compression. The analysed data led to the conclusion that functional socks have superior elasticity and comfort properties compared to the classical ones.

Key words: functional socks, classic socks, knitted structure, degree of compression, wearing comfort

1. INTRODUCTION

Nowadays, the question always arises about what socks to wear: polyamide/elastane (functional socks) or cotton/polyamide/elastane blended yarns socks (classic socks). The wearing comfort of the socks is the determining factor in choosing the optimal variant. Wearing comfort is ensured by the raw material from which the socks are made and by the structure of the knit [1-3].

Without performing an initial analysis, the 100 % cotton socks would be the optimal option, but there is the problem of high hygroscopicity of cotton, around 8.5 %, which in the case of moderate physical activity leads to the absorption and maintenance of moisture, a situation that



creates discomfort and even irritation if the wear is for a long time [4]. To reduce the hygroscopicity, a variant of 70 % cotton + 27 % polyamide + 3 % elastane socks is proposed, elastane being used only for the ankle fixation garniture. Using this composition, the hygroscopicity of the socks reaches values around 4 %. This value is lower than that of cotton, but still during wearing can create discomfort due to the relatively high moisture absorption capacity. For moderate physical activity, using socks with a composition of 97 % polyamide + 3 % elastane and structures with transferred stitches elements will allow the foot to breathe and to rapid elimination of moisture, hygroscopicity being around 3 %.

The rib 1:1 knitted structures with transferred stitches, due to their architecture allows high deformability compared to classic jersey structures. The structures used in the production of socks are based on the knitting technique with transferred stitches or transferred stitches elements. The final product, the sock, is spatially contoured with different properties and homogeneities on the surface, with a positive influence on wearing comfort. By knitting and spatial contouring, articles with various shapes and perfectly adapted to the foot are obtained. Also, these structures have dimensional stability and good shape, the basic rib 1:1 bond being stable and balanced in terms of internal energies. This is due to the arrangement of the structure's elements. The thickness of the rib 1:1 structure with transferred platinum loops will be higher than that of the jersey structure made in similar conditions, the thermal insulation capacity and air permeability being better. The structures have low desirability and do not run at the edges. The high elasticity in the transverse direction positively influences the wearing comfort [5, 6].

For all the presented situations, the same type of "air cool" or "respira" sports shoes will be worn, which allows the foot to breathe during the physical activity.

2. EXPERIMENTAL PART

The socks were made of yarns of the same fineness, Ne 40/1, with the same size group, 39-41, on the same type of circular knitting machines with a small diameter for socks, Merz. The structures of the socks were manufactured differently, the ones made of 97 % polyamide + 3 % elastane yarns (functional socks) having a 1:1 rib structure with transferred stitches for the opposite side of the sole and jersey structure for the areas of the sole, heel and toe. For better fixation, on the hollow portion of the foot, the socks were knitted in the 2:1 rib structure.

The 70 % cotton + 27 % polyamide + 3 % elastane socks (classic socks) were manufactured in jersey structure and 2:1 rib structure for ankle fixation garniture. For both types of socks, the percentage of elastane was 3 % and was used for the 2:1 rib structure. The socks were knitted on a circular socks machine with a small diameter, Merz CC4 II.

The technical characteristics of the Merz CC4 II knitting machine are presented in Table 1.

	Table 1 . The technical specifications of the Merz CC4 II knitting machine					
Technical specifications	Technical data					
Needle dial diameter [inch]	4 3⁄4					
Pitch [E]	E 24					
Number of needles	360					
Number of cam systems	4					
Number of threads guides	10 guides on system 1, and 8 on systems 2-3-4					
Needle bad rotation speed [rpm]	Variable working speed (90 rpm 320 rpm), optimum 240 rpm.					
Engine power [Watt]	400					
Power frequency [Hz]	50-60					
Overall dimensions	270 cm height, 90 cm length, 68 cm width and 133 cm of yarn package ring diameter					

Table 1. The technical specifications of the Merz CC4 II knitting machine



The functional and classic socks were evaluated through a series of analyses as follows: weight per piece, optical microscopy, hygroscopicity, hydrophilicity, elasticity in the direction of stitches' rows, and degree of compression.

The weight per piece was measured with an analytical balance Kern ABT 220-4M. The images of the knitted fabric structures of the two types of socks were captured with stereomicroscope Zeiss Stemi 2000 with AxioCam. The Sartorius MA 100 balance at 105 ^oC was used for hygroscopicity determination. Evaluation of the hydrophilic properties of the analysed socks was carried out according to the AATTCC Test Method 79-2007.

To determine the elasticity for the two socks variants the EN ISO 14704-3/2007 was followed. The socks from which the specimens were taken were conditioned for 12 hours in air-conditioned rooms with the parameters of the standard atmosphere: $T = 20 \pm 2$ ^oC, p = 760 mm colHg = 1 atm, $\phi 65 \pm 5\%$. Three specimens with the size of 50 mm x 50 mm from each type of socks were taken from the stitches' rows direction and analysed with an EMI dynamometer. The working parameters were: the gauge length of 100 mm ± 1 mm, a speed rate of 100 mm/min, and a tensile force of 100 cN.

The degree of compression was measured with a dynamometer according to NFG 30 102 - Textiles - Knitted goods - Determination of restraining force. The speed rate was set to 100 mm/min and values in the minimum, average and maximum points at the ankle were considered. Each sample was subjected to 6 elongations, the pressure being calculated with the force recorded during the sixth cycle. Before analysis, the samples were washed and dried, after which they were kept for 8 hours in air-conditioned rooms with a standard atmosphere.

3. RESULTS AND DISCUSSIONS

The values for the weight per piece, hygroscopicity and hydrophilicity of the two types of analysed socks are presented in Table 2. Because functional socks were made of different structures (1:1 rib structure with transferred stitches, jersey structure and 2:1 rib structure), the hydrophilicity evaluation was performed in each area.

Sample	Weight per piece [g]	Hygroscopicity [%]	Hydrophilicity [sec.]
97 % polyamide + 3 % elastan (functional socks) - 1:1 rib structure with transferred stitches area			3
97 % polyamide + 3 % elastan (functional socks) - jersey structure area	6.631	3	50
97 % polyamide + 3 % elastan (functional socks) - 2:1 rib structure			40
70 % cotton + 27 % polyamide + 3 % elastane (classic socks) - 1:1 rib structure	9.318	4	1

Table 2. The weight per piece, hygroscopicity and hydrophilicity of functional and classic socks

As can be seen from Table 2, for the classic socks that have in their composition predominantly cotton, the hygroscopicity is higher than that of functional socks where the percentage of polyamide is 97 %. The hydrophilicity values were around 1 second for classic socks due to the high moisture absorption capacity of the cotton fibres. Functional socks showed different values of hydrophilicity, these being around 3 seconds for the 1:1 rib structure with transferred stitches area and 40 - 50 seconds for the jersey and 2:1 rib structures areas. This is due to the lower absorption capacity of polyamide fibres and different structures of the socks.



The knitted structure images of the classic and functional socks are shown in Figures 1 to 4.



To study the behaviour during wearing, both types of socks have been subjected to the stretching solicitation in the direction of the stitches' rows. By stretching, the diameter of the yarn is reduced, the rows of stitches arranged face-to-back are flattened and amounts of threads migrate from the needle and platinum loops into the flanks leading to yarn to yarn friction forces at the points of contact, forces that oppose to elongation and the rows of stitches it's getting close to tangency. To evaluate the behaviour at stretching solicitation, three types of quota were considered as shown in Figure 5.



Fig. 5. The quotas in the initial state and after elongation of the analysed socks

Where: 1 - distance from the top of the sock to the middle of the heel, in a relaxed state;

2 – width on the gasket finish line, in a relaxed state;

1T - elongation at traction.

For each sample, 3 tests were performed, and the average values of the considered quotas are presented in Table 3.

Table 3 : The average	values of the co	nsidered quotas j	for stretching eva	luation of functiona	l and c	lassic socks
------------------------------	------------------	-------------------	--------------------	----------------------	---------	--------------

Sampla	Quota 1	Quota 2	Quota 1T
Sample	[mm]	[mm]	[mm]
97 % polyamide + 3 % elastin (functional socks)	205	85	424
70 % cotton + 27 % polyamide + 3 % elastane (classic socks)	190	80	342

The obtained results are summarized in Table 4 and were calculated according to equation (1).

$$F[cN/cm] = \frac{F_{device}[cN] \cdot 100}{l[cm]}$$

86

(1)



Where: F - the resulted force per cm [cN/cm]; F_{device} - force indicated by the device [cN]; A - test sample width [5 cm].

	Sample	Deformation	Deformation	Deformation	Deformation	Elongation	Elongation
Sample	width	size 1	size 2	force 1	force 2	1	2
	[cm]	[mm]	[mm]	[N]	[N]	[cN/cm]	[cN/cm]
Functional socks	5	30	60	0.7545	1.240	15.09	24.80
Classic socks	5	30	60	0.6997	1.387	13.99	27.74

The graphical representations of the deformation dependency [%] under the traction force action of the two types of analysed samples are represented in Figures 6 and 7.



Fig. 6. Elongation for functional socks

Fig. 7. Elongation for classic socks

For practice, the final values are of interest (those for deformations of 60 %). By analysing figures 6 and 7 it results that for 60 % deformations, lower tensile forces are applied and smaller deformations result for functional socks than for the classic ones. This leads to the conclusion that functional socks are more dimensionally stable than the classic ones due to the deformability properties of knit structures.

To evaluate the compression of the socks, the values in the minimum, average and maximum points at the ankle level were determined as shown in Figure 8, the pressure being calculated with the force registered during the sixth cycle, in point a.



Fig. 8. The compression lines

The parameters and test results for compression solicitation are presented in Table 5 and the degrees of compression for functional and classic socks are shown in Figures 9 and 10.

By analysing Figures 9 and 10 it results that the compression degree is 17.03 hPa for the functional socks and 14.81 hPa for the classic ones. A higher degree of compression for the functional socks leads to a better fixation on the foot and implicitly to a superior comfort to wearing



compared to classic socks. Because the classic socks have a lower degree of compression they can easily slip on the leg, twist in a spiral and create discomfort when wearing. Both types of socks correspond to degree 1 of compression.



Table 5. The parameters and test results for compression solicitation of functional and classic socks



Fig. 10. Degree of compression for classic socks

5. CONCLUSIONS

The lower hygroscopicity of the functional socks led to superior wearing comfort and a higher moisture transfer capacity compared to the classic ones. The 1:1 rib structures with transferred stitches and the 2:1 rib structures in the hollow area of the sole used to produce functional socks give a lower elongation to traction and implicitly superior dimensional stability compared to classic socks with jersey structure. Both types of socks correspond to degree 1 of compression. Functional socks have a higher degree of compression and ensure a better fit on the foot compared to the classic ones. The analysed data lead to the conclusion of better elasticity and comfort properties for functional socks compared to the classic ones.

REFERENCES

[1] M. A. Morris, H. H. Prato, N. L. White, "*Relationship of Fiber Content and Fabric Properties to Comfort of Socks*", Cloth. Text. Res. J., vol. 3 (14), pp. 14-19, 1984.

[2] K. Herring, D. Richie, "Friction blisters and sock fiber composition. A double-blind study", J Am Podiatr Med Assoc, vol.80 (2), pp. 63-71, 1990.

[3] F. Selli, Y. Turhan, "Investigation of air permeability and moisture management properties of the commercial single jersey and rib knitted fabrics" Tekstil ve Konfeksiyon, vol. 27 (1), pp. 27-31, 2017.

[4] P. Komárková, V. Glombíková, A. Havelka, "*Heat and Moisture transport of socks*", IOP Conf. Series: Materials Science and Engineering 254, vol. 64 (4), pp. 198-201, 2017.

[5] V. K. Dhanapal, R. Dhanakodi, "Influence of moisture management properties on socks made from recycled polyester, virgin cotton and its blends", FIBRES & TEXTILES in Eastern Europe, vol. 28, 4(142): pp. 76-81, 2020.

[6] S. J. Howarth, K. Rome, "A short-term study of shock-attenuation in different sock types", The Foot, vol. 6, pp. 5-9, 1996.



COMPOSITE TEXTILE STRUCTURES FOR PARIETAL DEFECTS

VISILEANU Emilia¹, CHIRIAC Laura², SCARLAT Razvan³, VLADU Alina⁴

^{1,2,3,4} Institutul National de Cercetare Dezvoltare pentru Textile si Pielarie, 16, Lucretiu Patrascanu street, sector 3, Bucharest, Romania, E-mail: <u>office@incdtp.ro</u>

Coresponding author: Visileanu Emilia, E-mail: e.visileanu@incdtp.ro

Abstract: Composite textile structures designed to repair parietal defects, were obtained from monofilament yarns of polyester, polylactic acid and multifilament of Ag and high-density polyethylene, on Shima Seiki SIG 123 knitting machine of E8 gauge. The mass of composite structures after finishing is placed between 29.6 g/m^2 and 158.9 g/m^2 . The textile structures from: PES/ Ag and PP/ Ag belong to low elastic modulus meshes (<10.9MPa - specific to the abdominal fascia) and those from: PES/ PE and PES/ PLA belong to high elastic modulus meshes (>10.9MPa). The anisotropy is suitable for all variants of textile structures with values less than 1.00. The best value of cell proliferation indicator - MTT (1,771) was recorded at the PES/PLA composite textile structure which is higher than the control value of 1.22; this structure also shows the lowest value of cell death indicator - LDH (0.405), but which is higher than the control value of 0.370; cell viability has a very high level in this structure (144.83%), higher than that observed in control cells (HT 29 cells in culture) of 100%.

Key words: composite, hernia, Young's modulus, anisotropy, biocompatibility.

1. INTRODUCTION

A composite material is a material obtained of two or more constituent materials having different physical or chemical properties which, when combined, generate a material with better characteristics than the properties of the individual components. Composite textiles are referring to a wide range of textile surfaces that can be obtained by weaving, braiding, knitting, but also to nonwoven materials. These materials are also known as technical textiles used for: transport, geotextiles, civil construction, road construction, aerospace, military, medicine, sports equipment, protective clothing, etc [1].

The concept of using meshes to treat hernias was introduced more than 50 years ago. Treating hernias using surgical meshes is now the standard procedure in most countries and is widely accepted as superior to primary suture repair [2, 3]. As a result, there has been a rapid growth in the multitude of meshes available and choosing the most appropriate one can be challenging.

The most important properties of a mesh are the type of filament, tensile strength and porosity. These determine the weight of the mesh and the biocompatibility [4, 5]. The required tensile strength is much lower than initially considered, and low weight meshes are considered to be superior due to the low risk of infection and shrinkage. In case of meshes placed in the peritoneal cavity, special attention should be paid to the risk of adhesion [6]. A wide variety of composite meshes have been developed to overcome this drawback, but so far neither seems to be superior to the other.



2. MATERIALS AND METHODS

Composite textile structures were developed on the Shima Seiki SIG 123 knitting machine of E8 gauge, equipped with the new Rapid Response R2CARRIAGE system from SHIMA SEIKI, which achieves a faster return of the carrier after each stroke. This requires less space to turn, which allows the area to be increased for running at full speed. This allows faster knitting along the way, generating an increase in productivity of over 10%. The variants of composite textiles are identified by the following notations: S1 - from multifilament yarns of PES and Ag, S2 - from multifilament yarns of PES and PE, S3 - from multifilament yarns of PES and monofilaments from PLA; S4 - made of multifilament PP and Ag threads. The design setup of the composite textile structures made on the Shima Seiki SIG 123 knitting machine are presented in fig. 1.



Fig. 1. The design setup - single jersey structure

Biocompatibility is the test for determining the potential toxicity resulting from body contact with a medical material or device. The biocompatibility of the materials was evaluated on HT-29 epithelial cells, cultured in DMEM medium supplemented with 10% fetal bovine serum. To evaluate biocompatibility, HT-29 cells were seeded at a density of 1.5 x 104 cells per well in 500 μ l of culture medium and incubated at 37°C (5% CO₂) together with the newly synthesized biomaterials for 24 hours. The evaluation of biocompatibility degree was achieved by MTT - cell proliferation tests, respectively LDH- (Lactate dehydrogenase) - cell death and cell viability (%).

3. RESULTS

The textile structures were finished on the laboratory equipment of INCDTP by applying the following technological flow:

✓ washing - degreasing with 2 g/l sodium carbonate, 2 g/l sodium hydroxide at 38°C, 2 g/l Kemapon PC/LF, 2 g/l trisodium phosphate, duration 30 min, temperature 60° C;

 \checkmark rinsing with water at 60°C, 40°C, 20°C;



- \checkmark rinsing with distilled water;
- ✓ laser or mechanical cutting to the dimension of 10/10 cm or 20/20 cm;

 \checkmark packaging in a package consisting of multilayer foil based on polyolefins, lined with special paper for the circulation of sterilizing agent and provided with sterilization indicators. Sterilization with ionizing radiation (table 1).

No.	Charact			UM	Variant				
10.	o. Characteristic			UM	S1	S2	S 3	S4	
1	Compos	ition		%	95% PES	68,5% PES	97,6% PES	34,1% PP	
1	Compos	nion			4,8% Ag	31,5% PE	2,4% PLA	65,2% Ag	
2	Mas	S		g/m ²	51,3	29,6	38,1	158,9	
	Density	Но	riz	wales/10 cm	70	72	50	90	
3	Density	Vort	PES	rows/10 cm	120	105	155	72	
		Vert	vert	Ag	rows/10 cm	2	2	2	2
4	Breaking	Но	riz	Ν	84,5	62,4	43,7	551,3	
4	resistance	Ve	ert	IN	117,10	74,3	31,6	42,3	
5	Elongation	Ve	ert	%	33,5	53,5	59,5	62,5	
3	at break	Ho	riz	%0	51,9	56,1	26,1	55,4	
6	Thickn	less		mm	0,77	0,27	0,75	0,86	
7	Deformation	nocista		kPa	139,0	98,4	91,5	471,1	
/	Deformation	resista	ince	mm	45,4	37,7	41,7	50,8	
8	Young's	wa	ırp	MDa	0,31	11,57	0,74	2,4	
8	modulus	We	eft	MPa	1,68	16,3	0,13	16,0	
9	Anisotr	ору		-	0,73	0,15	0,77	0,82	

Table 1. The physico-mechanical characteristics of the finished composites structures

The analysis of the data presented in table 1 shows the following aspects:

- the composite structure with the highest mass (S4 - 2PP + Ag) has the highest values of breaking strength (551.3 N) and resistance to deformation (471 kPa), but also the highest values of elongation (62.5 % and 155.5%) and of the deformation (50.8 mm);

- the composite structure S2 (PES + PE) with the lowest mass (29.6 g/m²) has good values of the breaking strength (62.4 N and 74.3 N) and of the deformation resistance (98.4 kPa), smaller elongations (53.5% and 56.1%) and the smallest deformation (37.7 mm);

- the textile structures S1 and S4 fall belong to low elastic modulus meshes (<10.9 MPa) which do not generate high shear forces but have a higher degree of deformation. In general, because the abdominal wall behaves almost twice as elastic vertically compared to the horizontal direction, meshes with higher elasticity are required in the cranio-caudal direction for the midline of the defect repair area;

- the textile structures S2 and S4 belong to high elastic modulus meshes (>10.9 MPa) providing a strong mechanical reinforcement of the abdominal wall, but with the disadvantage of increasing shear forces between the mesh and the abdominal wall;

- the anisotropy of the composite textile structures is very good having values <1.0, the best value, 0.15 recording for the variant S2.

The analysis of the pore shape and dimensions (table 2) shows that the pore surface is

between: 0.272 mm^2 (S3) and 0.502 mm^2 (S4). The analysis of SEM images of the composite structures (table 3) illustrates the alternation of the rows of meshes between the component threads of the textile structures. The analysis of the elemental composition (figure 2) shows the following aspects:



	Table 2. The pore shape and dimensions								
No.	Name Pore shape Dimension								
1.	S1-PES+Ag		P=2.5 mm S=0.440 mm ²						
2.	S2-PES+PE		S=0.273 mm ² P=2.26 mm						
3.	S3-PES+PLA		PES S=0.272 mm ² P=2.92 mm PLA Monofilament P=3.89 mm						
4.	S4-2PP+Ag		P=2.64 mm S=0.502 mm ²						

- for the textile structures S1 and S4 where Ag threads are present, the percentage of this element is 49%, respectively 43%, followed by carbon (23-30%); the presence of Ag threads attracts the presence of the following elements: indium (10-8%) and titanium (5-4%);

- the textile structures S2 (PES + PE) and S3 (PES + PLA) have the highest percentage of the element carbon (65%), followed by oxygen (33-32%), silicon (2%) and titanium (1%).

-for the textile structures S1 and S4 where Ag threads are present, the percentage of this element is 49%, respectively 43%, followed by carbon (23-30%); the presence of Ag threads attracts the presence of the following elements: indium (10-8%) and titanium (4-5%);

- textile structures S2 (PES + PE) and S3 (PES + PLA) have the highest percentage of the element carbon (65%), followed by oxygen (33-32%), silicon (2%) and titanium (1%).







d. S4 - PP + Ag



In table 3 and fig. 3 are presented the values of biocompatibility: MTT, LDH and cell viability, for the composite textile structures.

Variant	Fiber composition, %	MTT	LDH	Cell viability, %
S1	95% PES 4,8% Ag	1,192	0,421	97,46
S2	68,5% PES 31,5% PE	1,078	0,405	88,15
\$3	97,6% PES 2,4% PLA	1,771	0,405	144,83
S4	34,1% PP 65,2% Ag (PA)	1,089	0,437	89,06
Control	-	1,22	0,37	100,0

Table 3. The values obtained for biocompatibility



Fig. 3. MTT, LDH and cell viability

- the best value of the cell proliferation indicator - MTT (1,771) is measured for the composite textile structure made of PES/ PLA which is superior to the control value of 1.22; this structure also possesses the lowest value of the cell death indicator - LDH (0.405) which is higher than the control value of 0.370; cell viability has a very high level (144.83%) higher than that measured for control cells (HT 29 cells in culture) of 100%.



- composite textile structure of PES/Ag, is next, having a MTT a value of 1,192 (comparable to the control - 1.22) and LDH of 0.421 (higher compared to the control - 0.370) so that cell viability is at 97.46%, comparable to that of control (HT 29 cells in culture) of 100%.

The composite textile structures of PES/ PE and PP/ Ag are at close both in terms of MTT (1.078 and 1.089 respectively), LHD (0.405 and 0.437 respectively) and cell viability (88.15% and respectively 89.06%) but below the value of the control cells of 100%.

5. CONCLUSIONS

- The composite structure with the highest mass (S4 - 2PP + Ag) of 158.9 g/m² has the highest values of breaking strength (551.3 N) and deformation resistance (471 kPa), but also the highest values of elongation (62.5% and 155.5%) and deformation (50.8 mm);

-The S2 composite structure (PES + PE) with the lowest mass (29.6 g/m²) shows good values of breaking strength (62.4 N and 74.3 N) and deformation resistance (98.4 kPa), smaller elongations (53.5% and 56.1%) and the smallest deformation 37.7 mm.

- Textile structures S1 and S4 belong to low Young's modulus meshes (<10.9MPa) and S2 and S4 belong to high Young's modulus meshes (>10.9MPa).

- The anisotropy is very good for all variants of textile structures, registering values <1.00.

- The best value of the cell proliferation - MTT indicator (1,771) is obseved in case of the composite textile structure of PES/ PLA which is superior to the control value of 1.22; this structure also has the lowest value of cell death indicator - LDH (0.405) ; cell viability shows a very high level (144.83%) higher than that observed in control cells (HT 29 cells in culture) of 100%.

ACKNOWLEDGEMENTS

This research was carried out within the "Advanced Textiles for Parietal Defects" project funded by UEFISCDI within the PNIII Program - European and International Cooperation – Sub-programme 3.2 - Horizon 2020, ERANET project type.

REFERENCES

[1] B.B. Agarwal, K.A. Agarwal and K.C. Mahajan, "Prospective double-blind randomized controlled study comparing heavy- and lightweight polypropylene mesh in totally extraperitoneal repair of inguinal hernia: early results", Surg Endosc, PubMed, Google Scholar, 2009; 23: 242–7.

[2] Ö. S. Aslantürk, "In Vitro Cytotoxicity and Cell Viability Assays: Principles, Advantages, and Disadvantages, Genotoxicity - A Predictable Risk to Our Actual World", Marcelo L. Larramendy and Sonia Soloneski, IntechOpen, 2017, DOI: 10.5772/intechopen.71923.

[3] D. K. Yip, N. Auersperg, "The dye-exclusion test for cell viability: "Persistence of differential staining following fixation", In Vitro, 1972;7(5):323-329.

[4] R. L. Ruben, "*Cell culture for testing anticancer compounds*", Advances in Cell Culture, 1988;6:161-188 Eds. Marqmarosh K., Sato G.H., Academic press INC, CAL.

[5] Stone V, Johnston H, Schins RPF., "Development of in vitro systems for nanotoxicology: *Methodological considerations*", Critical Reviews in Toxicology, 2009;39(7):613-626. DOI: 10.1080/10408440903120975.

[6] T.L. Riss, Moravec R.A., Niles A.L., Duellman S., Benink H., Worzella T.J. and Minor L., "*Cell viability assays*", 2013 Mat 1 (updated 2016 Jul 1). In: Sittampalam GS, Coussens NP, Brimacombe K, et al. editors. Assay Guidance Manual [Internet]. Betsheda (MD): Eli Lily & Company and the National Center for Advancing Translational Sciences; 2004.



CHARACTERISATION OF METAL THREADS FROM ARCHAELOGICAL TEXTILES RELIGIOUS BY METHODS OF ARCHAEOMETRY.

VORNICU Nicoleta¹, BIBIRE Cristina¹, ZALTARIOV Mirela Fernanda²

¹Metropolitan Research Center T.A.B.O.R, 9 Closca Str.,700066, Iasi, Romania, E-mail: <u>cmctaboriasi@yahoo.com</u>

²Petru Poni" Institute of Macromolecular Chemistry, 41A Grigore Ghica Voda Alley 700487 Iasi, Romania, E-mail: zaltariov.mirela@icmpp.ro

Corresponding author: Nicoleta Vornicu, E-mail: cmctaboriasi@yahoo.com

Abstract: The paper presents the experimental results regarding the study of the chemical nature and the state of conservation of the metal filters used in the decoration of two textile garments (stihar and chess) of archaeological origin, recovered from the tomb of Metropolitan Gavriil Banulescu Bodoni, located in Căpriana monastery, monastic settlement from the medieval period in the Republic of Moldova.

Textile and metallic yarns in the structure of liturgical garments were analyzed following a multi-technical, non-invasive approach, using optical microscopy, pXRF and FTIR to identify the type of yarn and their morphological characteristics, manufacturing techniques used for yarn production and metal composition / alloys. The major elements detected during the XRF analysis are gold, silver alone or combined, lead and copper. Gold is usually found as a surface coating film for silver wires and silver bands. All the wire threads studied are drawn, the surface of these threads either bears the characteristic striations from the draw plate or the signs of the rolling process. Silver-copper alloys were detected in the strips that were cut from a metal sheet, whether gilt at one side or not. All of the fabrics now appear more or less brownish or blackish due to the decomposition of the human remains, and maybe also the degradation of the natural dyestuffs used for the silks.

Key words: metal thread, silver layer, corrosion, OM, pXRF, FTIR.

1. INTRODUCTION

Textiles play an important role in the rituals of the Orthodox Church, they can be consecrated objects that officially serve the liturgical act or the funeral ritual according to the hierarchy. The clothes also carry messages that are reflected in specific colors and symbols. The use of metals to decorate textiles began in the Middle Ages and the analytical research of metal threads began almost 35 years ago. [1,2] Metals were used in the manufacture of liturgical textiles for structural (buttons, buckle buttons, fasteners) or decorative purposes. Precious metals, gold and silver were used to brighten textiles. The manufacture of gilded silver and gold threads involves several stages of work such as gilding, hammering, spinning, rolling and wrapping around a textile core of silk or cotton. [3,4]

The research of archaeological textiles can bring information about the materials from which they were made but can also contribute to the description of the social environment in which they were created. Historical textiles are made of materials accessible to authors at that time. Knowledge of



complex mechanisms of degradation of archaeological textiles is essential for the correct application of conservation-restoration techniques.

The typology of alterations / degradations (physico-chemical) and the state of conservation of archaeological textiles depends on the following factors: the burial environment, the nature of the fibers and the microclimate existing at the opening of the site. For many centuries, Orthodox bishops and bishops were buried dressed in liturgical vestments to emphasize the identity of the deceased. Tombs identified during archaeological excavations can have a variety of preservation conditions, clothing usually survive for a long time only when processed with metal wires.

The analysis of such textiles from archaeological contexts is performed using optical microscopy and electron microscopy, X-ray fluorescence spectrometry, FTIR spectroscopy, microstratigraphic description and the like. [5,6]

The paper presents the results of physical-chemical investigations of textile and metallic yarns in two archaeological textiles (sack and stihar), extracted on August 25, 2016 from the tomb of Metropolitan Gavriil Bănulescu-Bodoni from Căpriana Monastery, Republic of Moldova.



Fig.1. Stihar



Fig.2. Sacos

2. MATERIALS AND METHODS

The purpose of these scientific investigations is to know the composition, structure and establishment of a diagnosis of the observed alterations that determined the state of conservation. The methods used for the scientific investigation of the textile materials studied are:

Macroscopic investigations

Macroscopic investigations refer to the types of physico-chemical and biological degradations of the textile material visible to the naked eye or with a magnifying glass due to microclimate and biological factors.

Photographic analysis (macrophotography, microphotography)

Traditional white light shooting techniques were used, with the light beam placed perpendicularly, using an Olympus digital camera and shooting software corresponding to the Olympus SZX 160 and U500XST2 microscopes. The photographic documentation obtained is used as a support in the interpretation of analytical data.

Optical Microscopy an UV

The microscopical researches have been carried out on an Olympus SZY 160 microscope correlated with program Quick Capture Pro 2.0 software and fluorescence illuminator RFA-16 and a Novex stereomicroscope Ap-8 Euromex at various magnifying powers up to the maximum 500 X.



XRF Spectrometry

We has use of XRF technique to know the elemental composition of the metals from which the objects are made. The experimental measurements were realized using an XRF spectrometer, type Innov X Systems Alpha Series (SUA). The analyzes carried out the presence of filters during the analysis, the absence of vacuum during the analysis, the absence of an inert gas flow.

Spectoscropie FT-IR

Fourier transform IR spectroscopy was performed with the Vertex 70 Bruker spectrometer, analyzing the composition of corrosion products and colored fibers in the structure of the analyzed archaeological textiles.

3. RESULTS AND DISCUSSIONS

The macroscopic analysis (visual, magnifying glass) found that the two pieces of clothing show the following types of destruction: tears, chamois, bends, stains (green, white, reddish), the fragility of the material, traces of earth and corrosion on metal accessories.

During the research by optical microscopy in incident white light, the presence of 4 types of threads was found: textile threads from fabric, textile threads from embroidery, metallic threads from fabric and metallic threads from embroidery. The textile yarns are made of natural fibers and are of a protein (silk) nature according to the results provided by OM-UV. Simple metal wires are tape or wire type and metal wires helically wound on textile core, tape type and wire type.



Table 1. Morphological characterization of textile yarns from the textile structure of Căpriana





The identification of textile yarns from embroidery and fabric was made based on the longitudinal microscopic appearance of the fibers obtained by defibrating the yarns in ultraviolet light. I noticed that in the case of the metal wire bag, they are in two colors, gold and silver, and in the case of silver, only silver. For the production of textiles, fabrics made of degummed natural silk yarns were used, which under a microscopic appearance have pigmentary inclusions suspected of being caused by active biological attack or traces of it. The presence of metal wires generates mechanical and chemical damage through corrosion products.

The technique of X-ray fluorescence spectrometry is widely used for a first determination of the nature of composite elements in the structure of ecclesiastical textiles.

Part name	Sample Cu Au Pb		Pb	Ag	Base metal	Coating film		
	Embroidery thread 1	-	-		100	Ag	-	
	Embroidery thread 2	-	-	0.10	99.90	Ag	-	
Sacos	Embroidery thread 3	0.75	-	-	99.25	Ag	-	
	Wire gallon 1	1.53	-	-	98.47	Ag	-	
	Wire gallon n 2	-	-	0.08	99.92	Ag	-	
	Fabric thread 1	1.59	-	-	98.41	Ag	-	
	Fabric thread 2	-	-	-	100.00	Ag	-	
	Wire little gallon	-	1.52	13.36	85.12	Ag	Au	
Stihar	Embroidery thread 1	2.28	2.94		94.78	Ag	Au	
	Embroidery thread 2	4.32	-	0.41	95.27	Ag	-	

Table 2. Composition of metal wires and accessories (XRF)



According to the results obtained, we identified the following types of metal wires: pure silver wires; silver wires with secondary elements (Pb); silver wires with secondary elements (Cu); silver wires with secondary elements (Cu, Pb); gilded silver threads. The determined elemental compositions indicate that all wires are silver-based and some of the investigated wires are contaminated with oxide and cupric chloride corrosion products.

The spectral analysis in the IR domain of the two textile samples taken in red and green was performed in order to know their nature.



Fig.3. Silk thread fabric, Sacos.



Fig. 4. FTIR spectrum of green threads



Fig.5. FTIR spectrum of red threads

As a result of the results obtained by interpreting the vibration spectra, he observes small variations in the vibration domains of the two samples, green wire and red wire.

Table 3. Results of the FTTR analysis (characteristic banas and their allocation		
Green thread	Red thread	
3283cm ⁻¹ ; v NH asymmetric (valence vibration)	3283 cm ⁻¹ ; v NH asymmetric (valence vibration)	
3069 cm ⁻¹ ;v NH symmetric (valence vibration)	3072 cm ⁻¹ ; v NH symmetric (valence vibration)	
2972 cm ⁻¹ ; v CH (valence vibration)	2962 cm ⁻¹ ; v CH (valence vibration)	
2928 cm ⁻¹ ; v CH ₂ asymmetric (valence vibration)	2924 cm ⁻¹ ; v CH ₂ asymmetric (valence vibration)	
2856 cm ⁻¹ ;v CH ₂ symmetric (valence vibration)	2854 cm ⁻¹ ; v CH ₂ symmetric (valence vibration)	
1626 cm ⁻¹ ;δ C=O (deformation vibration) (amide 1622 cm ⁻¹ ; δ C=O (deformation vibration) (amide		
I)		
1510 δ NH (deformation vibration) (amide II)	1514 cm ⁻¹ ; δ NH (deformation vibration) (amide II)	
1227 cm ⁻¹ ; v C-O + v C-N+ v C-C	1229 cm^{-1} ; v C-O + v C-N+ v C-C	
1068 cm ⁻¹ ; δ NH	1068 cm ⁻¹ ; δ NH	

Table 3. Results of the FTIR analysis (characteristic bands and their allocation

The presence of the groups -NH and - C = O clearly indicates the protein nature of these fibers, namely natural silk, a result confirmed by the morphological structure determined by OM.



The small differences between the experimental results are probably due to the degree of destruction and the degree of hydration of the protein chain.

4. CONCLUSIONS

The textile threads in the structure of the hierarch's sack and of the styrofoam are made of natural fiber, white silk and slightly yellowish. The metal accessories, the large buttons on the bags are based on Ag-Cu alloy covered with a thin gold envelope, and the small buttons are based on Cu-Ag alloy. The metal threads used in the sack and stitch fabric are based on Ag. At the level of the band-type threads in the braid and the embroidery of the styrum, the presence of a thin film of gold was identified. The determinations were performed in areas where the metal surface was with insignificant destruction in order to avoid errors due to possible corrosion products.

Two main types of metal threads were used in weaving and embroidery, namely threads and strips. Both have also been used in combination with organic yarns to produce more elaborate blended yarns. The wires and strips are wrapped around a fibrous core of plant origin, silk.

Based on their morphological characteristics we found that metal strips belong to two categories and were produced using two different techniques. The first type comprises strips with sharp edges, which have irregular width and variable thickness. These strips were cut from sheets of metal (Ag). The second type of tape was produced by flattening a silver wire. These strips have more rounded edges and a very uniform width along their length. The wrapped metal strips are based on the twisting of the tape around the fibrous core in S or Z formations. Most of the wrapped strips are twisted in an S shape. thread thickness and diameter, as well as the type and color of the organic core.

The presence of a small amount of copper or lead in the wires with a high silver content was probably made to strengthen the metal and to make it cheaper.

REFERENCES

[1] M., Járó, *Metal threads in Historical Textiles* in Molecular and Structural Archaeology: Cosmetic and Therapeutic Chemicals. NATO Science Series, Series II: Mathematics, Physics and Chemistry, G Tsoucaris & J Lipkowski (eds), 117, 2003, p. 163-178.

[2] E., Photos-Jones, A., Hendry, P., Kavasila, *SEM and TEM examination of Byzantine and Post-Byzantine metal threads in Greek Orthodox ecclesiastical textiles*, Archaeometry Issues in Greek Prehistory and Antiquity, Hellenic Society of Archaeometry, Y Bassiakos, E Aloupi & Y Facorellis (eds), Society of Messenian Archaeological Studies, (Athens), 2001, p. 429-434.

[3] N., Vornicu, C., Bibire, *Non-destructive XRF analysis of metals the ecclesiastical garments a 19-20 th century*, Art d'aujourd'hui, patrimoine de demain. Conservation-restauration de l'art contemporain", Colloque SFIIC (13es journées d'études), INP, Paris, 2009, pg 343.

[4] A., Karatzani, *Study and analytical investigation of metal threads from Byzantine/Greek ecclesiastical textiles*, X-Ray Spectrometry, 4, 37, 2008, p. 410–417.

[5] A.M., Hacke, C.M., Carr, A., Brown, *Characterisation of metal threads in Renaissance tapestries*, Proceedings of Metal, National Museum of Australia Canberra ACT, 2004, p.415-426.

[6] N. A., Sharairi, I. C. A., Sandu, Characterization of Dyeing Techniques of Late 19th Century Ottoman-Style Costumes from the Jordanian Museum of Cultural Heritage, Revista de chimie,vol. 69, 4, 2018, p.901.



VISUAL MAPPING STRATEGIES TO ORGANIZE, COMMUNICATE AND MAINTAIN DESIGN KNOWLEDGE

BELLEMARE Jocelyn

University of Quebec in Montreal (UQAM), School of Business and Management (ESG), Department of Analytics, Operations, and Information Technology (AOIT). Montreal (Quebec), Canada E-mail: <u>bellemare.jocelyn@uqam.ca</u>

Abstract: Responding to increasingly demanding customers, facing global competition and managing the life cycle of a product are all instances of problematic situations that force different players in the fashion, textiles and clothing industry to be more creative in their search for solutions. It therefore becomes in some cases a priority to implement simple systems in order to ensure the availability of tools and information, so that teams can react quickly to problems that slow down product development. The goal was to improve and facilitate the processes of innovation and clothing conceptualization while having a more reliable performance and a greater capacity for adaptation during the creative processes. Various tools have been developed over the past few years, such as mind mapping and several visual mapping tools in order to organize, communicate and maintain knowledge regarding collection design in the fashion industry. It is a practice open to organizing ideas and collaborating on a specific topic, all of this through a graphic representation that is creative and adapted to the development of thought. This article describes the characteristics and relevance of the application of visual mapping tools in the context of product development from a study with fashion designers. The results of this project confirm that the heuristic approach in a design process could potentially facilitate the marketing of fashion clothing products.

Key words: Fashion Apparel Industry, Product Development, Mind Mapping, Visual Management.

1. INTRODUCTION

Plenty of people would like to go into business and offer their own clothing collection on the market. But how should one envision this for people who do not yet have much experience in the fashion market and/or the fashion industry which seems to be very aggressive and ruthless at times to new players. This study was conducted in Quebec in the winter and spring of 2020, on a voluntary sampling basis from the various members, partners and collaborators of the École supérieure de mode at ESG UQAM. In total, 15 designers responded to a questionnaire and 10 interviews were conducted to complete the collected data. The consulted designers had a minimum of 5 years of experience, in order to ensure a minimal structure for the management of the improvement of the fashion product design. Respondents all participated in this survey on a voluntary basis. Of course, the participating designers were more likely to perform well and be well advanced in integrating design improvement practices. But since this portrait is part of a larger survey aimed at establishing benchmarks for all fashion manufacturing companies, this perspective only increases the impact of the good practices presented. It is therefore important to keep this aspect in mind when interpreting the results.



First of all, we therefore met the 15 fashion designers who wish to launch and/or reposition their own brand on the market, and this with a view to the next 5 years. For the experiment, we joined a group of three experts in illustration, design, pattern, tailoring, production and marketing/distribution in order to validate with them the various value-creating activities. The goal was to assess which steps were important for them, or even critical in the implementation to market a collection, and if there were any constraints and/or impediments on the development of their concepts. We found that one of the first problems (i.e. bottlenecks) at the start was with the design and the consistency in the development of a collection for a specific market. The implementation time was tedious and in some cases it became really difficult to make decisions in the stage of styling, selection and modification of models. We therefore focused on knowing and thus better understanding their vision of this issue. And it is in this context that we applied a heuristic perspective in order to better understand their product development and marketing process.

2. HEURISTIC APPROACH IN THE PROCESSES

The heuristic perspective applies to fashion designers because it is directly related to the art of inventing, of making discoveries while solving problems. An analytical approach that makes it possible to quickly reach conclusions in a context where companies must, more than ever, be as competitive as possible. The pressure is strong and is felt among designers, because the goal is more and more difficult to reach (through reduction of costs, distribution of work, management of the value chain and the supply chain, etc.) and all this must go through constant innovation (of the product, but also operational, managerial, marketing - commercial). This is how innovation therefore becomes an important pillar of the competitiveness of young companies. And it is in this approach that the concepts of cognitive maps, concept maps, and mind maps are diagramming techniques that comply with this perspective.



Fig. 1. Heuristic mind mapping approach for a new product development

As shown in Figure 1, a heuristic and innovation perspective allowing idea managers to develop new working methods, but above all an openness to new ways of thinking in order to be ever more innovative and therefore competitive. Remember that the concept of innovation is not



synonymous with technical or technological innovation in this approach. Indeed, innovation is the introduction of a new idea, the conception of a heuristic element that fits into this model by applying it to projects of great diversity.

Remember that cognitive mapping, mind mapping and conceptual mapping are three powerful visual mapping strategies for organizing, communicating and retaining knowledge in order to standardize the development process of the fashion product. They help us to present complex ideas and processes and recognize patterns and relationships, which could, in the first place, make it easier to tailor and SEO the brand DNA for a fashion designer, whereas cognitive maps, mind maps and concept maps look and feel similar. This similarity could sometimes even be a source of confusion. Indeed, there are several different ways to visualize a mental model. So we focused on the cognitive maps that could be very relevant in management within the industrial and creative sector of the fashion industry.

3. LITERATURE

The scientific reviews show that visual representations improve the learning process [1]. As a matter of fact, they make it possible to create a shortcut between the processing of information by the brain and real information [2]. In other words, visual representations demonstrate what one should mentally construct for oneself so that the brain does not have to create its own perception. Moreover, the latter can be easily biased or perceived in a different way by various individuals and this is why visual representation helps to avoid these interpretive biases. There are several visual tools such as photos, drawings, graphics and more. Another tool that helps facilitate learning and understanding is the mind map. How then should we properly apply the use of this tool for people who already have a creative approach, without hampering them too much?

It should be understood that a mind map is a flowchart-type representation that allows thinking to be reproduced through hierarchical links between different ideas [3]. The first works of Buzan, in 1970, make it possible to understand his observations on the functioning of the brain which works by association and visualization. The concept is to facilitate learning and understanding by representing the links between ideas [4]. This later becomes a very useful review tool [5]. Indeed, a study among students shows that the map is for 38.73% a predictor of learning [6]. It has many benefits, as it allows reflection, memory, and knowledge to be expressed and it allows creativity to be stimulated [5]. It is an excellent tool for diagramming, revision, learning, and it allows a better understanding and appropriation of the material under study [3]. It might be normal to confuse the concept of a mind map with that of Brainstorming, which is often used in industry. It is important to note the difference between the two however. Brainstorming is divergent, that is, it is used for the purpose of launching new, creative and random ideas on a central theme [7]. Brainstorming is often presented in the form of a list and can be imagined as a funnel that widens as more and more ideas are generated. Furthermore, the mind map is based on convergent thinking, where the attempt is more to give an answer, to provoke reflections and to establish connections between the previously suggested concepts, much more than to emit new ideas. It should be remembered that the mind map is a working tool that does not have a priority idea and neither does it have a vertex, because it is possible to create links between the concepts indefinitely [8]. As it is limitless it should be easier to integrate it into the concept of fashion product development in a distinctive design context.

4. DISTINCTIVE DESIGN

The fashion designer operates in an environment that is increasingly competitive and where it becomes difficult to source in smaller quantities, which seems to hamper product development.



Companies in the fashion and clothing industry face increasingly intense and efficient competition. The life cycle of products is shortening, the choice for consumers in terms of products and services continues to widen and companies are, therefore, constantly forced to innovate in order to position their leadership or to achieve profitability. The entry into the market of a global retail giant tilts the balance of power in the market for both luxury and fast fashion products. For the designer, adapting to this reality requires significant resources. The personalized and distinctive design apparel market which is beginning to take hold and structure itself by offering quality products at half the price in comparison with its direct competitors is a concrete example of this competition. Pressure from new entrants is a constant threat and also forces fashion designers to innovate constantly and efficiently and effectively. From this observation, we were interested in the use of the mind map to carry out our tests with our participants.

4.1 Conceptual experiments and applications

As part of the study we proceeded therefore with different applications such as MindManager, MindMeister, Wise mapping, Cmap Tools. The method of using the mind map was the same for everyone. Our goal was to have a simple and easily applicable method. The use of the mind maps was validated over a period of two months. It was also possible in some cases to do it on paper or online using applications such as Coggle, Mind 42. Figure 2 below shows some results.



Fig. 2. Creative thinking workshop and mind mapping experience within a quality approach

They all had to work on a horizontal plane in order to have the larger view rather than the high view. Then everyone had the choice to connect the secondary ideas, the tertiary ideas, and so on. These had to be hierarchical and classified by colour code or symbols of choice and mainly expressed in the form of keywords. Word choice was not evaluated in our case. Only the overview and connections of the mind map were evaluated as well as the means used to find solutions to solve their problems. Today's reality shows that fashion designers now face more complex challenges than ever before. Over the years companies have therefore had to equip themselves with better technological tools. These mainly consist of dealing with a much faster pace of work, a greater workload as well as a greater need for creativity than before.



5. DISCUSSION AND FINDINGS

Our study confirms that a large majority, or 70% of the designers, suggest using the mind map because it is a simple, quick and inexpensive way to set up. After a month of use and to our great surprise, we found in most of the mind maps characteristics such as price, assortment, range, type of distribution, not to mention the specific attributes that were related to the product, such as the material, colours and sizes offered.

80% of them confirm that the tool visually represents well the ideas, tasks and concepts related to their main subject. More than 30% of the designers confirm that the tool allows them in particular to standardize and clarify thinking in order to implement projects that can often turn out to be very complex, such as technical clothing. More than 40% of the participants affirm that the mind map is a way of considering all the possible solutions to a problem and allows them to develop a more objective point of view on situations which must sometimes be seen from different angles, as in the application of the implementation of a circular economy of responsible and ethical consumption.

Moreover, it seems to be uncontested that the mind map allows designers, among other things, to better collect, filter and organize information, to prioritize, to make more good decisions, to structure, to synthesize ideas and to work on several projects simultaneously. The study confirms that this allows them to develop their problem-solving skills while remaining significantly innovative and anticipating solutions to potential problems. Here is a list of advantages and inconveniences raised during our meetings.

Table 1. Observations and	l perceptions of the tool	by the participants
---------------------------	---------------------------	---------------------

Advantages and observations	Inconveniences and perceptions
 Improves employees' creativity. Transdisciplinary and collaborative approach (improves the productivity and efficiency of the work team). Human-focused (meets the real needs of customers and users). Experimental method (iterative) Reduces risk when launching the product or service on the market. Cost reduction through the use of models and prototypes Boosts innovation. 	 « Catch-all » approach. It's just a fad, magical thinking. Dedicated to an elite (of designers), who would be the only ones able to apply it (appearance of many consulting firms). Difficult to apply in large companies. Scepticism/Resistance from some business leaders It's a time-consuming approach.

Almost 20% of the respondents say that efficiency, productivity and profitability in terms of development time have increased significantly since using this approach over the past year. There seems to be a consensus on this modern way of organizing information and making connections which has many advantages and very few inconveniences.

The study confirms that the mind map therefore seems to remain a rudimentary tool that is easily understandable and usable both among young people in constant learning and among older people in the company, and this even at various hierarchical levels. One respondent affirms that since it does not require technology in itself, the mind map is a return to the basics which often allows one to see the picture a little bigger and to advance a little further than before while still keeping a focus on the goals to be reached.



6. LIMITS

However, our research has certain limitations which should be emphasized and which constitute many possible avenues of future investigation. First, for the sake of parsimony, we avoided incorporating other designer-related features that might have been relevant. Moreover, the fact that our research focuses on a single category of actors and that the start of the Covid-19 pandemic occurred at the same time also has an impact on the external validity of our results. The latter cannot be generalized, especially those which do not necessarily have the same characteristics as the product selected in our study. Also, although the number of respondents was small, the proportion of participants affected is still more than satisfactory and large enough to draw interesting conclusions.

7. CONCLUSIONS

In conclusion, we can say that the approach is effective and that it applies well to the fashion, innovation and conceptualization sector. Indeed this approach has proven itself in our case. The participants confirm that the tool makes it possible to standardize and clarify thoughts and implement projects that can often be very complex, as in the case of technical or even intelligent clothing. The tools produced in workshops as part of the approach have been an asset for fashion designers and have made it possible to get the best out of employees working on the fashion collection development project. However, we realize that this approach is not perfect, because it is not necessarily applicable for industries that work in silos and that have less time to give to employees to properly carry out the steps that prepare the ground. In short, visual mapping is a simple, fast and inexpensive way to visually represent ideas, tasks, words and concepts related to the development of fashion collections.

REFERENCES

[1] Yegan Pillay, Huan-Tang Lu & Levi Funk (2020) Introducing Mind Mapping as a Creative Counseling Modality, Journal of Creativity in Mental Health, 15:4, 474-481, DOI: 10.1080/15401383.2020.1746721

[2] Osborn, A. F. (1988). Constructive imagination - Créativité. Paris: Bordas.

[3] Régnard, D. *Apports pédagogiques de l'utilisation de la carte heuristique en classe, Éla*. Études de linguistique appliquée, vol. 158, no. 2, 2010, pp. 215-222.

[4] Buzan, Tony, and Barry Buzan. The Mind Map Book. London: BBC Books, 1995. Print.

[5] Buzan, T., Griffiths, C., & In Harrison, J. (2014). *Mind maps for business: Using the ultimate thinking tool to revolutionise how you work.* (2nd ed.). Pearson.

[5] Liedtka, J.; Salzman, R.; Azer, D. *Design Thinking for the Greater Good: Innovation in the Social Sector*; Columbia Business School Publishing: New York, 2017.

[6] Delengainge, X., Neveu, P., Vincenzoni, C. *Managez avec le Mind Mapping* : *illustrations de Franco Masucci*, Paris, Dunod, 2016, 196 p.

[7] Hariyadi S; Corebima A.D; Zubaidah S; Ibrohim S. *Contribution of Mind Mapping, Summarizing, and Questioning in the Rqa Learning Model to Genetic Learning Outcomes*. Journal of Turkish Science Education 2018, 15 (1), 80–88 DOI: 10.12973/tused.10222a.

[8] Castellano, S., Davidson, P. & Khelladi, I. Creativity techniques to enhance knowledge transfer within global virtual teams in the context of knowledge-intensive enterprises. J Technol Transf 42, 253–266 (2017). DOI:10.1007/s10961-016-9509-0



STRATEGIC VISION FOR RECOVERY OF MOLDOVA'S FASHION INDUSTRY AFTER COVID-19

GHEORGHITA Maria¹, NISTOR Doina²

¹Technical University of Moldova, Faculty of Economic Engineering and Business, Department Economics and Management, 41, Dacia bd., nr. 10 block of study, MD-2060, Chisinau, Republic of Moldova, E-Mail: <u>maria.gheorghita@emin.utm.md</u>

² Technical University of Moldova, Faculty of Economic Engineering and Business, Department Economics and Management, 41, Dacia bd., nr. 10 block of study, MD-2060, Chisinau, Republic of Moldova, E-Mail: doinitanistor@gmail.com

Corresponding author: Gheorghita Maria, E-mail: maria.gheorghita@emin.utm.md

Abstract: The Covid-19 pandemic situation has severely influenced both the European and Moldovan fashion industry, which is directly connected with the European one. For one year there have been essential changes in both the supply and consumption chains. Consumer preferences have changed, which has further underlined sustainable, ethical, and slow fashion. Requirements for social responsibility, accountability, and transparency, for the impact on the environment have amplified. In such conditions, it is necessary to review the vision of the industry both for the post-pandemic and in the medium- and long-term period. This paper reflects the current situation in the fashion industry of the Republic of Moldova, highlighting the impact of the pandemic on it. To overcome the situation generated by the pandemic Moldova's Light industry needs a new vision to account not only for the COVID-19 crisis but also the behavior and mindset of the new generation of consumer trends, such as Sustainable fashion, Slow fashion, Social fashion, Ethical fashion, for both its manufacturing services as well as the emerging domestic fashion brands. In the longer run, the industry needs to align itself to the new technologies, sustainability, digitalization, and constant innovation without harming the environment, since garment manufacturing remains one of the highest polluters in the world. Innovation, digitalization, and green economy remain key development goals for this industry.

Key words: Covid-19, sustainability, innovation, digitalization, recovery plan.

1. INTRODUCTION

According to the International Monetary Fund, 2020 is likely to be the worst year for the global economy since the Great Depression.[1].

After travel and tourism, fashion and luxury together are the most negatively impacted of all consumer goods and services, as retail stores shutter and consumer purchasing shifts to necessities. From April to May 2020, sales decreased by 60-70% in the worldwide fashion and luxury industry – with footfall in retail and recreation stores down by 44% in the U.S., 52% in Germany, 78% in India, and 59% in Brazil [2].

The Global Fashion Index analysis provided by McKinsey showed a 90 percent decline in economic profit in 2020 for fashion companies after a 4 percent rise in 2019 [3]. Effectively all garment manufacturing companies have experienced decreased revenue, negative impacts on orders



and their profit throughout the pandemic. The sector also has also been one of thehardest hit by negative unsupportive customer action, with almost a quarter of companies reported experiencing payment terms adjusted in the costomer's favour and discounted prices [4].

This correlates with global and EU experiences in the garment sector, with retailers closing and millions of workers left without work and in precarious positions because of the pandemic. A similar situation is observed in the fashion industry of Moldova. The COVID-19 pandemic has disrupted the Moldovan Light industry like never, and this industry has suffered more than most from the outbreak's social and economic effects [5]. As a result of the China-driven supply chain disruption and consumer market shutdown, Moldovan apparel factories have struggled with a severe reduction of manufacturing orders from their European clients. Many factories ceased work and sent their employees on forced vacations. Although factories have survived the lockdown, but many of them encountered drastic economic setbacks through the end of 2020.

2. CURRENT SITUATION IN THE MOLDOVAN FASHION INDUSTRY

Advantages of the Customs Union, proximity to the European market, flexibility and speed of production organisation, availability of qualified human resources fosters Moldova as a competitive player. The Fashion Industry in the Republic of Moldova (cooled Light Industry), which includes textiles, apparel, footwear, and fashion accessories, represents a key industry of Moldova's economy, accounting for about 14% of the total exports of the country and 5% of GDP in 2019, with a constant yearly increase since 2015 of about 8% in domestic and export sales. At the beginning of 2020, the Light Industry was at the top of the three exporters with an export value of 379.2 million US dollars. The main export market for the Moldovan light industry is the European Union market, which means that it is heavily influenced by changes in this market.

Altogether, in 2019 there were approximately 680 enterprises located throughout the country. Most of the production is carried out by micro, small and medium-sized enterprises, which are very flexible regarding the size of orders (figure 1).



Fig.1. The structure of Moldovan Light Industry enterprises [5].

Due to the highly labor-intensive nature of the industry, it remains a large employer in Moldova employing approximately 26,620 Moldovans, the majority of whom are female and youth from rural areas.

According to the business carried out by Moldovan light industry enterprises, currently 75% of the number of enterprises provide C&M, CMT and FOB services, about 20% operate only under their own-brand and about 5% of the total number of enterprises have a model of mixed business, it


provides services for foreign clients and produces under their own-brand. During the last years, the share of enterprises that produce and sell under their own brand is increasing.

The year 2019 was an unfavorable year for service providers, a year in which for the first time in the last 5 years there was a decrease in exports by about 11%. This trend has worsened further in 2020 due to the COVID outbreak.

In March 2020, a state of emergency dictated by the COVID-19 pandemic was announced, which lasted until the end of May. To assess the influence of the pandemic situation on the industry, the APIUS association with the support of the Moldova Competitiveness project, financed by USAID, Sweden embassy and UK aid (MCP) conducted a survey at the end of May 2020, which showed that 70% of factories reported up to 70% drop in sales creating serious financial constraints, 25% of the companies have suspended their economic activity, of which 5% went into technical unemployment. The number of employees within the Light Industry has decreased on average by 33%. Moldova's apparel exports in the second quarter of 2020 fell from 85897,2 mil US\$ to 62585 mil US\$ or by more than 38 percent, and the outlook is gloomy, dependent on the recovery of the heavily disrupted and slashed European fashion industry and market (figure 2).



Fig, 2. Exports in year 2020, Mil US\$ [5].

Moldova's top business partners in Italy, Germany, France and Romania reduced manufacturing orders by 35 to 60 percent. EURATEX, the European Apparel and Textile Confederation, reports a 35% fall in its clothing sector turnover.

After the state of emergency was removed, both in Europe and in the Republic of Moldova, orders began to arrive. However, the value of exports in 2020 constituted 339 Mil. US\$ or only about 89% compared to 2019 (figure 3).



Fig. 3. The evolution of export [5]



As a value, the export in 2020 was approximately at the level of yer 2015. Thus, the industry was thrown away 5 years ago. To survive and maintain the staff, many companies have made, and some of them continue to make masks and protective medical equipment.

A similar situation was with the sales on the local market. These are about 77% compared to 2019. Many companies have turned to e-commerce, but the results are still modest. The beginning of the year 2021 shows that it will be a rather difficult year both for companies that provide services for international clients, as well as for companies that manufacture and distribute under their own-brand on the local market. Thus, the outlook is gloomy, dependent on the recovery of the heavily disrupted and slashed European fashion industry and market.

3. THE RECOVERY VISION OF MOLDOVA'S FASHION INDUSTRY IN THE POST-CORONA ERA

As a rapid response to the COVID-19 pandemic, MCP has been supporting the Moldovan light industry to cope with the crisis, understanding impact, secure international orders, tapping into e-commerce, and define a local resilience strategy for medium-term recovery. Moldova's Light Industry should focus on capitalizing on the industry growth over the recent years (including its shift to high value-added) while re-strategizing and preparing for a changing industry after COVID such as:

- Protecting critical assets, such as safeguarding employees (and their livelihoods), value chain partnerships and international clients, while building the trust of consumers. Mitigate order cancellation from existing EU clients, and re-position/remarket to resilient EU clients/brands in competitive fashion segments.
- Integrate sustainability through the industry and business recovery strategies and make it central to post-pandemic decision making.
- Increasing the export of sustainable fashion under the own-brand in both the regional and the European market to ensure a high added value. Investments in some Moldovan brands with high potential to become well recognized both on the domestic and foreign market.
- Taking advantage of digitalization, innovative business models and automation to accelerate efficiency and competitiveness of factories, as well as international trade and e-commerce (or eCommerce). Prepare for re-investment and upgrade of factories.
- Accelerate transparency and fair trade to demonstrate positive environment and social impact by domestic fashion manufacturers and designers, while positioning Moldova as an ethical and sustainable fashion manufacturing destination.

Moldova's recovery plans need to be aligned with its key market for fashion manufacturing services, the European Union. The European Apparel and Textile Confederation EURATEX on February 2, 2021, responded with clear ideas to the roadmap for a European Textile Strategy. The EC initiative should push the textile and clothing industry to be more competitive, innovative, and sustainable [7].

Moldova's Light industry's new vision needs to account not only for the COVID-19 crisis but also the behavior and mindset of the new generation of consumers that place a stronger emphasis on sustainability. Moldova's Light industry needs to connect to consumer trends, such as Sustainable fashion, Slow fashion, Social fashion, Ethical fashion, for both its manufacturing services as well as the emerging domestic fashion brands. A few Moldovan designers have been embracing slow fashion and sustainable fashion values; however, a stronger movement needs to be created in the sector.



In the longer run, the industry needs to align itself to the new technologies, sustainability, and constant innovation without harming the environment, since garment manufacturing remains one of the highest polluters in the world.

Innovation, digitalization, and green economy remain key development goals for this industry, and although there are pilot initiatives and business champions embracing these trends in Moldova, still efforts must be made for the industry to innovate at its core and adopt these trends industry-wide [8].

During the last five years, MCP supported the industry to advance technological innovations, factory automation and digitization, incentivizing factories to upgrade via purchasing high precision equipment, CAD-CAM for automation of pattern design and cutting, printing and embroidery machines, special machines, etc. The MCP SMART factory program assisted factories to implement production engineering and Lean Management tools supporting factory efficiency and its ability to compete in terms of production costs. This direction must be continued.

Digitalisation of the Light Industry may take place in different forms, from digitalisation of the value chain to the utilisation of new technologies in manufacturing. Improving the digital transformation of the sector is crucial for the Republic of Moldova because the industry is facing a low level of productivity, and a high level of digitalization is one of the main factors for increasing productivity.

Another main challenge of the Light industry is keeping up with the quality standards required for the integration of Moldovan enterprises into the global value chains and enhancement of exports. Major large-scale European buyers set certain prerequisites on the production of suppliers, among which social compliance and environmental standards are the pioneering ones. With MCP support, more than 20 factories implemented Integrated Management Systems and social compliance certifications, ensuring better working conditions, and attracting quality conscious European buyers. To maintain the economic relations that Moldovan enterprises have, they must continue to adapt to the social and environmental conditions put forward by the main actors.

Taking into consideration that SMEs constitute the largest share of the total number of Moldovan Light Industry enterprises, there is an important challenge, to understand, and to undertake necessary transformation, to foster their adaptation to the evolving global standards, decrease their input costs and increase export capacities.

After the pandemic, factors such as quality and durability will become more important, given expected economic uncertainty and high levels of unemployment among consumers. It is also important to note that the underlying drivers for environmental transformation on the manufacturing side (namely that environmental pollution and inefficient processes are simply unsustainable in the long term) will persist throughout the current crisis. The reduction in human mobility and consumption caused by shelter-in-place and lockdowns reveals the benefits of environmental renewal as cleaner air and restored [2].

In order to address these issues in Moldova and to create and support the opportunities mentioned above for Light Industry enterprises, a special role must be played by the APIUS association. APIUS as a sectoral association must assume the role of the leading association in the sector in the transition to superior digital skills and to help SMEs adapt to the needs of global standards and to meet the European customer's requirements.

Moldova's light industry recovery strategy in the post-Corona era calls for a shared vision, underpinned by strong coordination and partnership among industry stakeholders and development partners. This document should be practical and actionable. It should be a clear and goal-oriented strategy, translating the mega-trends and EURATEX vision to Moldova's situation.



4. CONCLUSIONS

The Light Industry, which includes textiles, apparel, footwear, and fashion accessories, is a key industry of Moldova's economy, being in the top three eaportrs. COVID-19 pandemic has disrupted the Moldovan Light industry like never before. COVID outbreak negatively influenced the export trend of the Moldovan garment industry, contributing to a reduction by over 13%.

Moldova's Light industry needs to rethink its forward-looking strategy and understand where to go next after (and during) COVID-19, how to adapt, how to fit into the new reality of the EU fashion market/industry, and the global one. It needs a clear strategy to describe the position of the Moldovan Light Industry in the future to prioritize business environment improvement actions, as well as actions that would allow enrolling of the light industry of Moldova in regional and global changes in the fashion industry and supply chain, define what Moldova needs to do to maintain and increase competitiveness and preserve jobs, and form the foundation to advocate for a needed policy or regulatory changes.

Moldova's recovery plans need to be aligned with its key market for fashion manufacturing services, the European Union. The European Appael and Textile Confideration EURATEX onboarded to develop a new strategy for recovery and long term resilience built on fair trade, sustainability and digital transformation.

In the longer run, the industry needs to align itself to the new technologies, sustainability, and constant innovation without harming the environment, since garment manufacturing remains one of the highest polluters in the world. Innovation, digitalization, including e-commerce and green economy, remain key development goals for this industry.

REFERENCES

[1] The Great Lockdown: Worst Economic Downturn Since the Great Depression [Online]. Available <u>https://blogs.imf.org/2020/04/14/the-great-lockdown-worst-economic-downturn-since-the-great-depression/</u>

[2] Rebuilding a More Sustainable Fashion Industry After COVID-19. [Online]. Available: <u>https://apparelcoalition.org/wp-content/uploads/2020/04/Weaving-a-Better-Future-Covid-19-BCG-SAC-Higg-Co-Report.pdf</u>

[3] The State of Fashion 2021, BOF McKinsey&Company [Online]. Available: <u>https://www.mckinsey.com/~/media/McKinsey/Industries/Retail/Our%20Insights/State%20of%20fa</u> <u>shion/2021/The-State-of-Fashion-2021-vF.pdf</u>

[4] Just-style newsletter. [Online]. Available: <u>https://www.just-style.com/news/garment-manufacturers-remain-hardest-hit-by-covid-19_id140606.aspx</u>

[5] Statistica.md. [Online]. Available:

 $\underline{https://statbank.statistica.md/pxweb/pxweb/ro/40\% 20 Statistica\% 20 economica/?rxid=b2ff27}{d7-0b96-43c9-934b-42e1a2a9a774}$

[6] Roadmap for an "EU Strategy for Textiles". [Online]. Available: <u>https://euratex.eu/139/euratex-for-comprehensive-eu-textile-strategy/</u>

[7] Trends in Apparel, [Online]. Available: <u>https://www.cbi.eu/market-information/apparel/trends</u>



STUDY ON THE MECHANICAL PROPERTIES OF LEATHERS REINFORCED WITH SUPPORTING MATERIALS

ÇOLAK Selime¹, KAYGUSUZ Meruyert²

¹Pamukkale University, Denizli Vocational School of Technical Sciences, Traditional Handicrafts Department, Denizli/Turkey, E-mail: <u>scolak@pau.edu.tr</u>

²Pamukkale University, Denizli Vocational School of Technical Sciences, Textile, Apparel, Footwear and Leather Department, Denizli/Turkey, E-mail: <u>meruyert_k@hotmail.com</u>

Corresponding author: KAYGUSUZ Meruyert, E-mail: meruyert k@hotmail.com

Abstract: Depending on the design chosen for the production of leather goods, leather can be used alone or together with various supporting materials which give form and strength to it. Reinforcements are extremely important component of leather goods whether wallet or purse, handbags or suitcases, belts or portfolio. Reinforcements which provide support to the leather are used to change the drape and handling of leather goods. The physico-mechanical properties of the leather goods may vary depending on the supporting materials, in particular, salpa and syntex with different thicknesses were examined. For this purpose, the measurement of thickness, tear load, tensile strength and percentage extension have been performed. The highest tensile strength and tear load results were obtained for the leather used with syntex 1 and syntex 2 as 23.5 N/mm² and 397.5 N, respectively. In spite of high values of the percentage extension of syntex 1 and syntex 2, these values were decreased when they were used with leather. It was also determined that the tensile strength and tear load values of the leather. It was also determined thickness of salpa materials.

Key words: Leather, Leather handicrafts, Reinforcement materials, Salpa, Syntex

1. INTRODUCTION

Leather plays an important role among handicrafts and industrially manufactured products. All kinds of bags, luggage and other travel items with different functions, sizes, structures and different materials used together, and small items such as belts, desk pads, wallets, key rings, accessories etc. in other words articles made of leather are considered as leather goods [1]. The physical properties of leather constitute the important quality parameters that determine the performance characteristics in their areas of application [2]. In leather goods production, the leathers that do not have all the desired properties and do not give the desired form alone are reinforced with different supporting materials. Many of the designs would not be possible and most of the modern leather goods would be soft and shapeless without reinforcements [3]. The choice of supporting materials varies according to the product, its model, visual and physical harmony of the product design and the technical features (durability, etc.) it will bring to the applied area. If the leather you choose has too much drape or is too pliable, then you can consider laminating reinforcement to the back of the leather to change its hand.



A good reinforcement to consider is salpa which is a bonded leather fiber material [4]. Salpa or bonded leather is made from leather shavings and leather pieces obtained as waste in the leather industry. It is produced in layers by milling of vegetable leather scraps as well as chrome shavings, mixed with fat and a binding agent (latex) [5]. Salpa gives to the leather features such as appearance, touch and stiffness. Another option is syntex (or microbase) which is a microfiber reinforcement material which is lightweight, durable and flexible [6]. Both of these materials (salpa and syntex) sold in different thicknesses ranging from 0.5 mm to 2.0 mm.

Leathercraft and leather goods production constitute an important branch of the leather industry. Leather exhibits different physical behaviors depending on the differences in production and the chemicals used. Unlike most textile materials, leather has irregular fiber structure [7] which makes leather goods differ in terms of mechanical properties [8]. Even the natural fibrous structure of leather gives its unique physical properties of handle and ability to accommodate to the stresses and forces subjected during its use [9], the physico-mechanical properties of the leather goods may vary depending on the supporting materials used, since these materials also have different mechanical properties. There is a dearth of information on the mechanical properties of the reinforcements used with leather. Therefore, this study was performed to understand and select proper material for a particular application and to learn which combination has better performance and durability.

2. MATERIALS AND METHODS

2.1 Materials

In the study, calf leathers (Sepiciler, Turkey) processed for leathercraft were used. Syntex (SF Trade Leather, Turkey) with two different thicknesses (syntex-1 and syntex-2) and salpa (SF Trade Leather, Turkey) with three different thicknesses (salpa-1, salpa-2 and salpa-3) were used as supporting materials in the production of leathercraft samples.

2.2 Methods

Sampling and conditioning

After measuring the thicknesses of leather, salpa and syntex samples, the leather and the supporting materials were bonded with the same type of adhesive. All the specimens have been prepared in a certain amount for the physical tests to be applied in three parallel. The test samples for the physical tests were taken from the sampling locations as specified in ISO 2418, and conditioning has been performed according to ISO 2419 at $23 \pm 2^{\circ}$ C temperature and $50 \pm 5\%$ relative humidity [10,11].

Measurement of physico-mechanical properties

The thickness of the test samples was measured using a Satra (UK) thickness measuring device in accordance with the ISO 2589 standard [12]. Physical properties of leather and supporting materials were tested according to ISO 3376 using Shimadzu AG-IS (Japan) device [13]. The leather and supporting materials for the tear load test were prepared by cutting the samples in the dimensions specified in the standard using the steel template cutting blades according to ISO 3377-1 [14].

3. RESULTS AND DISCUSSION

The mechanical properties of leather are expected to be sufficient to the stresses applied onto the leather in the process of creation as well as physical changes in the material which result



from the application of force on product during the usage [15]. For this reason, strength properties are very important in terms of technological and the production knowledge. High tensile strength is one of the most desired features in finished leathers and leather goods. In addition, it is very important that the elongation is at a sufficient level in terms of the fact that there should not be any deformation on the grain and the fibers should not deform irreversibly in the case of pulling the leather into the mold or flexing and bending. Since leather belongs neither to the category of 'textiles', nor to the category of more rigid materials, such as cardboard, plastic or acrylic, and it is at the same time soft and flexible, as well as sturdy and strong [16], its incorporation or combination with flexible or foldable displays or support structures should be investigated to reveal the appropriate structures which would provide the properties that are required from different leather goods. Tensile strength and percentage extension test results of leather, supporting materials and the leather reinforced with these materials obtained in this study are given in Tables 1 and 2.

Materials	Tensile Strength, N/mm ²	Extension, %
Leather	22.7±0.7	58.6
Syntex-1	14.7 ± 1.1	93.5
Syntex-2	18.3±1.7	103.8
Salpa-1	17.1 ± 1.2	39.6
Salpa-2	6.9±1.4	120.9
Salpa-3	11.5 ± 2.2	35.7

Table 1 Tensile strength and extension values of the investigated materials

	Table 2	Change in the	e tensile strength and	l extension of the	leather used with	supporting material
--	---------	---------------	------------------------	--------------------	-------------------	---------------------

Tensile Strength,	Extension,
N/mm ²	%
23.5±2.3	49.5
17.5±0.7	53.4
15.6 ± 3.8	63.6
$16.5 \pm 1,5$	61.5
16.7±2.1	69.7
	N/mm ² 23.5±2.3 17.5±0.7 15.6±3.8 16.5±1,5

When the results of the study were examined, it has been observed that the physical tests of leather and supporting materials used give different results depending on whether they are used alone or together (Table 1-4). Calf leather has the tensile strength and percentage extension values as 22.7 N/mm² and 58.6%, respectively. Syntex-1 has the tensile strength of 14.7 N/mm² and 93.5% of extension, but when leather is used with syntex-1 the tensile strength increases (23.5 N/mm²), while the percentage extension value decreases (49.5%). Thus, while the tensile strength of the product increases, the problems caused by extension will decrease.

The tensile strength of syntex-2 is 18.3 N/mm², but when it was adhered to leather the tensile strength has been determined as 17.5 N/mm². It has been found that the tensile strength is slightly lower when syntax-2 used with leather. Percentage extension of syntex-2 is 103.8%, this value decreases to 53.4% when it used with leather. The reason for the low strength is thought to be due to the thickness of syntex-2 being thicker than syntex-1.

Salpa-1 alone has the tensile strength of 17.1 N/mm² and the extension of 39.6%. When it was bonded to leather, it has 15.5 N/mm² and the extension of 63.6%. It has been observed that when salpa-1 is attached to leather, the tensile strength decreases and the extension increases. While the tensile strength of salpa-2 was 6.9 N/mm² and the extension was 120.9%, when leather and salpa-2 were adhered the tensile strength was determined as 16.5 N/mm² and the extension was



determined as 61.5%. Vice versa situation has been observed in comparison with salpa-1 + leather as the tensile strength of leather + salpa-2 was increased and the extension was decreased.

When tested alone salpa-3 had tensile strength as 11.5 N/mm^2 and the extension as 35.7%, when leather and salpa-3 were used together, the tensile strength was found as 16.7 N/mm^2 , the extension was determined as 69.7%. It was determined that the tensile strength values are increased with the increase of salpa thickness. The same could not be said regarding the extension values. However, the mean value of the tensile strength of the leather used with the salpa materials with different thicknesses is around 16.3 N/mm^2 and the mean extension is 64.9%.

UNIDO has advised tensile strength min 20 N/mm² for acceptable quality of calf leathers [17]. It is stated that the tensile strength value should be at least 22.5 N/mm² and 25 N/mm² in vegetable and chrome tanned leathers for bags, luggage and saddlery [18]. These leathers with thickness over 1.25 mm should have the tensile strength value 15 N/mm² at least [19]. When the tensile strength values of the leather and reinforced leathers are evaluated, it was seen that the obtained values are in the range of the acceptable limits.

In a study, hybrid leather (laminated Permair leather, thickness 1.7 mm) obtained by hot plate pressing of a separately moulded microporous polymer film to split leather surface using adhesive layer had tensile strength value as 21.9 N/mm². The lamination of split leather by the microporous polyurethane film improved tensile property of the hybrid leather [20]. It was also reported that when sheep nappa leather was fused with the interlining having nylon, the tensile strength and tear load of this leather were increased [21].

When the leather is under the effect of a force, it first shows elongation, and then it comes to the rupture stage and the elongation increases at this stage. In addition, while less elongation causes the leather to tear easily, excessive elongation can cause deformation of the leather. The elongation value of the vegetable tanned leather for bags, suitcases and sandals should be max 50% [19]. In other reference, the percentage elongation of the vegetable and chrome tanned leather for saddlery and bags in thickness of 2 - 4 mm should be 50% and 75% maximum [18]. If the elongation value is higher than the desired values, it may cause deformations and deformities. It is seen that the elongation or extension values in this study are within the maximum values specified in the mentioned studies.

Material	Tear load,	Thickness,
	Ν	mm
Leather	271.1±0.3	1.9±0.05
Syntex-1	54.5±0.7	$0.4{\pm}0.01$
Syntex-2	162.2±0.3	1.1 ± 0.01
Salpa-1	8.4±0.2	$0.4{\pm}0.01$
Salpa-2	44.9±0.5	0.7 ± 0.01
Salpa-3	21.2±0.4	0.9 ± 0.02

 Table 3. Tear load and thickness values of leather and supporting materials

Table 4. Tear load and thickness values of the leather reinforced with supporting material.
--

Materials + leather	Tear load,	Thickness,
	Ν	mm
Syntex-1 + leather	299.0±1.5	2.3±0.02
Syntex-2 + leather	397.5±1.2	2.9±0.07
Salpa-1 + leather	239.3±0.6	2.4 ± 0.05
Salpa-2 + leather	253.5±0.7	2.7 ± 0.02
Salpa-3 + leather	257.6±0.3	2.8±0.01



When the tear load of the leather (thickness 1.9 mm) was found as 271.1 N and the tear load of syntex-1 (thickness 0.4 mm) as 54.5 N, the tear load value for the bonded leather and syntex-1 (thickness 2.3 mm) was determined as 299.0 N. It means that when the leather will be reinforced with syntex-1, the tear load of the product will increase. While the tear load of syntex-2 (thickness 1.1 mm) was 162.2 N, the tear load of the syntex-2 adhered to leather (thickness 2.9 mm) was found to be 397.5 N. Thus, when leather and syntex-2 are adhered, the tear load of the obtained product will also increase. The tear load values of salpa-1, salpa-2 and salpa-3 were determined as 8.4 N, 44.9 N and 21.2 N, respectively. The tear load values for the leather reinforced with these materials were found to be 239.3 N, 253.5 N and 257.6 N, respectively (Table 3, 4). It was observed that the tear load values are increased with the increased thickness values of the leather adhered with salpa materials (2.4 mm, 2.7 mm and 2.8 mm).

Tear load is one of the strength properties sought in order to know the resistance of the finished leathers against tearing under any force effect during their use. The tear strength of the leather can vary with the natural structure of the leather, the processes applied and the type of tanning [22]. It was stated that the tear strength depends on the thickness and type of the leather [23]. It has been reported that tear load of shoe upper leather should be of 30 N [17]. It is seen that the tear load values determined in this study are higher than the acceptable values. For this reason, it can be interpreted that the risk of tearing of the reinforced leather is low in the face of the stress and tensile effects that may arise during both the manufacture of leather goods and during use.

4. CONCLUSIONS

Supporting materials are all materials that facilitate the durability, appearance and production of the goods. The quality of the material used in the production of leather goods affects the price as well as the durability of the product and its market position. Depending on the thickness variation of the supporting materials used, it was determined that the tensile strength, percentage extension and tear load properties of the samples differ when the leather and the supporting material were used together. The best physico-mechanical properties results were obtained for the leather used with syntex 1 and syntex 2 materials in terms of the tensile strength and tear load as 23.5 N/mm² and 397.5 N, respectively. In spite of high values of the percentage extension for syntex 1 and syntex 2, these values were decreased when they were used with leather and the best percentage extension result was found for the leather used with syntex 1 as 45.5%. It was also determined that the tensile strength and tear load values are increased with the increased thicknesses of the leather adhered with salpa materials.

ACKNOWLEDGEMENTS

This study was financially supported by Ege University Scientific Research Projects Coordination Unit under the project number of 16 MÜH 069.

REFERENCES

[1] UNIDO, "Future trends in the world leather and leather products industry and trade", United Nations Industrial Development Organization, Vienna, 2010.

[2] K. M. Nalyanya, K. P. Rop, A. Onyuka, Z. Birech, A. Sasia, "*Effect of crusting operations on the physical properties of leather*". Leather and Footwear Journal, vol. 18(4), pp. 283-294, 2018.



[3] S. A. Natesan, "Manual for leather accessories and leather ware", Central Leather Research Institute, India, 2000.

[4] Anonymous, 2019. Using reinforcements in bag construction. [Online]. Available: https://leatherworkschool.com/2019/03/using-reinforcements-in-bag-construction

[5] Anonymous, 2019a. [Online]. Available: http://instyle.in/reinforcements

[6] Anonymous, 2021. [Online]. Available: https://www.lorenzinet.com/en/production/leather-reinforcement/nonwoven-reinforcement-syntex

[7] A. G. Ward, "The mechanical properties of leather", Rheologica Acta, vol. 13, pp. 103-112, 1974.

[8] V. Urbanija, J. Gersak, "*Impact of the mechanical properties of nappa clothing leather on the characteristics of its use*", Journal of the Society of Leather Technologists and Chemists, vol. 88, pp. 181-190, 2004.

[9] R. M. Nofal, "Water vapor permeability and dimensional stability of leather after cyclic mechanical treatments", International Journal of Multidisciplinary Research and Development, vol. 4(2), pp. 64-68, 2017.

[10] ISO 2418:2017, Leather - Chemical, Physical and Mechanical and Fastness tests - Sampling location, International Organization for Standardization, Geneva, 2017.

[11] ISO 2419:2012, Leather - Physical and Mechanical tests - Sample preparation and conditioning, International Organization for Standardization, Geneva, 2012.

[12] ISO 2589:2016, Leather - Physical and mechanical tests - Determination of thickness, International Organization for Standardization, Geneva, 2016.

[13] ISO 3376:2011, Leather - Physical and mechanical tests - Determination of tensile strength and percentage extension, Geneva, 2011.

[14] ISO 3377-1:2011, Leather - Physical and mechanical tests - Determination of tear load - Part 1: Single edge tear, Geneva, 2011.

[15] P. Kavati, Ch. Bangaru, A. Baran Mandal, "Sewability of sheep nappa garment leather", Research Journal of Textile and Apparel, vol. 18(2), pp. 49 – 55, 2014.

[16] V. Tsaknaki, Y. Fernaeus, M. Schaub, "*Leather as a material for crafting interactive and physical artifacts*", in Proc. of the Conference on Designing interactive systems (DIS'14). Association for Computing Machinery, New York, USA, 5-14, 2014.

[17] UNIDO, "Acceptable quality levels in leathers", United Nations Publications, Sales Nr. E.76 II. B.G., New York, 1994.

[18] G. John, "Possible defects in leather production", Druck Partner Rübelman GmbH, Hemsbach, 1997, 379 p.

[19] A. Toptaş, "*Deride kalite tespiti*", İstanbul Üniversitesi Dericilik Araştırma Geliştirme ve Eğitim Merkezi, İstanbul, 1998, 310 p. (in Turkish).

[20] V. Jankauskaitė, E. Strazdienė, A. Laukaitienė, "Stress distribution in polymeric film laminated leather under biaxial loading", Proc Estonian Acad Sci Eng, vol. 12(2), pp. 111-124, 2006.

[21] K. Phebe Aaron, "Investigations on physical properties of materials and their influence in leather apparel construction". PhD Thesis, University of Madras, Chennai, India, 2011.

[22] H. Özgünay, "Farklı Modifiksayon İşlemlerinin Meşe Palamudu Tanenlerinin Yapısında Meydana Getirdiği Değişimlerin Spektroskopik Yöntemlerle İncelenmesi". Ege Üniversitesi Fen Bilimleri Enstitüsü Doktora Tezi, 150 s, İzmir, 2005. (in Turkish).

[23] A. J. Harvey, "Footwear materials and process technology". Swiftprint Centre Ltd, New York, USA, 1986.



BEHAVIOUR OF IR REFLECTIVE DYES AND PIGMENTS ON DIFFERENT FATLIQUORED UPHOLSTERY LEATHERS

ORK EFENDIOGLU Nilay¹, YAKUT Fatih¹ MUTLU Mehmet Mete¹,

¹Ege University, Engineering Faculty, Department of Leather Engineering, Bornova, Izmir, Turkiye E-mail: <u>nilay.ork@ege.edu.tr</u>

Corresponding author: Mutlu, Mehmet Mete, E-mail: mete.mutlu@ege.edu.tr

Abstract: IR Reflective dyes and pigments are designed to make absorption in the visible region of the electromagnetic spectrum like the standard dyes but to reflect the Infra-Red region of the spectrum as a difference; which enables the items dyed with them to heat less for darker shades when exposed to sunlight. In this study, a standard acid dyestuff and an IR reflecting acid dyestuff are used on leathers produced with 4 different fatliquoring processes. They are finished with standard and IR reflective pigments respectively. Then reflection behaviors of the resulting leathers were measured with an IR spectrophotometer to visualize the working theory. Later, the fastness properties of leathers were tested to see if they are applicable to leather articles and resistant to wear. Spectrums showed that black IR reflective dyes and pigments stop absorbing and start reflecting light with wavelengths above 700 nm which results cooler leathers under solar radiation. Rubbing fastness results showed that leathers, treated with IR reflective dyes and pigments, had better results with a slight difference than other standard acid dyestuff groups, which proved the durability of this application during wearing. When different fatliquoring processes were compared, it was not seen significant differences in final colors, reflection properties, and rubbing fastness results.

Keywords: Leather, IR Reflective, Dyes, Dyestuff, Pigments

1. INTRODUCTION

The transformation of animal skin into leather is a complex process encompassing several steps, among which leather dyeing adds value to the material for market purposes [1,2]. The process of dyeing in the leather making is carried out to impart color and esthetics to tanned hides and skins. The behavior of dyes is primarily determined by the charge characteristics of both the dye and the leather to be dyed [3,4]. Black dyes and pigments occupy a pivotal position with the growing global demand in the textile, cosmetic, paper, and printing industries. Black-colored products absorb all the sun radiation including near IR and cause a heating problem. This results comfort problems for customers using black dyed leather items like motorcycle seats, motorcycle clothing, automobile steering wheels, seats, and sometimes black shoes. So reflecting the solar radiation in the infrared area of the radiation spectrum higher than 700 nm is a solution for the heating problem without affecting the visible color. For this reason, IR reflective dyes and pigments are designed [5]. Another important process of leather production is fatliquoring, which is done to give softness, elasticity, handle properties, and fullness to the leathers. The Fatliquoring process also affects physical and mechanical properties [6], such as strength, elongation, water absorption, water vapor permeability, gas permeability [7,8,9], thermal properties, and light fastness properties depending on the fatliquor type and amount used. The leather industry uses different kinds of fatliquors including either untreated or



emulsified oils, fats and fatty alcohols, paraffin waxes, mineral oils, olefins, processed hydrocarbons, sulfated and sulfochlorinated products [10,11]. In our previous study, it was found that waterproofing fatliquors decreased the moisture content and water absorption properties of leathers and resulted less heating of the leathers [5]. In this study; reflectance spectrums, rubbing, and light fastness properties of IR reflective dyes and pigments on leathers fatliquored with natural, synthetic, and waterproof fatliquors are investigated comparing with a standard dyeing and finishing process.

2. EXPERIMENTAL

2.1 Material

4 groups of leather samples, fatliquored with different fatliquoring materials as shown in Table 1, were used as material. These samples were divided into 4 pieces and 2 of them were dyed with standard dye and the other 2 samples were dyed with IR reflecting dyes. The IR reflecting dyes & pigments, the mixture of natural and synthetic fatliquors, and waterproofing fatliquors were products of TFL Turkey. Natural and synthetic fatliquors were products of Zschimmer & Schwarz. The leather samples dyed with standard acid dyestuff dye were finished with a standard upholstery finishing recipe while the IR reflective dyed samples were finished by using IR reflective pigments [5]. The experimental design can be seen in Table 1.

Table 1. Experimental Design							
Fatliquoring system	A - Standard Dyed	B - IR Reflecting &					
	Leather	Pigments Dyed Leather					
1- Combination of Selected Natural and Synthetic Fatty Substances (Lecithin)	1A	1B					
2- Synthetic Fatliquoring Agent	2A	2B					
3- Natural Fatliquoring Agents	3A	3B					
4- A Waterproof Fatliquoring System	4A	4B					

Table 1. Experimental Design

2.2 Method

Hunterlab UltraScan Pro color measurement spectrophotometer was used to measure spectral reflectance of leather samples between the wavelengths of 350-1050 nm, which includes near UV, visible, and near IR region of the electromagnetic spectrum.

Lightfastness tests of the leather samples were performed by using the Atlas Xenotest Alpha+ test instrument by using the test standard ISO 105-B02 (2014) [12]. Samples were exposed to artificial light under controlled conditions, together with a set of blue wool fabric test reference materials (no.1 to no.8). The color fastness is assessed by comparing the color change of the exposed and the unexposed regions of the test specimen when the test reference no.4 faded to grade 4 of the grayscale.

The CIE L*, a*, b* color coordinates of the samples were measured by using CM3600d Konica Minolta spectrophotometer with the respective parameters of D65 illuminant, wavelengths range of 360-740 nm, 5 mm diameter of measuring space, and 10° light incident angle. Color measurements were repeated after the light fastness test on the light-exposed areas of samples. The color differences of leathers before and after the light fastness test were calculated according to the CIE Lab-76 color difference formula [13].

Colorfastness is of critical importance while caring for the apparel. Color is vulnerable to changes due to abrasion, exposure to heat and light, laundering, and perspiration [14,15]. Bally Finish Tester was used to test colorfastness to cycles of to-and-fro rubbing of the leather samples in dry,



wet, and perspiration conditions by using standard felts according to test standard TS EN ISO 11640 (2013) [16]. The leather samples and the test felts were evaluated by using grayscales mentioned in the standards 423-2 EN 20105-A02 (1996) and TS EN ISO 105-A03 (2019) respectively [17,18].

3. RESULTS AND DISCUSSIONS

The visible light perceived by humans ranges from wavelengths of approximately 380–750 nm [19]. Absolute thresholds vary from person to person and according to viewing conditions. Wavelengths from approximately 10–380 nm are termed ultraviolet. The IR portion of the spectrum ranges from approximately 750 nm–1 mm, with near-infrared (NIR) ranging from 750–1400 nm [19]. Objects exposed to NIR radiation (such as from an alternate light source) absorb, reflect and transmit these photons to varying degrees [20]. The solar energy from the near-IR and IR region of the solar spectrum are responsible for the heat build-up of dark-colored leather articles [21].

The graphics of spectral reflectance of leather samples with standard and IR reflective dyeing applications are shown in **Fig.1-4**. The spectral reflectance data on different wavelengths of samples are shown in Table 2. It is seen that the leathers dyed with standard acid dyestuff made absorption at all wavelengths; while leathers with IR reflecting dyes & pigments made absorption only at the visible region and made reflection at the IR region of the spectrum. This behavior was the same for all leather samples which had 4 different fatliquoring processes.





Fig. 2. Spectral reflectances of 2A and 2B





Fig. 3. Spectral reflectances of 3A and 3B



Fig. 4. Spectral reflectances of 4A and 4B

		Wavelength (nm)							
	500	700	750	800	900	1000			
Group A	4.23	4.06	4.01	3.88	3.91	3.52			
Group B	4.35	4.92	26.78	59.65	77.02	84.01			

 Table 2. Means of Spectral reflectance data of leather samples in different wavelengths

Rubbing fastness (dry, wet, perspiration) test results of leather samples with standard acid dyestuff and IR reflecting dyes & pigments are given in Tables 3 and 4. When two tables were compared, rubbing fastness results showed that leathers, treated with IR reflective dyes and pigments had slightly better results.

	Leather			Felt			
	Dry	Wet	Perspiration	Dry	Wet	Perspiration	
1A	5	5	5	4/5	4	3/4	
2A	5	5	5	4/5	4/5	4	
3A	5	5	5	5	4/5	4	
4A	5	5	5	4/5	4	3/4	

Table 3. Rubbing fastness test results of leathers with standard dyes

Table	e 4. Rubbing fastness test results o	of leathers	with IR	reflecting	dyes & pigments

	Leather			Felt			
	Dry	Wet	Wet Perspiration		Wet	Perspiration	
1B	5	5	5	5	4/5	4	
2B	5	5	5	5	4/5	4	
3B	5	5	5	5	4/5	4	
4B	5	5	5	5	4/5	4	

Lightfastness results of leather samples with standard and IR reflecting dyes & pigments were given in Tables 5 and 6. On a typical scale, the Delta E value can range from 0 to 100. When the Delta



E value is between 0 and 1, the observer does not notice the difference [22]. All Delta E values of samples were found lower than 0.5 which we can conclude that both of dying systems have good lightfastness properties and the different fatliquoring materials used in the study had no negative effect on lightfastness.

		Before			After		ΛE
	L	а	b	L	а	b	
1A	24.699	0.234	-0.554	24.815	0.295	-0.650	0.163
2A	24.826	0.166	-0.566	24.965	0.228	-0.648	0.172
3A	24.693	0.175	-0.530	24.745	0.245	-0.643	0.143
4A	24.665	0.198	-0.528	24.835	0.273	-0.620	0.208

Table 5. Lightfastness test results of leathers with standard dyes

Table	6. Lightfastness test resul	lts of le	eathers with	IR re	eflecting a	dyes ð	k pigments

		Before		After			$\Delta \mathbf{E}$
	L	а	b	L	а	b	
1B	24.334	-0.030	-0.785	24.590	-0.025	-0.783	0.256
2B	24.503	-0.025	-0.791	24.543	0.020	-0.713	0.088
3B	24.165	-0.048	-0.725	24.473	-0.058	-0.708	0.085
4B	24.166	-0.053	-0.711	24.175	-0.010	-0.678	0.037

4. CONCLUSION

IR reflecting dyes and pigments can be used to make absorbance in the visible region of the light spectrum while making reflection in the near IR region. This provides less heating for darker shades for leather articles and so provides thermal comfort for users. In this study, this working theory of IR reflecting dyes & pigments was demonstrated with spectral reflectance graphics and spectral data of the samples.

Additionally, the fatliquoring technology which affects moisture content and thermal properties of leathers, thus the heating of samples was investigated. It was found that the experimented fatliquors do not affect either reflectance values or the fastness properties.

The dry, wet, and perspiration rubbing and lightfastness test results show that IR reflecting dye and finishing application does not have any negative effect on performance values, on the contrary, has slightly better results, and can be applied without affecting the rubbing and light fastness properties of leather items.

ACKNOWLEDGEMENTS

The authors would like to thank Ege University Research and Application Center of Textile and Apparel Manufacturing (TEKAUM) for IR reflectance tests and State Planning Organization (DPT) for the instrumental support (Project number 2007-DPT- 001).

REFERENCES

[1] A.A., Haroun and H.F., Mansour, "Effect of cationisation on reactive printing of leather and wool", Dyes Pigm., vol. 72, pp. 80-87, 2007.

[2] D., Pellegrini, M., Corsi, M., Bonanni, R., Bianchini, A., D'Ulivo, E., Bramanti, "Study of the interaction between collagen and naturalized and commercial dyes by Fourier transform infrared spectroscopy and thermogravimetric analysis", Dyes Pigm., vol. 116, pp. 65-73, 2015.

[3] P., Britten, R.G., Stosic, C.B., Wood, A., Hudson, "*Recent advances in grain damage coverage and color fastness*", JALCA, vol. 95, pp. 236-243, 2000.



[4] S.V., Kanth, R., Venba, G.C., Jayakumar, N.K., Chandrababu, "*Kinetics of leather dyeing pretreated with enzymes: Role of acid protease*", Bioresour. Technol., vol. 100, pp. 2430-2435, 2009.

[5] M.M, Mutlu, N., Ork, F. Yakut, "Effects of fatliquing process on leathers coloured with *IR reflective dyes and pigments*", Annals of the University of Oradea Fascicle of Textiles, Leatherwork, vol. 18(2), 155-160, 2017.

[6] A., Yorgancioglu, "*Emulsification and application of a thymol loaded antibacterial fatliquor for leather industry*", Journal of Industrial Textiles, online publish, 2020.

[7] X.C., Wang, Y., Li, H.J., Wang, B., Xu, "Preparation of modified hyperbranched polyglycerols and its application in fatliquoring", Fine Chem, vol. 32(12), pp. 1399-1401, 2015.

[8] K.H., Sizeland, R.L., Edmonds, M.M., Basiljones, N., Kirby, A., Hawley, S., Mudie, R.G., Haverkamp, "*Changes to collagen structure during leather processing*", J. Agric. Food Chem., vol. 63(9), pp. 2499-2505, 2015.

[9] D., Hao, X., Wang, X., Liu, X., Zhu, S., Sun, J., Li, O., Yue, "A novel eco-friendly imidazole ionic liquids based amphoteric polymers for high performance fatliquoring in chromium-free tanned leather production", Journal of Hazardous Materials, vol. 399, 2020.

[10] D., Sun, Q., He, W., Zhang, L., Xin, B. Shi, "Evaluation of environmental impact of typical leather chemicals. Part I: Biodegradability of fatliquors in activated sludge treatment", J. Soc. Leather Technol. Chem., vol. 92, pp. 14-18, 2008.

[11] R., Saranya, A., Tamil Selvi, J., Jayapriya, R., Aravindhan, "Synthesis of fat liquor through fish waste valorization, characterization and applications in tannery industry", Waste and Biomass Valorization, vol. 11, pp. 6637-6647, 2020.

[12] TSE, TS EN ISO 105-B02, Tests for colour fastness - Part B02: Colour fastness to artificial light: Xenon arc fading lamp test, 2014.

[13] CIE (Comission Interantionalle DeL'Eclairage), Official recommendations on uniform colour spaces colour difference equations metric colour terms, 1976.

[14] A.D., Gun and B., Tibor, "Color, colorfastness, and abrasion properties of 50/50 bamboo/cotton blended knitted fabrics in three different stitch lengths", Textile Research Journal, vol. 81(18), pp. 1903-1915, 2011.

[15] A.M., Maruf and U., Chowdhary, "Color measurement and colorfastness of different weaves and dimensional forms", J Textile Sci & Fashion Tech., vol. 8(1), 2021.

[16] TSE, TS EN ISO 11640, Leather- Tests for colour fastness- Colour fastness to cycles of to - and - fro rubbing, 2013.

[17] 423-2 EN 20105-A02, Textiles-Tests for colour fastness - Part A02: Grey scale for assessing change in colour, 1996.

[18] TS EN ISO 105-A03, Textiles - Tests for colour fastness - Part A03: Grey scale for assessing staining, 2019.

[19] G., Verhoeven, "Imaging the invisible using modified digital still cameras for straightforward and low-cost archaeological near-infrared photography", Journal of Archaeological Science, vol. 35, pp. 3087–3100, 2008.

[20] B.W., Baker, A.D., Reinholz and E.O., Espinoza, "*Digital near-infrared photography as a tool in forensic snake skin identification*", Herpetological Journal, vol. 22, pp. 79–82, 2012.

[21] J., Fennen, F., Döppert and M., Hess, "Solar reflective leather", in IULTCS II. Eurocongress, Istanbul, 24-27 May, 2006.

[22] W. Mokrzycki, and M. Tatol, "*Color difference Delta E - A survey*", Mach. Graph. Vis., vol. 20(4), pp. 383-412, 2011.



INNOVATIVE TECHNOLOGIES BASED ON MEMBRANES FOR CONCENTRATING EXTRACTS FROM OLIVE CAKES

SIMION Demetra¹, GAIDAU Carmen¹, PAUN Gabriela², STANCA Maria¹, STOICA Tonea³

¹The Research Development National Institute for Textiles and Leather, Division Leather and Footwear Research Institute, 93 Street Ion Minulescu, 031215, Bucharest, Romania, E-mail: <u>demetra.simion@gmail.com</u>

²National Institute for Research and Development of Biological Sciences, Center of Bioanalysis, 296 Splaiul Independentei, Bucharest 6, 060031, Romania, E-mail: <u>gpaunroman@gmail.com</u>

³SC Taro Comimpex SRL, Int. Giurgiului, 28A, Jilava, Ilfov, 077120, Romania, <u>tarocomimpex@yahoo.com</u>

Corresponding author: Simion Demetra, E-mail: demetra.simion@gmail.com

Abstract: The aim of the paper is to concentrate extracts from olive cakes using innovative technologies based on membranes. Among the modern methods of purification and concentration of biologically active compounds (polyphenolic) in extracts are membrane technologies (microfiltration and ultrafiltration). These technologies, compared to all the other classics, have the advantage of separating, purifying or concentrating a certain compound in a single phase, cold, without the intervention of chemical reagents, with very low energy consumption. Concentrated extracts from olive cakes, obtained by membranes technologies, can be used as tanning and retanning agents, alternative to petroleum origin materials in leather industry.

Olive cake is rich in polyphenolic compounds, fats, tannins, non-tannins, possible to be extracted, concentrated, chemical processed in view of developing a new tanning product. The use of new renewable materials from oil industry as biobased tanning material for leather industry represents an important step in lowering carbon footprint of both sectors and complies with circular economy principles. The antioxidant and antimicrobial properties of olive oil cake can be exploited in view of increasing the efficiency of the new product.

The paper presents the concentration by membrane technologies and characterisation of five kinds of olive cake water and water-organic solvent extracts as future tanning-retanning materials.

Key words: membrane technologies, olive cake extracts, polyphenols, concentrate extracts, retanning materials in leather industry

1. INTRODUCTION

The European Union is the main producer, consumer and exporter of olive oil. The EU produces about 67% of the total quantity worldwide (M.D. Niculescu, C. Gaidau; 2014). There are eight different categories of olive oil: extra virgin olive oil; virgin olive oil; lampante olive oil; refined olive oil; olive oil composed of refined and virgin oil; olive cake oil; crude olive oil; refined olive cake oil. Not all of these categories can be sold to consumers. At retail, they can only buy extra virgin olive oil, virgin olive oil, olive oil composed of refined and virgin olive oil and olive cake oil. The different categories of olive cakes are classified according to quality parameters, in terms of: physicochemical characteristics, such as acidity level, peroxide index, fatty acid content and sterol content; organoleptic (sensory) characteristics, lack of organoleptic defects (C. Gaidau, M. Niculescu, E. Stepan, D.-G.



Epure, M. Gidea; 2014). The purpose of the exploitation of olive cakes is to make extracts from them and use as a source of bioactive compounds with added value. There are several extraction methods: ultrasonic cold maceration (fig.1), microwave-assisted extraction, microwave-assisted gravitational hydrodiffusion. The best known is ultrasonic cold maceration. It is possible to extract and concentrate some biological compounds from extracts by unconventional membrane technologies (Antunes, A.P.M., Attenburrow, G., Covington, A.D., Ding, J, 2008).



Fig. 1. Ultrasonic assisted maceration extraction method [3]

The aim of this paper is to concentrate by membrane technologies and characterise five kinds of olive cake water and water-organic solvent extracts as future tanning-retanning materials.

2. EXPERIMENTAL

2.1. Materials and Methods

Four types of olive waste originated from 2-phase extraction process, by-products of the olive cake: Arbequina, Palomar, Agro Igualada, Polpa d'oliva, in dry condition were received from different Spanish olive oil companies. The characterization of olive solid waste was performed according to standardized methods for tanning materials: dry substance (SR EN ISO 4684:2006), ash (SR EN ISO 4047:2002), total nitrogen and protein content using 6.25 conversion factor (SR EN 5397: 1996), extractible substances (SR EN ISO 4048: 2018), total residuum, total soluble substances, non tannins, tannins (shaking method), insoluble substances, tanning power, binding power (SR 1883: 2008) and pH (STAS 86193/3: 1990). The total phenols were analyzed following Folin-Ciocalteu method. To obtain the phenols content, 15 g of olive waste sample were magnetically stirred for 24 hours in 60 mL solution of 80% methanol. After stirring, the samples were ultrasounded for one hour and then filtered on Whatman paper. The UV-VIS spectra (JASCO V550) were recorded at $\lambda = 740$ nm on a calibration curve made with gallic acid. ATR-FTIR spectroscopy was performed on solid extracts in order to compare the chemical profile of different olive waste products and to understand the differences in their tanning properties by using a Jasco FT-IR 400 equipment from JASCO, Metertech. Four aqueous extracts were made from olive cakes: Arbequina, Palomar, Agro Igualada, Polpa d'oliva, by ultrasound for 1 hour, at pH = 2 (fig.2). Polpa d'oliva was selected for this study.



Fig. 2. Four aqueous extracts made from olive cakes: 1- Arbequina, 2-Palomar, 3- Agro Igualada, 4- Polpa d'oliva, by ultrasound for 1 hour, at pH = 2



The extraction methods were based on the variation of different conditions in aqueous and alcohol medium, at 55°C or by ultrasound (Elmo ultrasound bath, 280 W) and are presented in Table 1 for ground Polpa d'oliva.

Table 1: Extraction methods for olive extracts			
Sample	Extraction conditions		
1	water at pH= 2, 1: 40 (w/w), 1 h ultrasound.		
2	water at pH= 2, 1: 40 (w/w), 4 h at 55° C.		
3	water, 1: 40 (w/w), 4 h at 55°C.		
4	water: ethanol = 1:1 (v/v), pH=2, 1:40 (w/w), 1h ultrasound.		
5	water: ethanol = 1:1 (v/v), pH= 2, 1:40 (w/w), 4h at $55^{\circ}C$		

Extracts obtained from olive cakes - 5 extracts (1-5) were processed by membrane methods: microfiltration (MF) followed by ultrafiltration (UF) through a laboratory plant KMS Laboratory Cell CF-1 purchased from Koch Membrane (Germany), fig.3. The microfiltration was performed by microfiltration membranes made of cellulose acetate, with a pore size of 0.45 μ m, at a pressure of 3-4 bar resulting in the retention of all coarse particles in suspension. Ultrafiltration was performed through regenerated cellulose membranes with a cut-off of 3,000 Da; the concentration was achieved in the ratio 1: 3, at a pressure of 8 bar.



Fig. 3. Laboratory installation KMS Laboratory Cell CF-1

3.RESULTS AND DISCUSSIONS

3.1. Olive cake powders characterization

The 4 powders of olive cakes: Arbequina, Palomar, Agro Igualada, Polpa d'oliva were studied. The tanning power has the values which are not in agreement with total phenol content. The tanning power was ranked as follows: Arbequina > Palomar > Agro Igualada > Polpa d'oliva.

The physical-chemical characteristics of olive powders of olive cakes are presented in Table 2.

Characteristics				
	Arbequina	Palomar	Agro	Polpa
			Igualada	d'oliva
Dry substance, %,	89.31	86.08	90.24	92.24
Ash, %	2.65	1.74	2.40	6.18
Total nitrogen, %	0.76	0.70	1.17	2.23
Protein, %	4.75	4.38	7.31	13.93
Extractible substances %	14.48	10.90	11.71	7.92
Total residuum, %,	29.49	34.73	25.00	20.72
Total soluble substances, %	18.54	9.31	14.22	13.98
Non tannin, %	15.14	6.88	12.06	11.94
Tannin, %	3.40	2.43	2.16	1.77
Insoluble substance, %	10.95	25.42	10.78	8.51
pH (1:10), pH units	5.10	5.05	5.52	6.15
Tanning power, %	3.10	1.77	1.97	1.52
Binding power, %	nd	nd	nd	nd

 Table 2. Physical-chemical characteristics of olive powders



The main components of olive cake powders in ATR-FTIR spectras (fig.4) can be recognized from the main functional groups vibrations: polyphenols, alcohols and carboxylic compounds (3258-3311.18 cm⁻¹), cellulose (2922.59-2925.48 cm⁻¹ and 2853.15-2854.43 cm⁻¹), hemicelluloses in Agro Igualada and Arbequina (1740-1743 cm⁻¹), oleuropein (1022.05-1078 cm⁻¹), proteins (1631.48-1637.27 cm⁻¹ and 1598 cm⁻¹), phenols (1371.14-1375.96 cm⁻¹) and β-glycoside links (1151-1156.12 cm⁻¹). The transmittance in ATR-FTIR spectras at polyphenol wave number was recorded for Arbequina olive powders as the most intense, compared to the others, which is in agreement with tanning affinity.



Fig. 4. ATR-FTIR of olive powders: 1- Palomar, 2- Polpa d'oliva, 3- Agro Igualada, 4- Arbequina

Polyphenols are indicated in the FTIR-ATR spectra as being specific to the wave number: 3400 cm^{-1} . It is observed quantitatively in the initial samples, depending on the values of the transmittance in the spectra, the following order, the largest amount of polyphenols in: Remolta Palomar (1)> Agro Igualada (3)> Polpa d'oliva (2)> Arbequina (4). In this study Polpa d'oliva was selected. In fig.5 is the ATR-FTIR spectra for OBE tanning agent.



Fig. 5. ATR-FTIR spectra for OBE tanning agent



3.2.Olive Extracts Characterization

For the four extracts in figure 2, the total polyphenol content was determined to select which of the 4 types of olive cake is the best for the study. The principle of the method used is based on the property of phenols to form with the complex phosphowolframic reagent (Folin-Ciocâlteu reagent) blue complexes, which can be dosed spectrophotometrically at λ =760 nm. Basically, 5 ml of the extract sample is treated with 5 ml of Folin-Ciocalteu reagent, stirred and then filtered. 1 ml of filtrate is made up to a 10 ml graduated flask with. 20% sodium carbonate solution, and after about 2 minutes read the absorbance of the sample at 660 nm compared to the 20% sodium carbonate solution used as a control. The polyphenol content was calculated using a standard curve made with chlorogenic acid (CA), in the range of concentrations 0-800 mg/L, the concentration of polyphenols in the extracts being expressed as mg CA / L, according to the table 3.

Table 3. Experimental results for the polyphenol content of the samples with: 1- Arbequina, 2-Palomar, 3- Agro Igualada, 4- Polpa d'oliva, by ultrasound for 1 hour, at pH = 2

Sample	Dilution	Absorbance at λ =760 nm	Concentration., µg ACE/mL
1	-	0,6170	480,2
2	-	0.6813	511.2
3	-	0.6441	481.7
4	10	0.2344	1568.7



The ATR-FTIR spectras for these four extracts from figure 2 are presented in figure 6.

Fig. 6. ATR-FTIR of olive extracts: E1- Palomar, E2- Polpa d'oliva, E3- Agro Igualada, E4- Arbequina

There are significant differences in the spectra for the initial powders compared to the extracts obtained. The total phenols of tested extracted methods performed on Polpa d'oliva cakes presented in table 1 conclude that the extraction in water and assisted by ultrasound was the most efficient (15.32 mg GAE/g from olive cakes). Similar phenol concentrations, between 7.72-9.42 mg GAE/g from olive cakes, were extracted in water or in water heated at 55°C for 4 hours, or in water by ultrasound for 1 hour. Further research will be carried out for high molecular weight phenols identification (tannins, anthocyans, catechol-metaninic polymers etc. in correlation to extraction methods and tanning properties. *All these 5 extracts from table 1 were concentrated by membrane technologies*.



3.3.Membrane technologies

Microfiltration (MF) is the process that most closely resembles ordinary filtration and is used to separate particles with dimensions between 0.1-10 µm, such as suspended solid particles, viruses, bacteria, veast cells, etc. Ultrafiltration (UF) is a process of separating colloidal substances and compounds with a molecular weight between 1000 and 100,000,000 Da, using filtration through membranes at low pressures. The separation through ultrafiltration membranes is due to the sieving mechanism, after which the solvent and the smaller molecules of the solute are transported through the pores of the membrane, which retains only the molecules of some solutions with dimensions larger than the pore size. Five extracts obtained from olive cakes (from table 1) were processed by membrane technologies: microfiltration (MF) followed by ultrafiltration (UF) through a laboratory plant KMS Laboratory Cell CF-1 purchased from Koch Membrane (Germany), figure 3. The microfiltration was performed by microfiltration membranes made of cellulose acetate, with a pore size of 0.45 μ m, at a pressure of 3-4 bar (figure 7). Ultrafiltration was performed through regenerated cellulose membranes with a cut-off of 3,000 Da at a pressure of 8 bar (figure 7).





Fig. 7. Membranes: a) microfiltration membrane (MF) after passing the extract; b) ultrafiltration membrane (UF) after extract concentration

a)



Fig. 8. Technological flow of concentration through membrane processes

The results of the ultrafiltration process were measured by permeate flow and polyphenol retention. The permeate flow is calculated according to equation (1):

$$J = \frac{V}{A \cdot t}$$
(1)

where: V = permeate volume, A = effective membrane surface, t = time required to collect volume V of permeate. Retention, % R, is calculated according to equation (2)



$$R = \left[1 - \frac{c_{\rho}}{c_{f}}\right] \times 100$$

(2)

where C_p and C_f =concentration of the monitored component in the permeate and in the feed solution, respectively.

3.4.Concentrated Extracts Characterization

Five extracts obtained from olive cakes (from table 1) were concentrated by technological flow presented in figure 8. The determination of the polyphenols content of the 5 concentrated extracts by membrane technologies (from table 1) was performed using the Folin-Ciocalteu method. The principle of the method used is based on the property of phenols to form complexes with blue phosphowolframic reagent (Folin-Ciocalteu reagent), which can be dosed spectrophotometrically at a wavelength of 760 nm. The total polyphenol content was expressed in μ g equivalents of chlorogenic acid (ACE) / mL extract. At microfiltration, flow values of 297,5÷556,3 L/m³h, were obtained for the samples taken, the lowest value being recorded for sample 1. At the ultrafiltration of the 2 sample, the value of the permeate flow was 8.23 L/m³h, and the retention degree of the total polyphenols was 51.6% (C_f = 847.02 μ g ACE / mL; C_p = 410.01 μ g ACE / mL). At the ultrafiltration of the 5 samples concentrated from table 1, the degree of retention of the total polyphenols was presented in table 4:

Sample concentrated	e of retention of the total polyphenols from 5 extracted concentratea The degree of retention of the total polyphenols from 5 extracted concentrated by membrane technologies, %
1	59,9
2	51,6
3	42
4	46
5	49

Table 4. The degree of retention of the total polyphenols from 5 extracted concentrated

The best results were obtained for sample 1 with extract conditions: water at pH= 2, 1: 40 (w/w), 1 h ultrasound. Research is in progress regarding to create new retaining materials.

4. CONCLUSIONS

1. In this research were concentrated extracts from olive cakes using innovative technologies based on membranes. Among the modern methods of purification and concentration of biologically active compounds (polyphenolic) in extracts are membrane technologies (microfiltration and ultrafiltration). These technologies, compared to all other classical ones, have the advantage of separating, purifying or concentrating a certain compound in a single phase, cold, without the intervention of chemical reagents, with very low energy consumption.

2.Concentrated extracts from olive cakes, obtained by membranes technologies, can be used as tanning and retanning agents, alternative to petroleum origin materials in leather industry.

3.Olive cake is rich in polyphenolic compounds, fats, tannins, non-tannins, possible to be extracted, concentrated, chemical processed in view of developing a new tanning product. The use of new renewable materials from oil industry as biobased tanning material for leather industry represents an important step in lowering carbon footprint of both sectors and complies with circular economy



principles. The antioxidant and antimicrobial properties of olive oil cake can be exploited in view of increasing the efficiency of the new product.

4. This paper presents the concentration by membrane technologies and characterisation of five kinds of olive cake water and water-organic solvent extracts as future tanning-retanning materials.

ACKNOWLEDGEMENTS

The works were supported by a grant of the Romanian National Authority for Science Research and Innovation, CCCDI-UEFISCDI, project numbers 144 and 145/2020, ERANET-MANUNET-OLIPO-1 and 2, within PNCDI III.

REFERENCES

[I] M.D. Niculescu, C. Gaidau, "*New collagen extracts conditioned for applications in crop protection against pests*", REV. CHIM. (Bucharest), 65, 12, p.1457-1461, 2014.

[II] C. Gaidau, M. Niculescu, E. Stepan, D.-G. Epure, M. Gidea, "New Mixes Based on Collagen Extracts with Bioactive Properties, for Treatment of Seeds in Sustainable Agriculture", Current Pharmaceutical Biotechnology Journal, 14,9, p. 792-801, 2014.

[III] Antunes, A.P.M., Attenburrow, G., Covington, A.D. and Ding, J. (2008), "Utilisation of oleuropein as a crosslinking agent in collagenic film", J Leather Sci, 2, 1-12.

[IV]. C.Gaidau, M. Stanca, D. Simion, G. Paun, "*Olipo-wet olive pomace, a new renewable source for leather retanning*", Conference: The 8th International Conference on Advanced Materials and Systems, November 2020, DOI:10.24264/icams-2020.II.10

[V] Esposto, S., Taticchi, A., Di Maio, I., Urbani, S., Veneziani, G., Selvaggini, R., Sordini, B., Servili, M.: *"Effect of an olive phenolic extract on the quality of vegetable oils during frying"*, Food Chem. 176, 184–192 (2015). https://doi.org/10.1016/J.FOODCHEM.2014.12.036

[VI] Morvová, M., Onderka, M., Morvová, M., Morva, I., Chudoba, V.: "*Pyrolysis of olive mill waste with on-line and ex-post analysis*", Waste Biomass Valoriz. 10, 511–520 (2019). https://doi.org/10.1007/s1264 9-017-0126-4



CONTENTS

No	Paper title	Authors	Institution	Page
	STUDY OF RESISTANCE	AMIRSHIRZAD		
	TO SHARP OBJECT	Forough ¹ ,	^{1, 2,3} Amirkabir University of	
1	PENETRATION AND	MOUSAZADEGAN	Technology, Textile	5
L	SPREAD OF THE	Fatemeh ² ,	Engineering Faculty, 424	5
	RESULTING RUPTURE IN	EZAZSHAHABI	Hafez Ave, 15875-4413,	
	WOVEN FABRICS	Nazanin ³	Tehran, Iran	
2	EXPERIMENTAL RESEARCH ON OBTAINING A CHROMATIC PALETTE ON HEMP FABRIC BY COMBINING WELD AND MADDER DYES	BUDEANU Ramona	"Gheorghe Asachi" Technical University of Iasi, Faculty of Industrial Design and Business Management, 67 D. Mangeron Street, Iasi 700050, Romania,	11
3	THE ANALYSIS OF THE SYSTEM "HUMAN– CLOTHING– ENVIRONMENT" WITH APPLICATION IN THE DESIGN OF FUNCTIONAL ADAPTIVE PRODUCTS	FLOREA-BURDUJA Elena ^{1,3} , RARU Aliona ^{2,3} , FARÎMA Daniela ³ , IROVAN Marcela ¹	 ¹ Technical University of Moldova, Faculty of Textile and Polygraphy, 4 Sergiu Radautan Street, Chisinau MD-2019, Republic of Moldova ² Technical University of Moldova, Direction of Academic Management and Quality Assurance, 168 Stefan cel Mare Street, Chisinau MD-2004, Republic of Moldova ³ Gheorghe Asachi Technical University of Iasi, Faculty of Industrial Design and Business, 29 Mangeron Street, Iasi 700050, Romania 	17
4	BIOACTIVE HYDROGEL TYPE CARRIER SYSTEMS AIMED FOR TEXTILE WOUND DRESSING	CHIRILĂ Laura ¹ , ȚIGĂU Andreea ¹ , CONSTANTINESCU R. Rodica ² , OLARU Sabina ¹ , POPESCU Alina ¹ , STAN Miruna ^{3,4}	 ¹National Research & Development Institute for Textiles and Leather, 16 Lucretiu Patrascanu Street, 030508, Sector 3, Bucharest, Romania ² National Research & Development Institute for Textiles and Leather, Leather and Footwear Research Institute (ICPI) Division, 93 Ion Minulescu Street, 031215, Bucharest, Romania ³ Department of Biochemistry 	21



-			· · · · · · · · · · · · · · · · · · ·	
			and Molecular Biology,	
			Faculty of Biology,	
			University of Bucharest, 91-	
			95 Spl. Independentei,	
			050095 Bucharest, Romania	
			⁴ Research Institute of the	
			University of Bucharest-	
			ICUB, University of	
			Bucharest, 91–95 Splaiul	
			Independentei, 050095	
			Bucharest, Romania	
			^{1, 2} , "Gheorghe Asachi"	
			Technical University of Iasi -	
			Romania, Faculty of	
			Industrial Design and	
			Business Management, Blvd.	
	DEVELOPMENT OF	DANILA Victoria ¹ ,	Mangeron, No. 59A, 700050,	
5	FUNCTIONAL PRODUCTS	CURTEZA	Iasi, Romania	29
5	FOR PREMATURE BABIES	Antonela ² ,		47
	USING THE CAD SYSTEM	BALAN Stela ³	³ Technical University of	
			Moldova, Faculty of Textile	
			and Poligraphy, avenue	
			Stefan cel Mare si Sfint 168,	
			2004-MD, Chisinau, Republic	
			of Moldova	
			^{1, 2} University of Oradea,	
			Faculty of Energy	
			Engineering and Industrial	
		DOBLE Liliana ¹ ,	Management, Department	
	SMART VALORIFICATION	BÖHM Gabriella ² ,	Textiles, Leather and Industrial Management,	
6	OF RECICLABLE TEXTILE	PORUMB Camelia	410058, Oradea, România,	33
	WASTE	Luminița ³	+10050, Oradea, Romania,	
		Lummița	³ University of Oradea,	
			Faculty of Art, Departament	
			of Vizual Art, 410058,	
			Oradea	
			^{1, 2,3,4} The Research-	
			Development National	
		ENE Alexandra ¹ ,	Institute for Textiles and	
		MIHAI Carmen ² ,	Leather, 16 Lucretiu	
	NEW PARADIGMS IN	SCARLAT Razvan ³ ,	Patrascanu Street, 030508,	
7	DESIGNING THE MEDICAL	GROSU Catalin ⁴ ,	Bucharest, Romania	37
	TEXTILES	HERTZOG Radu -	5600	
		Gabriel ⁵ ,	^{5, 6} "Cantacuzino" National	
		POPESCU Diana ⁶	Military-Medical Institute for	
		I UI LOUU Dialia	Research and Development,	
			103 Splaiul Independentei, 050096, Bucharest, Romania	
		I ÁPEZ	¹ Departamento de Ingeniería	
		LÓPEZ-	Textil y Papelera, Universitat	
	RECOVERY OF REACTIVE	RODRÍGUEZ	Politècnica de València	
	COLORANT WITH	Daniel ¹ ,	Plaza Ferrándiz y Carbonell	
8	HYDROTHALCITE AND	MICÓ-VICENT	s/n, Alcoi, Spain	43
	REUSE FOR PRINTING	Barbara ² ,	s, ii, i iiosi, spain	
		MONTAVA Ignacio ¹ ,	² Departamento de Ingeniería,	
		BOU-BELDA Eva ^{1*}	Universitat Politècnica de	



9	SORPTION OF TEXTILE DYES ON SORBENT MADE FROM COTTON TEXTILE WASTE - EQUILIBRIUM ADSORPTION	MICIC Aleksandra ¹ , RISTIC Ivanka ² , DJORDJEVIC Suzana ³ , RISTIC Nebojsa ⁴ , DJORDJEVIC Dragan ⁵	València Plaza Ferrándiz y Carbonell s/n, Alcoi, Spain ^{1,5} University of Nis, Faculty of Technology, 16000, Leskovac, Serbia ^{2,3,4} Academy of Vocational Studies Southern Serbia, Department of Technological Art Studies Leskovac, 16000, Leskovac, Serbia	47
10	LABORATORY STUDIES ON NATURAL FABRICS TEXTILE PRINTING WITH SPIRULINA PLATENSIS SOURCED PHYCOCYANIN	MOLDOVAN Simona ¹ , FRANCO Esther ¹ , PASCUAL Javier ¹ , FERRANDIZ Marcela ¹ , BONET-ARACIL Marilés ² , GISBERT-PAYÁ Jaime ² , BOU-BELDA Eva ²	¹ Aitex, Technological Research Institute on Textile. Plaza Emilio Sala nº 1, 03801. Alcoy, Spain ² Universitat Politècnica de València, Textile and Paper Department, Plaza Ferrándiz y Carbonell s/n, 03801, Alcoy, Spain	53
11	OPTIMIZATION OF PROCESS CONDITIONS OF SILK FABRIC DYEING WITH GALINSOGA PARVIFLORA LEAF EXTRACT FOR ANTIBACTERIAL APPLICATION.	MUSINGUZI Alex ^{1, 2} , MWASIAGI, J. Igadwa ¹ , NIBIKORA Ildephonse ² NZILA Charles ¹	 ¹ Moi University, Faculty of Engineering, Department of Manufacturing, Industrial and Textile Engineering, P.O. Box 3900-30100, Eldoret, Kenya. ² Busitema University, Faculty of Engineering, Department of Polymer, Textile and Industrialisation Engineering, P.O. Box 236, Tororo, Uganda. 	59
12	VIRTUAL PROTOTYPING OF PROTECTIVE CLOTHING FOR OVERSIZED SUBJECTS	OLARU Sabina ¹ , POPESCU Georgeta ¹ , TOMA Doina ¹ , BADEA Ionela ¹ , GROSU Cristina ^{1,2}	 ¹National Research and Development Institute for Textiles and Leather, 16 Lucretiu Patrascanu Street, 030508, Bucharest, Romania ² Gheorghe Asachi Technical University of Iasi, Faculty of Industrial Design and Business Management, 29 Mangeron Boulevard, 700050, Iasi, Romania 	65
13	BAMBOO FIBER ANTIBACTERIAL EFFECT (A REVIEW)	RARU Aliona ^{1,3} , FLOREA-BURDUJA Elena ^{2,3} , IROVAN Marcela ¹ , FARÎMA Daniela ³	 ¹ Technical University of Moldova, Direction of Academic Management and Quality Assurance, 168 Stefan cel Mare Street, Chisinau MD-2000, Republic of Moldova ² Technical University of Moldova, Faculty of Textile and Polygraphy, 4 Sergiu 	71



			MD-2000, Republic of Moldova	
			³ Gheorghe Asachi Technical University of Iasi, Faculty of Industrial Design and Business, 29 Mangeron Street, Iasi 700050, Romania	
14	CHARACTERIZATION OF SOME TEXTILE ARTIFACTS FROM PLOPIS WOODEN CHURCH – PART OF UNESCO WORLD HERITAGE SITES	SECAREANU Lucia- Oana ¹ , SANDULACHE Irina ¹ , MITRAN Elena- Cornelia ^{1,2} , LITE Mihaela- Cristina ^{1,2} , IORDACHE Ovidiu ¹ , PERDUM Elena ¹	 ¹ The National Research and Development Institute for textiles and Leather (INCDTP), Materials Research and Investigation Department, 16 Lucretiu Patrascanu, 030508 Bucharest, Romania ² University Politehnica of Bucharest, Faculty of Applied Chemistry and Materials Science, 1-7 Gheorghe Polizu, 011061 Bucharest, Romania 	77
15	COMPARATIVE STUDY BETWEEN POLYAMIDE/ELASTANE AND COTTON/POLYAMIDE/ELA STANE SOCKS	SZABO Monica ¹ , DOCHIA Mihaela ² , GAVRILAŞ Simona ³	 ¹ "Aurel Vlaicu" University of Arad, Faculty of Engineering, Department of Automation, Industrial, Textile and Transportation Engineering, Postal address, 310330, 2-4 Elena Dragoi Street, Arad, Romania ² "Aurel Vlaicu" University of Arad, Research Development Innovation in Technical and Natural Science Institute, Postal address, 310330, 2-4 Elena Dragoi Street, Arad, Romania ³ "Aurel Vlaicu" University of Arad, Esculty of Food 	83
16	COMPOSITE TEXTILE STRUCTURES FOR PARIETAL DEFECTS	VISILEANU Emilia, CHIRIAC Laura, SCARLAT Razvan, VI ADU Alina	Arad, Faculty of Food Engineering, Tourism and Environmental Protection, Department of Technical and Natural Sciences, Postal address, 310330, 2-4 Elena Dragoi Street, Arad, Romania Institutul National de Cercetare Dezvoltare pentru Textile si Pielarie, 16, Lucretiu Patrascanu street,	89
17	CHARACTERISATION OF METAL THREADS FROM ARCHAELOGICAL TEXTILES RELIGIOUS BY	VLADU Alina VORNICU Nicoleta ¹ , BIBIRE Cristina ¹ , ZALTARIOV Mirela Fernanda ²	sector 3, Bucharest, Romania ¹ Metropolitan Research Center T.A.B.O.R, 9 Closca Str.,700066, Iasi, Romania	95



		[2	
	METHODS OF		² Petru Poni" Institute of	
	ARCHAEOMETRY		Macromolecular Chemistry,	
			41A Grigore Ghica Voda	
18	VISUAL MAPPING STRATEGIES TO ORGANIZE, COMMUNICATE AND MAINTAIN DESIGN	BELLEMARE Jocelyn	Alley 700487 Iasi, Romania University of Quebec in Montreal (UQAM), School of Business and Management (ESG), Department of Analytics, Operations, and Information Technology (AOIT).	101
	KNOWLEDGE		Montreal (Quebec), Canada	
19	STRATEGIC VISION FOR RECOVERY OF MOLDOVA'S FASHION INDUSTRY AFTER COVID-	GHEORGHITA Maria ¹ , NISTOR Doina ²	 ¹ Technical University of Moldova, Faculty of Economic Engineering and Business, Department Economics and Management, 41, Dacia bd., nr. 10 block of study, MD-2060, Chisinau, Republic of Moldova ² Technical University of 	107
	19		Moldova, Faculty of Economic Engineering and Business, Department Economics and Management, 41, Dacia bd., nr. 10 block of study, MD-2060, Chisinau, Republic of Moldova ¹ Pamukkale University,	
20	STUDY ON THE MECHANICAL PROPERTIES OF LEATHERS REINFORCED WITH SUPPORTING MATERIALS	ÇOLAK Selime ¹ , KAYGUSUZ Meruyert ²	² Pamukkale University, Denizli Vocational School of Technical Sciences, Traditional Handicrafts Department, Denizli/Turkey ² Pamukkale University, Denizli Vocational School of Technical Sciences, Textile, Apparel, Footwear and Leather Department, Denizli/Turkey	113
21	BEHAVIOUR OF IR REFLECTIVE DYES AND PIGMENTS ON DIFFERENT FATLIQUORED UPHOLSTERY LEATHERS	ORK EFENDIOGLU Nilay ¹ , YAKUT Fatih ¹ MUTLU Mehmet Mete ¹	¹ Ege University, Engineering Faculty, Department of Leather Engineering, Bornova, Izmir, Turkiye	119
22	INNOVATIVE TECHNOLOGIES BASED ON MEMBRANES FOR CONCENTRATING EXTRACTS FROM OLIVE CAKES	SIMION Demetra ¹ , GAIDAU Carmen ¹ , PAUN Gabriela ² , STANCA Maria ¹ , STOICA Tonea ³	¹ The Research Development National Institute for Textiles and Leather, Division Leather and Footwear Research Institute, 93 Street Ion Minulescu, 031215, Bucharest, Romania ² National Institute for Research and Development of Biological Sciences, Center of	125



	Bioanalysis, 296 Splaiul Independentei, Bucharest 6, 060031, Romania	
	³ SC Taro Comimpex SRL, Int. Giurgiului, 28A, Jilava, Ilfov, 077120, Romania	