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it Sumar

Contents

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Preparation and physical characteristics of porous phase change PU/PEG membrane

GUI-ZHEN KE WEI-DONG YU

REZUMAT – ABSTRACT

Obținerea şi caracteristicile fizice ale membranelor poroase PU/PEG cu schimbare de fază

Membranele poroase cu schimbare de fază au fost obținute din poliuretan (PU) cu diferite procente de polietilenglicol (PEG), dimetilformaldehidă (DMF) şi carbonat de amoniu. Au fost studiate structura morfologică, comportamentul la schimbarea de fază şi principalele caracteristici fizice ale membranelor. Rezultatele au arătat că membrana PU/PEG posedă evidente caracteristici structurale poroase, o temperatură de tranziție adecvată şi o entalpie de tranziție ridicată. Odată cu creşterea conținutului de polietilenglicol (PEG) sau de solvent (DMF) din compoziție, membrana a înregistrat o scădere a rezistenței la rupere, a alungirii la rupere, a modulului inițial şi a rezistenței la abraziune. Rezistența la încovoiere a membranei depinde, în mare măsură, de temperatura de testare. Rezultatele testelor de permeabilitate la apă şi de absorbție a umidității au indicat faptul că membrana prezintă o funcție de autocondiționare a permeabilității la apă, iar creşterea conținutului de polietilenglicol duce la creşterea permeabilității la apă, prin micropori şi grupări hidrofile.

Cuvinte-cheie: materiale cu schimbare de fază, membrană poroasă, poliuretan, polietilenglicol, caracteristici fizice

Preparation and physical characteristics of porous phase change PU/PEG membrane

Porous phase change membranes were prepared with selected PEG binary systems and liquid PU. The morphology structure, phase change behaviors and essential physical characteristics of the membrane were studied. The results showed that the PU/PEG membrane had obvious porous structural feature, suitable transition temperature and high transition enthalpy. As the content of working material PEG or solvent DMF increased, breaking strength, breaking elongation, initial modulus and abrasion resistance of the membrane decreased. Bending rigidity of the membrane depended on testing temperature largely. The results of moisture permeability and absorption testing indicated that the membrane had auto-conditioning moisture permeability function and the increase of PEG content strengthened the moisture permeability through micropores and hydrophilic groups.

Key-words: phase change materials, porous membrane, polyurethane, polyethylene glycol, physical characteristics

Recently high performance functional materials with novel structure have been developed widely. Thermal storage phase change materials (PCMs), as particular smart materials, play an important role among them [1–3]. PCMs are generally solid at room temperature. When the temperature becomes warmer, PCMs liquefy and absorb and store heat, thus cooling the ambient. Conversely, when the temperature drops, the materials will solidify and give off heat, warming the ambient [4]. Nowadays polyethylene glycol (PEG) is one of the most widely used phase change material in preparation of various PCMs for its wide range of transition point, high phase transition enthalpy, good thermal stability and so on [5–7].

It is well known that the composite phase change materials with porous structure have been used in the buildings, which happen solid-liquid phase change in microcosmic and keep solid state in macroscopy during the transition [8–9]. Though outstanding advantages such as higher contents of working materials and stable shape, the porous PCMs are always rigid that prevents its application in other areas. At the same time temperature-adaptable fiber and fabrics currently are always prepared with pure phase change materials and carrier materials which can not supply satisfied effects for its low contents of working materials and low transition enthalpy [10–11].

So, how to prepare a flexible porous PCM used as temperature-adaptable fabrics with higher transition enthalpy becomes very interesting problems. In this paper, the preparation, morphology characters, essential wearing characteristics of the membrane such as mechanical properties, bending rigidity and moisture permeability were studied.

EXPERIMENTAL PART

Materials and chemicals

Liquid polyurethane (PU, 30% solid content) was of chemical grade and purchased from Shandong Yantai HuaDa Chemicals Industry.

Polyethylene glycol (PEG) 1000 and 2000, *N,N*-dimethylformamide (DMF) and ammonium carbonate ($(NH_4)_2CO_3$) were of chemical grade and supplied by Shanghai Sinopharm Chemical Reagent Co. Inc. $(NH_4)_2CO_3$ was grinded into powders by high speed disintegrator before use.

Membrane preparation

PEG 1000 and 2000 were blended with mass ratio 1:1 and melted into liquid for binary systems. The

homogenous mixture solution was obtained by dissolving PU and the melted PEG in DMF with vigorous stirring. When the mixture solution cooled down to ambient temperature, $(NH4)_2CO_3$ superfine powder was added into above mixture. After stirring for another 2 hours, the bubbles in the blended solution were removed using vacuum pump. Secondly, after degassing, the resulting solution was cast onto a plane glass model using a glass bar. The cast film was placed at ambient temperature for 2 minutes, and then immersed into water at room temperature for 3 minutes. And then the model was taken out of water and dried in the air for about 5 hours. Finally the membrane was removed from the model and placed in climatic chamber with condition 50ºC and 70 ~ 80% R.H. for at least three hour. After steam treatment, the films were stored in ventilation environment for at least one week before measurements. During membrane preparation, when PEG content changed, the concentration of PU was kept at 15% in PU and DMF blended solution; and when DMF content changed, the weight ratio of PEG to PU was kept at 3:1.

Membrane characters measurements

The surface morphology of the films was investigated using a scanning electron microscope (Hitachi, model S-450) at 20 kV with gold powder coating on the samples. The cross-sectional fracture surfaces of the specimen were obtained by cooling in liquid nitrogen followed by breaking.

Differential scanning calorimetry (DSC) was carried out using a 204 F1. Samples (5–10 mg) were sealed in aluminum pans and measurements were performed in nitrogen atmosphere at a heating rate of 10°C/minutes over the temperature range of 0 to 100°C. In the first scanning, the sample was kept at 0°C for 10 minutes and heated to 100°C, then kept this temperature for 10 minutes. In the second scanning, the sample was cool down to 0°C and the second scan was recorded.

Moisture regain, %, was determined on dried samples kept at 20°C and 65% RH for enough time and moisture regain of the film was defined using equation (1):

$$\textit{Moisture regain} = (W_2 - W_1)/W_1 \times 100 \qquad (1)$$

where:

W_1 and W_2 represent the dry weight and humid weight of tested samples respectively.

Mechanical properties of the membrane were measured using Instron 5566 with 30 mm gauge at the strain rate of 100 mm/minutes at room temperature. Dimensions of the specimen used were 100 mm × × 28 mm by size. Measurements of five tests were averaged for each sample.

Bending length of the membrane was measured according to ISO9073-7:1995 (inclined plane method).

Test procedures of abrasion resistance were carried out with Rotary Platform Abraser (Abrader). 150 # grinding wheel was used as abrasive. The pressure loaded on specimen was 500 g. Operating speed was maintained at 3000 rpm. The rotor revolution was recorded when the surface of the specimen appeared a hole. Measurements of five tests were averaged for each specimen.

Moisture permeability measurement: The film was put on a cup (inner radius 60 mm, height 22 mm) containing 10 mL water at constant temperature & humidity test chamber (20ºC, 65% R.H.). The cup was placed right side up and the tested side of membrane contacted with the air in the cup. The moisture permeability was calculated using equation (2):

$$\textit{Moisture permeability}\ (g/m^2 \cdot h) = \frac{B}{t \cdot A} \qquad (2)$$

where:

B is the weight loss of water in cup;

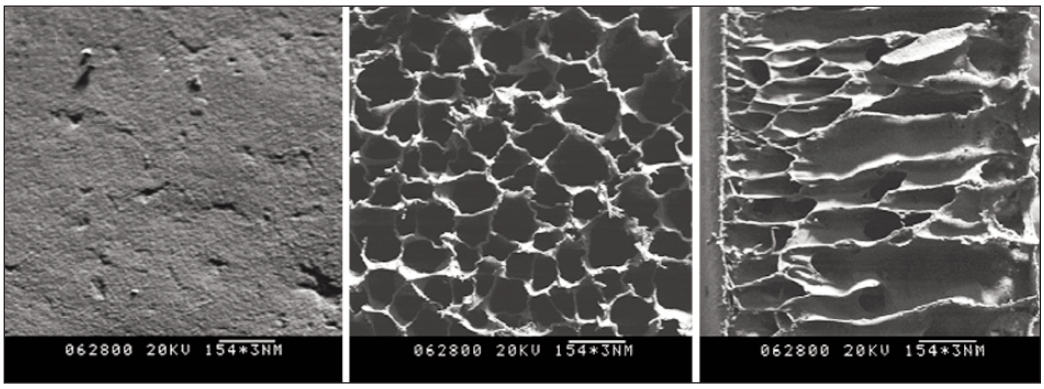
A – the vaporing area of water;

t – the time interval of specimen placed on the cup.

RESULTS AND DISCUSSIONS

Morphology of the membrane

Figure 1 shows the morphology and porous structure of the membrane. From figure 1 *a*, it can be seen that membrane surface was smooth and compacted. Figure 1 *b* showed the sub-layer structure of the membrane. It was observed that in the sublayer round or hexagon pores as beehive stand side by side for a certain distribution. Observed from the cross section



a ***b*** ***C***

Fig. 1. SEM photos of the membrane:
a – surface × 200; ***b*** – sub-layer × 200; ***c*** – cross-section × 200

(fig. 1 *c*), the pore grew along the thickness direction of the membrane.

The porous membrane was composed of two sections: compacted surface and porous sublayer. The smooth surface could provide the membrane with most of strength and good protection to prevent leaking of working materials. Porous sublayer was the supporting structure of the whole membrane and provided the room which working materials filled with. In the sublayer PEG was divided into small unit by a lot of pores and lost the flowability even at high temperature. In addition, flexible hand of the membrane also rooted in the porous sublayer structure.

Phase change behavior

In order to study the phase change behavior of the PEG in the porous membrane, the membrane of different PEG content was measured by DSC. DSC curves of pure PEG are shown in figure 2, porous membrane's in figure 3 and figure 4 and the data of their transition temperature and enthalpy are summarized in table 1.

The results show that there is a melting peak at about 51°C in the heating scanning DSC curve of PEG 1000/2000 and the latent heat of fusion is 181.7 J/g, and about 52°C in the heating scanning DSC curve of porous membranes and the enthalpy is 108.1 J/g to 129.1 J/g. In addition, the cooling DSC curves of PEG and porous membrane show similar characters. The enthalpies of the positive process and the reverse process are close, and the temperature decides the direction, which indicates that the porous membrane is a transition reversible latent storage material. Though the porous membrane has the similar thermal properties as the pure PEG, T_{on1}, T_{end1} and T_{p1} of PEG in the porous membrane increase in

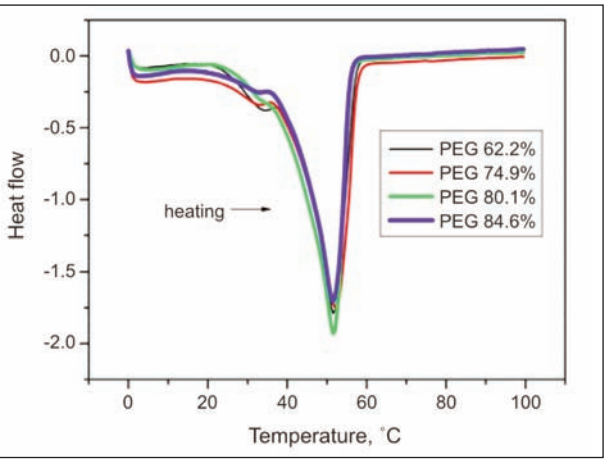


Fig. 3. DSC heating curves of the membrane with different PEG content

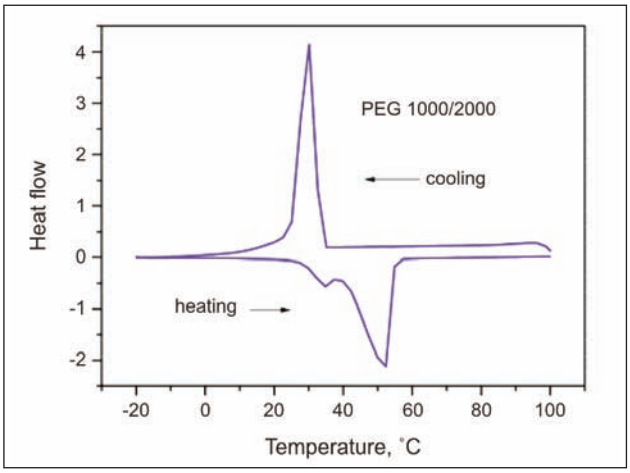


Fig. 2. DSC curves of PEG 1000/2000

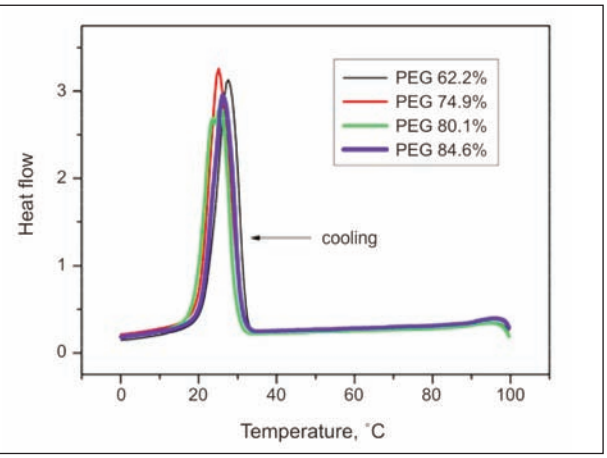


Fig. 4. DSC cooling curves of the membrane with different PEG content

Table 1

TRANSITION TEMPERATURE AND TRANSITION ENTHALPY OF THE MEMBRANE WITH VARIOUS PEG CONTENT

PEG, %	Heating				Cooling			
	T_{on1}, °C	T_{p1}, °C	T_{end1}, °C	ΔH_1, J/g	T_{on2}, °C	T_{p2}, °C	T_{end2}, °C	ΔH_2, J/g
62.2	43.5	51.5	55.8	108.1	31.0	26.1	20.9	85.18
74.9	43.0	51.7	55.5	127.7	29.6	25.6	19.8	81.08
80.1	44.1	52.2	57.7	119.7	31.4	25	19.8	92.4
84.6	44.5	51.6	57.3	129.1	32.1	27.6	22.1	100.3
100.0	42.8	50.8	55.2	181.7	33.7	31.4	24.2	155.3

* T_{on} is initial temperature; T_{end} – final temperature; T_p – peak temperature; ΔH – enthalpy; *1* – heating; *2* – cooling

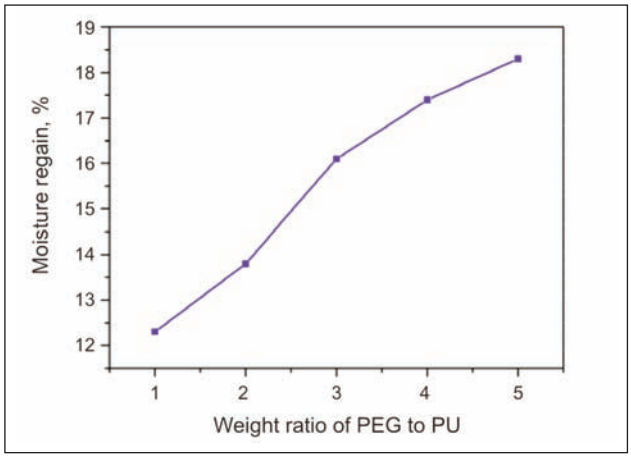


Fig. 5. Moisture regain of porous phase change membrane with different PEG content

the heating direction; but T_{on2}, T_{end2} and T_{p2} diminish in the cooling direction. These mean that PU blocked PEG to carry out phase change and reduces its sensitivity of temperature; when PEG crystallizes, PU weakens the integrity of the PEG crystallization, which make its transition temperature dropped. Moreover it is observed that there is a big difference in their transition states. When heating, PEG1000/2000's state changes from solid to liquid, while porous membranes' phase transition is a process from solid to quasio-solid and the membrane can be regarded as solid–solid PCM.

Moisture regain of the membrane

Moisture adsorption of the membrane resulted from hydrophilic groups of PEG and surface absorption of porous membrane. The relationship between moisture regain and PEG content is shown in figure 5. It was observed that the moisture regain almost increased linearly with the increase of PEG content. It was due to high content of PEG in the membrane. PEG occupied most of surface area and filled part of pores. Thus moisture absorption was mainly related to hydrophilic groups of PEG.

Tensile properties of the membrane

Mechanical properties of the membrane with different PEG content are presented in table 2. It shows that as PEG mass percentage increased, breaking strength, breaking elongation and initial modulus of the membrane decreased obviously. The addition of PEG blocked the aggregation of PU macromolecule during film formation and occupied some space, which made membrane structure became loose. After membrane formation, when the membrane was drawn, the crystallization and adhesion of PEG would restrain the extension of PU macromolecule, which reduced macromolecule amounts loaded at the same time and thus increased non-simultaneous breakage of the membrane. As a result, breaking strength and extension and initial modulus of the membrane decreased to some extent.

When weight ratio of PEG and PU was kept constant at 3:1, the membrane was made with different DMF content. The tensile properties of the membrane formed of different DMF content are shown in table 3. It indicated that tensile strength, breaking elongation and initial modulus of the membrane decreased with the increase of DMF content in solution. The increase of solvent weight ratio affected the aggregation of PU macromolecule, and after DMF volatizing, bigger pores were formed in the film. As a result, the lateral bonding of the membrane was weakened and mechanical properties were decreased. Therefore DMF content in film-forming solution should not be too high.

Bending rigidity of the membrane

Bending length is an index of bending rigidity and they are positive interrelated. As figure 6 showing, bending length of the membrane with different PEG content decreased gradually as testing temperature increased.

As environment temperature increased, phase change material PEG became soft gradually until it changed to liquid state from solid state. Thus the change of temperature produced huge effects on PEG morphology and rigidity. Correspondingly membrane flexibility depended on environmental temperature largely.

Figure 6 also shows that the relationship between bending length and PEG content was complex and depended on testing temperature largely. It can be observed that membrane bending length increased with the increase of PEG percentage at 10°C, but decreased at 50°C. It was mainly due to phase change behavior of PEG. PEG has strong crystallization under phase change temperature and lower

Table 2

THE TENSILE PROPERTIES OF THE MEMBRANE WITH DIFFERENT PEG CONTENT

PEG content, %	Breaking extension, mm	Breaking strength, MPa	Initial modulus, N/mm^2
47.8	307.7	0.6853	6.327
62.2	263.6	0.4785	5.324
74.9	153.8	0.2969	3.342
80.1	110.3	0.2131	2.634
84.6	84.2	0.1945	2.261

Table 3

THE TENSILE PROPERTIES OF THE MEMBRANE FORMED UNDER DIFFERENT DMF CONTENT

PEG content, %	Breaking extension, mm	Breaking strength, MPa	Initial modulus, N/mm^2
0.5	252.7	0.4579	4.564
0.8	241.5	0.4193	3.91
1.0	153.8	0.2969	3.342
1.2	129.0	0.2483	2.979
1.5	139.5	0.2352	3.322

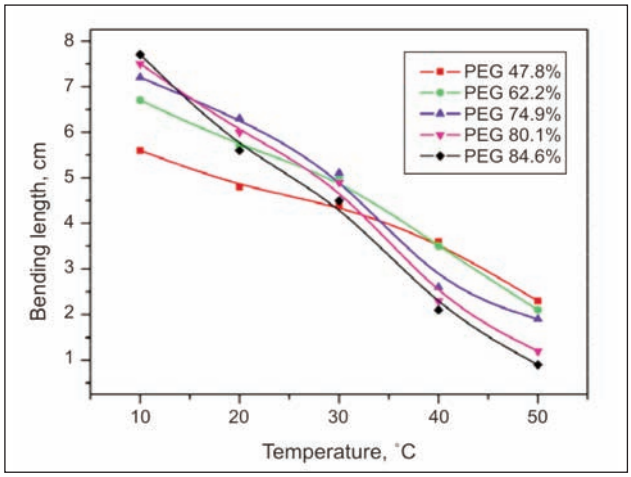


Fig. 6. Bending length of the membrane under different temperature

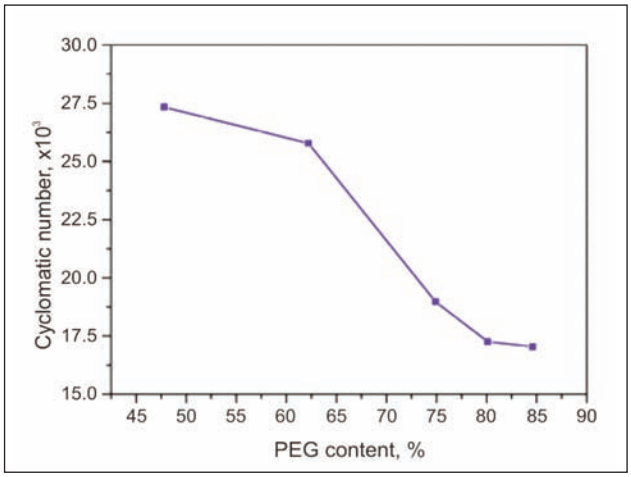


Fig. 8. The relationship between abrasion resistance and PEG content

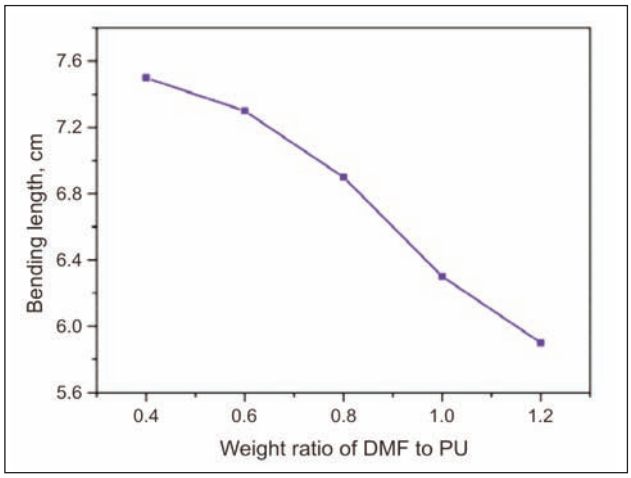


Fig. 7. The relationship between bending length and weight ratio of DMF to PU

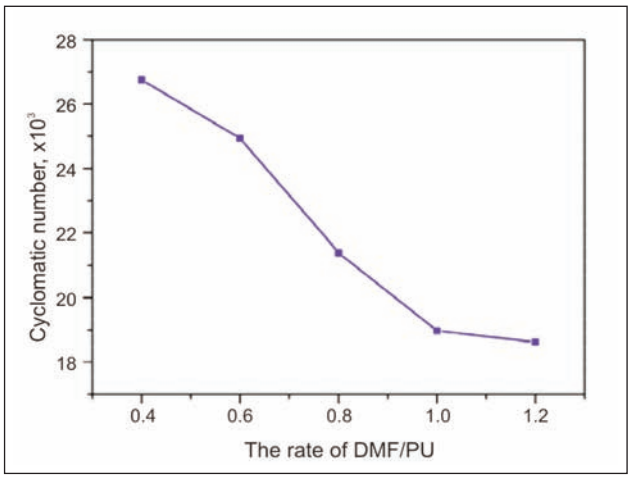


Fig. 9. The relationship between abrasion resistance and ratio of DMF to PU

flexibility than PU. And thus at lower temperature PEG content addition resulted in the increase of membrane bending rigidity. However, when ambient temperature reached above transit point (about 30°C), the whole membrane became very soft and was easy to bend. While around transit point both porosity structure and phase change behavior of PEG played important roles on bending rigidity of the membrane. Bending length of the membrane formed with f different DMF content is shown in figure 7. Obviously the bending length decreased with the increase of DMF content. The increase of solvent DMF resulted in the increase of the porosity and the decrease of the initial modulus of the membrane. Thus the bending rigidity and bending length of the membrane decreased.

Abrasion resistance of the membrane

Abrasion resistance measurement results of the membrane with different PEG content are shown in figure 8. It is observed that the abrasion resistance decreases as PEG content increases. The attenuation tendency slows down while PEG percentage reached 80%. It is mainly due to the increase of PEG content resulting in the dramatic reduction of mechanical properties including breaking strength and elongation, initial modulus and lateral bonded strength. Thus the surface layer of the film was worn out and tore more easily. When PEG content reached higher percentage, PEG formed continuous phase in the membrane, and thus slowed down the reduction tendency of abrasion resistance.

Figure 9 shows the effects of DMF content on abrasion properties of the membrane. The results are similar to the effects of PEG content. The increase of DMF content leads to the breakdown of mechanical properties.

Moisture permeability of the membrane

The membrane is made of carrier material (PU porous membrane) and work material (PEG). The first has moisture permeability function through micro pore and the latter has moisture permeability function through hydrophilic groups. Thus the membrane has dual moisture permeability function.

As table 4 showing, with the increase of PEG, moisture permeability of the membrane increased gradually. The increase of PEG content expanded the size of pore in the membrane, which may enhance

Table 4

MOISTURE PERMEABILITY OF THE MEMBRANE WITH DIFFERENT PEG CONTENT					
Weight ratio of PEG to PU	1/1	2/1	3/1	4/1	5/1
Moisture permeability, g/m^{2}h	4 126	4 378	4 532	4 663	4 729

moisture permeability through micro pore. In addition, the increase of PEG content increased the density of hydrophilic ether link which is typical nonionic hydrophilic groups, and thus moisture transferred along these "molecule steps" more easily and moisture permeability was enhanced further.

Moisture permeability of the membrane (PEG:PU = = 3:1) under different temperature was measured. The results are presented in figure 10. Obviously moisture permeability increases with the increase of environment temperature and there is a sharp increase between 30°C and 50°C. It may be due to that phase change happened in the membrane and PEG macromolecule thermal motion was accelerated, which resulted in the increase of micropore instantly and enhanced the transport of moisture.

Human body usually feels hot and sweats at this temperature range. Sweat moisture can penetrate through the membrane and at the same time the melting phase change of PEG is endothermic reaction. Thus correspondingly the temperature of microclimate can be reduced. Under phase change temperature, PEG in membrane began to be consolidation and crystallization and release latent heat. Though moisture permeability decreased, it can still satisfy the need of sweat transferring and keep the comfortability of the fabric. By combining moisture permeability with thermal activation, the membrane possesses auto-adjusting moisture permeability function.

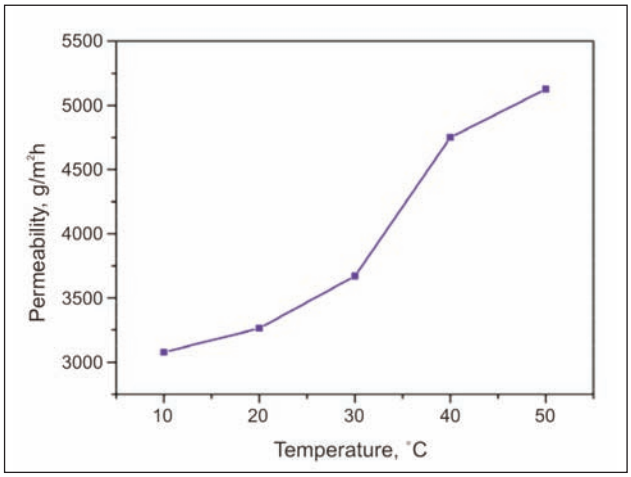


Fig. 10. Moisture permeability of the membrane under different temperature

CONCLUSIONS

Porous phase change membranes were prepared with PEG1000/2000, PU, DMF and $(NH_4)_2CO_3$ powder. Morphology observation showed the smooth surface and porous sublayer structure of the membrane. Phase change behavior measurement indicates that the compound membrane has suitable transition temperature and high transition enthalpy. Measurements of mechanical characters showed that the tensile properties and abrasion resistance of the membrane decreased with the increase of PEG content and DMF content in film forming solution respectively. The bending rigidity of the membrane decreased with the increase of the weight ratio of DMF to PU and the ambient temperature respectively, and it depended on environmental temperature largely. The results of moisture permeability testing indicated that the increase of PEG content strengthened the moisture permeability of the membrane through micropores and hydrophilic groups. And the moisture permeability of the membrane increased with the increase of testing temperature. In summary, the membrane has a great potential in the textile for thermal energy storage function.

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INDUSTRIA TEXTILĂ ÎN LUME

CONSORȚIU EUROPEAN PENTRU DEZVOLTAREA COMPOZITELOR TERMOPLASTICE

În octombrie 2012, cinci organizații olandeze – ***TenCate Compozite Avansate al Nijverdal, DTC Dutch Thermoplastic Components of Almere, Kok & Van Engelen din Den Haag, National Aerospace Laboratory*** of the Netherlands ***şi VIRO Hengelo***, au încheiat un parteneriat în domeniul compozitelor termoplastice, destinate industriei de automobile.

Obiectivul principal al creării consorțiului european ***eTAC*** a fost promovarea utilizării în sectorul auto a unor materiale compozite avansate, dezvoltate prin tehnologii inovative în domeniul materialelor, proiectării, producției şi automatizării proceslor tehnologice. *TenCate*, o companie de top la nivel mondial, activă în domeniul textilelor tehnice şi al materialelor avansate, afirmă că partenerii săi au mulți ani de experiență în exploatarea compozitelor termoplastice uşoare, folosite în industria aeonautică, având roluri importante în producerea şi prelucrarea unor astfel de materiale.

În prezent, prin folosirea compozitelor termoplastice, se poate obține, în multe cazuri, o reducere a masei autovehiculelor şi, în consecință, a consumului de energie. Deoarece capacitatea de prelucrare nu atinge încă viteza cerută pentru producția de masă, aplicațiile s-au limitat la autovehiculele de lux, realizate la scară mică şi la prețuri mari. Având în vedere acest impediment, pentru producția de masă, partenerii vor dezvolta o poliamidă ranforsată cu fibre de carbon.

Acest consorțiu îşi propune să realizeze un punct de contact unic, care să acopere toate aspectele legate de prelucrarea şi producția de automobile. Atât producătorii, cât şi furnizorii vor avea acces la informații de specialitate de ultimă oră şi la soluții pentru problemele lor. Prin birourile din Olanda, Marea Britanie şi Germania expertiza este realizată la nivel internațional şi acoperă întregul lanț de aprovizionare, inclusiv testarea, validarea şi producția pilot.

Consorțiul va continua să lucreze cu rețelele deja existente şi cu centrele şi instituțiile de cercetare din Olanda şi din străinătate. Totodată, sunt căutați şi alți parteneri, care să lucreze pe proiecte individuale în domeniul auto, sau să devină membri permanenți ai eTAC.

Technical Textiles International, octombrie 2012, p. 2

Virtual prototyping design of bulletproof vestsg

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REZUMAT – ABSTRACT

Proiectarea virtuală a tiparelor vestelor de protecție balistică

Proiectarea în spațiul virtual este un domeniu de mare interes, atât în grafica asistată de calculator, cât şi în proiectarea industrială a produselor şi bunurilor de larg consum, respectiv a produselor de îmbrăcăminte. Lucrarea prezintă o variantă de simulare a modului de aşezare a vestei de protecție balistică pe manechin, în scopul verificării tiparelor proiectate/prototipării, respectiv a gradului de acoperire/protecție pe care îl poate asigura. Simularea în spațiul virtual a produsului îmbrăcat îi permite producătorului să optimizeze numărul de prototipuri fizice necesare pentru testele reale de validare a vestei şi pentru stabilirea nivelului optim al parametrilor tehnologici necesari fabricației.

Cuvinte-cheie: vestă de protecție balistică, prototipare, simulare în spațiu virtual

Virtual prototyping design of bulletproof vests

Design in virtual environment is an area of great interest, both for computer-aided graphics and industrial products and consumer goods, or garments. This paper presents a simulation variant of how is laying a bulletproof on a mannequin in order to verify the pattern design, coverage and protection area which can provide. The simulation in virtual space of bulletproof vest dressed on mannequin allows the manufacturer to optimize the number of physical prototypes needed to validate it and to establish the optimum level of technological parameters for manufacturing process.

Key-words: bulletproof vest, prototyping, simulation in virtual environment

A major concern for all countries is to protect against all forms of military aggression. During its mission of fighting, soldier or police officer should be protected against environmental factors (cold, moisture, heat, sunlight), the risks of battle – ballistic fragments, bullets, knives, chemical and biological agents, flame and heat [1]. Major requirements of protective equipment are:

- physical requirements (durability, wear resistance, tensile strength);
- physiological needs (lightweight, comfort wear, maintaining mobility action);
- requirements the field of battle (mechanical shock protection, flame resistance, resistance to chemical and biological).

Appearance of "smart materials" determined important changes in production of special protective clothing and offered the possibility to re-design the whole system of fighter equipment [2–5].

Currently, worldwide attention focuses to military and police personnel to protect them against bullets and splinters and from this reason the textile equipment has to become more efficient.

THE PRINCIPLE OF WORKING METHOD

A ballistic resistant body armor means a capacity (tested in laboratory and on the battlefield) to stop firearms projectiles to the level of protection that you should provide.

A ballistic impact protection system, as used in protective clothing, absorbs the impact of a gun-fired projectiles fired from handguns, shotguns, and small fragments from explosives such as hand grenades. Soft body armor is products that are generally made up of different layers of woven, laminated films and outer containment covers [6, 7]. The choice of material, number of layers and lay up direction of fibers (filaments) all have a significant effect on the ballistic performance, respectively a protection level according to the international standards [8].

The ballistic vest consists of several layers of ballistic fabric such as Kevlar® stitched together. The strength of the vest can vary mainly due to material type and number of plies. The rigid armor has a concave shaped plate and provides enhanced ballistic protection. Materials for the plate consist of a ceramic face with a fiber reinforced back. The plates can vary due to material type, ceramic thickness, backing thickness, and resin used to bond the materials together. Typically, the plate is placed on the chest and back of a soldier, where it protects the major organs located in these regions.

There are several types of ballistic fabrics that are used today, but among them Kevlar® is the most commonly known ballistic aramid fiber [3, 7]. It is a product of DuPont company and has had several generations of development. Kevlar® 29 was the first ballistic protective material developed. Then new generation of Kevlar® 49, Kevlar Protera and Kevlar

KM2® consists of organic fibers, which have long molecular chains that are highly oriented with strong interchain bonding. This results in properties that are excellent for high stress applications. For example, Kevlar® 29 is stronger than steel at 1/5th the weight. Kevlar's dynamic strength properties include high tensile strength to low weight, low elongation to break high modulus (structural rigidity), high toughness (work to break), high cut resistance, flame resistant and excellent dimensional rigidity [9].

The standard structure of a bulletproof vest involves a several layers of ballistic material protection, assembled in a called "ballistic package". This package is inserted into a pouch made of nylon or cotton fabric. Ballistic package can be permanently fixed in or removable cover.

The number of layers in the package structure can vary quite wide limits (from 10–14 to 32–40), depending on the characteristics of the material used for ballistic protection. An example of ballistic package is presented in figure 1 and has the following features:

- *Base material* – cordura 560 fabrics treated to resist fire and covered with a waterproof film;
- *Lining* – cotton (67%) and polyester (33%). It is necessary to combine the cotton with polyester because the cotton has a low resistance to the action of agents;
- *Ballistic-protection materials* – Kevlar in several number of layers (for example 22 layers) to achieve Level IV ballistic protection (according NIJ0108.01) and a plate of Twaron ceramic composite and ceramic materials;
- *Accessories* – Velcro straps of different widths and sizes to fix the vest on body.

Vests are manufactured for two heights (I and II) and several sizes. According to the level of risk mission and environment the outside material can have a design that mimics the surrounding nature, to ensure a high protection/camouflage (fig. 2 *a, b*).

The patterns vest are designed knowing the values of several dimensions (table 1). These patterns are made

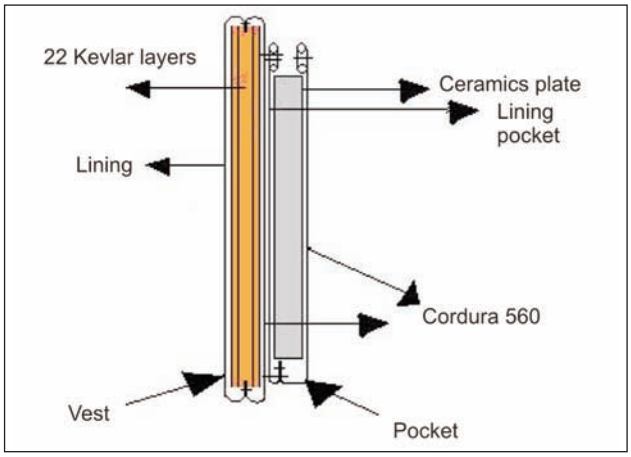

Fig. 1. Structure of ballistic package

a ***b***

Fig. 2. Variants of bulletproof vest:
a – bulletproof vest for wood conditions;
b – bulletproof vest for desert conditions

Table 1

BULLETPROOF VEST SIZING TABLE (EXAMPLE)

Symbol	Dimension description	Sizes/Values, cm			TOL +/–
		48	52	56	
A	The front width at 5 cm lower from neckline (from middle point)	32.5	33.5	34.5	1
B	The back width at 10 cm lower from neckline (from middle point)	32	33	34	1
C	The front width at waist level	47	52	57	1
D	The back width at waist level	80	85	90	1
E	The back length on middle line	40.5	42.5	44.5	1
F	The front length on middle line	3	38	40	1
G	The neckline width	5.5	5.5	5.5	0.5
H	The shoulder length	8	8	8.5	0.5
I	The back width at shoulder level	39	40	41	0.5
K	The length of front plate pocket	35	35	35	0.5
L	The width of plate pocket (front and back)	29	29	29	0.5
M	The length of back plate pocket (without flap)	32.5	32.5	32.5	0.5
N	The width of horizontal back pocket	29	29	29	0.5
O	The flap width of back pocket	6	6	6	0.5

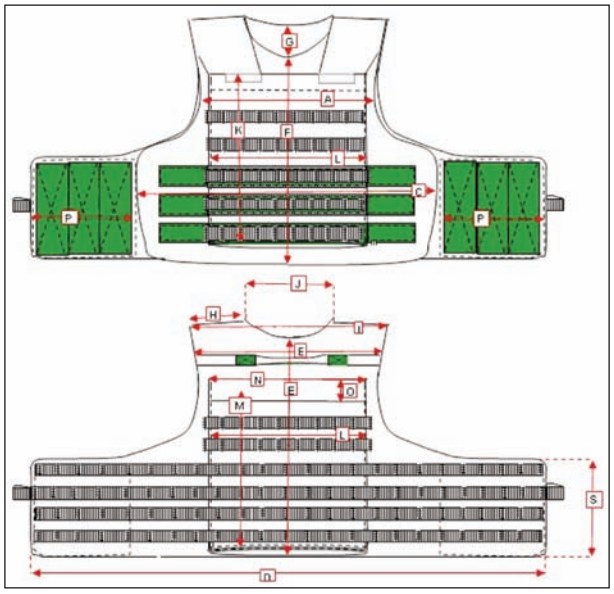

Fig. 3. Bulletproof vest characteristic dimensions

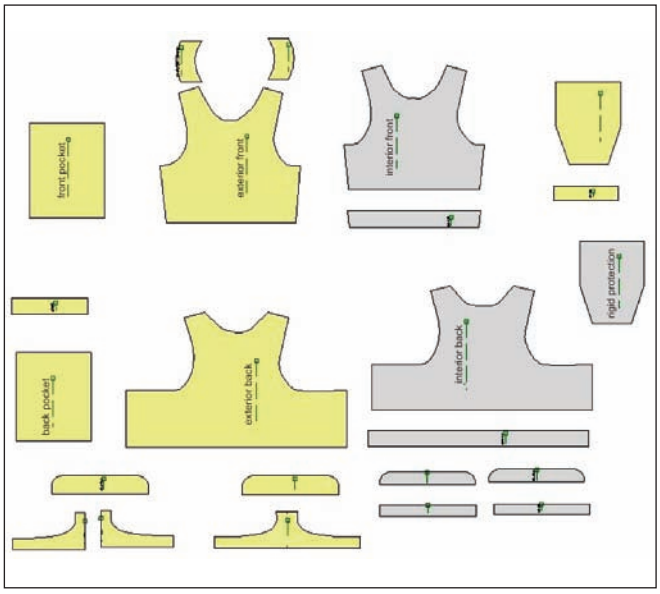

Fig. 4. Bulletproof vest pieces

either using parametrical design programs (Autodesk) or specialized design systems (CAD systems: Gemini, Gerber, Lectra, Optitex etc.) knowing the values of characteristic dimensions (fig. 3). For example, the final shape of bulletproof vest pieces (fig. 2 *b*) designed with GEMINI CAD System (GPE – Gemini Pattern Editor) is presented in figure 4.

After 2D design patterns is required the test of covering and protective capacity. The bulletproof manufacturing and test process involve sometimes-higher costs, because the test needed to repeat in order to establish the optimal structure, size and correct position of ballistic package [10, 11].

Clothing modeling in 3D environment has progressively become a topic of large investigation for computer science. Since the early '90s, scientific and commercial communities have developed several cloth modeling systems having in minds different points of view and goals. Basically, two main categories of software products can be distinguished: *a* – for cloth visualization; *b* – for cloth design. IT applications allows cloth visualization aim at producing images that look real for computer animation applications, while systems for garment design focus on the definition and construction of functional cloth shapes for real manufacture (e.g. clothing, upholstery etc.). These applications are very good to be used to design protective bulletproof clothing.

The development of 3D design software, such as Modaris 3D Fit, Optitex, Autodesk 3dsMax etc. allow the simulation of a dressed bulletproof on mannequin in virtual environment. By this way is possible to verify the vest size, geometry, structure and position of ballistic package in order to reduce the design costs from manufacturing process (sometimes is possible to reduce the test costs of bullet protection).

The main steps, which must be taken for a bulletproof vest simulation dressed on mannequin (fig. 2 *b*) using 3dsMax program, are as follow:

- Launching the programmer (from *Program files* or by double click the shortcut icon from desktop); after launching, the following image appears: main menu, toolbar menu, side up-down menu and 4 viewport for visualize different working stages – top, left, front and perspective (fig. 5);
- Mannequin design stage/or import a mannequin according to the purpose: design a bulletproof vest; the mannequin must be dressed because under the vest it must have a layer of underwear and than a shirt;
- Import patterns designed in 2D programs. The program allows importing the following file types: *.3 DS, *.DWG, *.DXF, *.IPT, *.IAM etc. The patterns of bulletproof vest from figure 2 *b* were obtained using different commands and function from Gemini CAD system. In the first stage, there were designed the patterns of the main elements of the bulletproof vest, front and back and than, those draws were graded, according to dimensional values from table 1. The main patterns (front and back) were splits into different pieces, for designing all the structural elements of the garment. In conclusion, the bulletproof vest simulation can be done using the main patterns (front

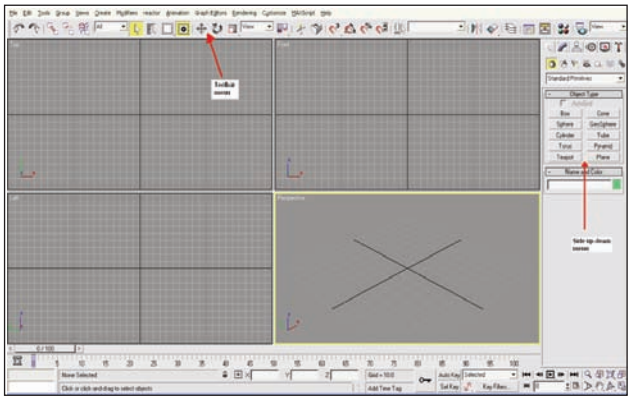

Fig. 5. 3dsMax starting working session

and back) but considering the number and thickness of all materials from its structure;

- Positioning the patterns near the mannequin. The patterns are positioned by applying different commands, such as: selection, rotation and displacement. The programmer imposes to convert each selected pattern into an editable spline curve (fig. 6);
- The transformation of spline curves in mesh surface. In side up-down menu, we select *Garment Maker* and in automatically way all available on the desktop patterns from the working session are transformed into mesh surface. For a good positioning phase near mannequin each pattern (panel) is rotated (using Rotate command), displacement (using Move command) and than modeled for a good position. It is very important to check the correct placement of the pattern: the outer side must be on exterior and backside to the mannequin (figure 7 and figure 8);
- Establishing the sewing lines. The sewing line are declared by selecting the needed line (with Ctrl key activated) and than by selecting the *Create Seam* button, from *Garment Maker* – side up-down menu (fig. 9) [7];
- The bulletproof vest simulation on mannequin. The simulation is performed in successive steps:
  - from side up-down menu is selecting *Cloth* button (*Modifier List*);
  - from Object sub-menu is selecting *Object Properties* button and after that is choosing the material from which the vest is made and parameters for the material [4, 5];
  - than is selected the ballistic plate and mannequin to interact with the bulletproof vest. After selection, for ballistic plate it is choose its material and for the mannequin is established the collision distance at 0.3 values (fig. 10). The simulation is made for a structure with 5 Kevlar layers, 1 metal mesh and a ballistic plate of 22 mm thickness;
  - from side up-down menu, *Object Properties* → *Simulation Parameters* is disabled the *Gravity* button. From side up-down menu *Simulation*, is selecting *Simulate local (damped)* button and all the vest layers are approaching to the mannequin, according to all initial data. The process can be interrupted by activating the Esc key. After re-activating the *Gravity* button and *Simulate local (damped)* button the process may continue until final stage (fig. 11). After placing the vest on the mannequin it is analyzing: the position of ballistic plate, how does it fit with the body and how does it protect the body against bullets action.

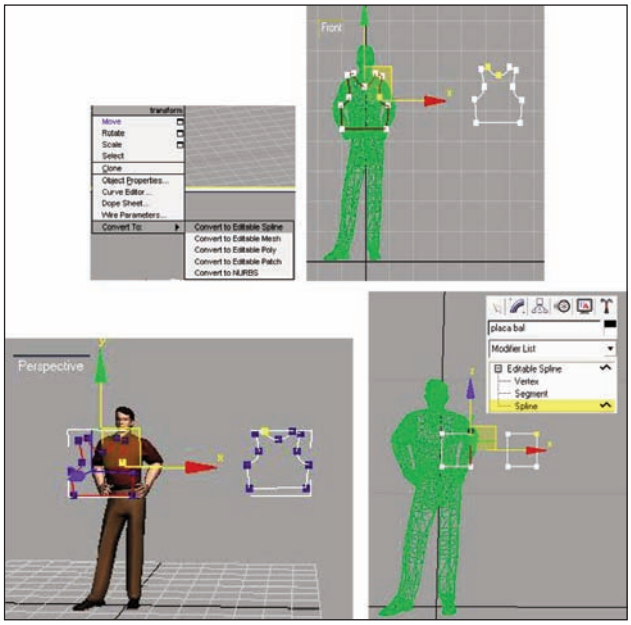

Fig. 6. Patterns movement and positioning

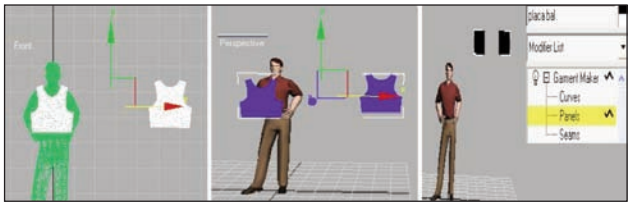

Fig. 7. The patterns positioning phase

Fig. 8. Patterns positioning and modeling phase

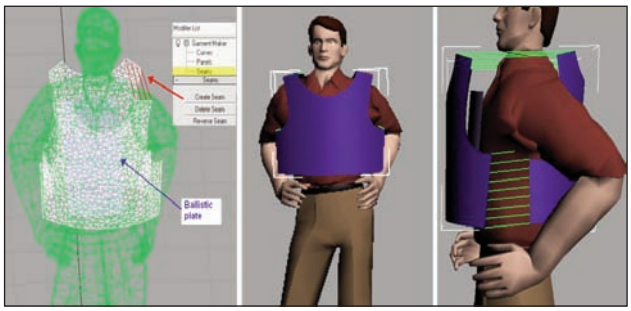

Fig. 9. Declaring sewing lines

When the bullet hits the fabric, the wires induce a shock wave. The first wires achieved by resisting moves to the connection points of the fabric, where they encounter other wires come together. Such induced shock wave can propagate in a much larger number of fibers, which has a positive effect: the whole energy of the projectile is absorbed over a larger area of fabric. Speed train where energy dispersion is a direct relationship of interdependence of fiber modulus. In fact, a bulletproof vest has two main objectives: catching the bullet and reducing physical trauma because of the impact.

Materials used to manufacture the bulletproof vest consist of several layers of extremely strong fibers

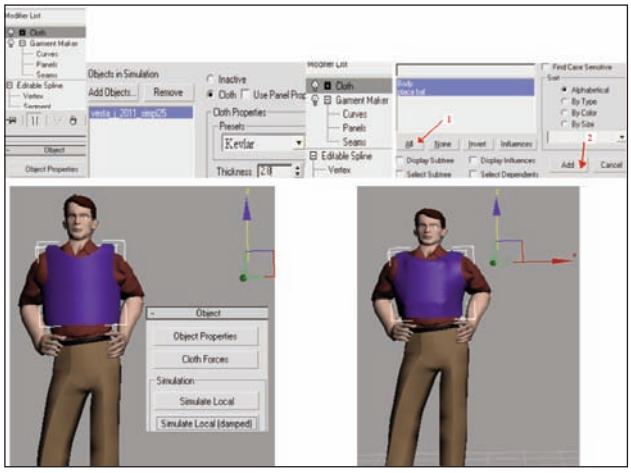

Fig. 10. Simulation stages

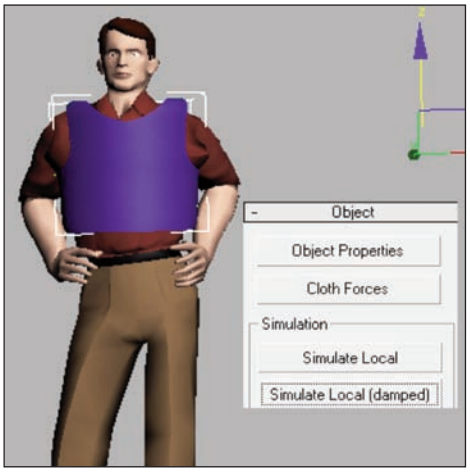

Fig. 11. Intermediary and final stage of simulation

that catch and deform a bullet, dispersing its force over a larger portion of the vest fibers. A deformable handgun bullet mushrooms into a dished plate on impact. The unique fibers absorb the energy from the deforming bullet, bringing it to a stop before it can penetrate and causing blunt force trauma under the impact point (fig. 12).

When the projectile speed exceeds the ballistic limit, the absorption capacity of the system decreases very fast. In the same time, the fabric deformation increases with the decrease of distance of point impact. Such vests are working under the principle of a wire mesh. After penetration and braking in the first layer, splinters or bullets, if they have not penetrated through the wires (bullet-pointed), are stopped by the fibers that were not broken.

After the process is finished, it is checking: completion of the process, check:

– dimensional correspondence between the vest and mannequin;
– geometry contour lines;
– the placement of ballistic plate;
– the aspect of sewing lines, fixed systems and generally vest aspect;
– the degree of deformation of the layers;
– the shape and aspect of impact area.

According to the results it is possible to adopt the following changes: the size patterns, the geometry of contour lines, to use a different solution to fix the vest on mannequin, to change the number and type of materials from the vest structure and the position of ballistic plate.

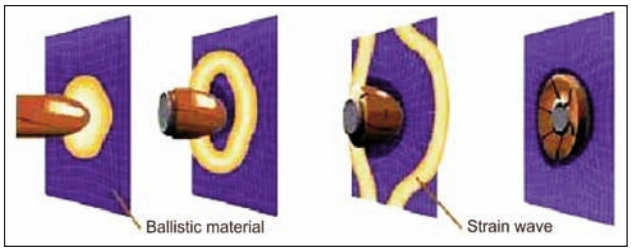

Fig. 12. Testing vest with bullet

CONCLUSIONS

The main military nations have research programs geared towards future combat and protective clothing as integrated systems. The programs tend to be led by military threats or capability gaps doctrine, rather than exploitation of new technologies for the sake of it.

The systems approach involves all the major stakeholders, including, strategic planners, users, equipment capability managers, operational analyzers, R & D scientists, producers, contracts staff etc.

The important parameters to be kept in mind are:

– improve protection against natural and battlefield threats, of different type;
– maintain thermo-physiological comfort or survival in extreme conditions;
– improve compatibility between and within different clothing components;
– improve manufacturing technology and develop new materials to upgrade safety condition;
– to reduce manufacturing costs and to ensure the environment protection by using IT to check bullet-proof vests (dimensional and functional point of view);
– reduce weight and bulk of materials.

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INDUSTRIA TEXTILĂ ÎN LUME

DEZVOLTAREA POLIAMIDEI CONSOLIDATE CU FIBRE DE CARBON

Două mari companii germane, ***BASF SE***, cu sediul în Ludwigshafen – un important producător de poliamidă şi ***SGL Group***, cu sediul în Wiesbaden – un specialist în fibre de carbon, şi-au propus o colaborare în domeniul dezvoltării şi eficientizării materialelor compozite, destinate producției de masă a componentelor auto.
Principalul obiectiv al acestui parteneriat îl constituie crearea unei matrice reactive din poliamidă consolidată cu fibre de carbon, special dezvoltate şi tratate în vederea modelării prin transfer de răşini termoplastice şi turnării prin injecție a reactivilor, ambele tehnici fiind considerate mai rapide şi mai economice decât cele tradiționale.
Cu ocazia *Expoziției europene a materialelor compozite,* care s-a desfăşurat la Düsseldorf – Germania, în perioada 9 – 11 octombrie 2012, cele două companii au purtat discuții cu vizitatorii standurilor cu privire la posibile colaborări în acest domeniu.
BASF – în calitate de important producător de poliamidă şi caprolactamă, un precursor de bază al poliamidei, va contribui la dezvoltarea sistemelor de matrici polimerice, în timp ce *SGL Group* se va concentra pe dezvoltarea fibrei de carbon şi a proceselor de prelucrare la temperaturi înalte.
Şeful Departamentului de Tehnologie şi Inovație al SGL Group, Dr. Hubert Jäger, sublinia faptul că numai printr-o finisare specială se poate crea o legătură optimă, care să permită fibrelor de carbon să transfere proprietățile lor unice de rigiditate şi rezistență materialelor realizate.
De asemenea, Şeful Departamentului de Cercetare a Materialelor Structurale, Dr. Martin Jung, arăta că, pentru a obține o bună umezire a fibrei şi pentru scurtarea duratei ciclurilor în procesele de modelare prin transfer de răşini termoplastice şi turnare prin injecție a reactivilor, BASF va utiliza o caprolactamă foarte reactivă, cu vâscozitate redusă.

Technical Textiles International, octombrie 2012, p. 2

Chitosan and its derivatives as an adsorbate for cellulose fibres' anti-microbial functionalizations

LIDIJA FRAS OLIVERA ŠAUPERL

REZUMAT – ABSTRACT

Utilizarea chitosanului şi a derivaților săi ca adsorbanți în funcționalizarea antimicrobiană a fibrelor celulozice

Lucrarea are ca scop un studiu comparativ privind utilizarea potențială a chitosanului comercial (a grupelor amino primare) şi a derivaților acestuia (a chitosanului cu compuşi cuaternari de amoniu) ca adsorbanți în funcționalizarea suprafețelor textile. Au fost analizate proprietățile fizico-chimice şi s-a pus accent pe determinările grupelor funcționale ale fibrelor şi pe caracterizarea proprietăților antimicrobiene ale fibrelor celulozice funcționalizate. S-a ajuns la concluzia că chitosanul cuaternar, în comparație cu chitosanul comercial (grupele amino), are o capacitate mai mare de adsorbție şi de introducere a grupelor amino de pe suprafața fibrelor, contribuind, astfel, la o eficiență antimicrobiană ridicată.

Cuvinte-cheie: chitosan, chitosan trimetilic, adsorbție, grupe amino, funcționalizare antimicrobiană

Chitosan and its derivatives as an adsorbate for cellulose fibres' anti-microbial functionalization

The purpose of this paper was to carry out a comparison between commercial chitosan (primary amino groups) and its derivatives (chitosan with quaternary ammonia compounds) regarding their potential usage as adsorbates for the functionalization of textile surfaces. The physical-chemical properties were analysed with an emphasis on the fibre functional groups' determinations, plus a characterization of the antimicrobial properties of the functionalized cellulose fibres. It was concluded that quaternary chitosan when compared to commercial chitosan (amino groups), adsorbs in a larger quantity on the fibre surface, and introduces a higher content of available amino groups on the fibres' surfaces, thus consequently leading to greater antimicrobial efficiency.

Key-words: chitosan, tri-methylated chitosan, adsorption, amino groups, antimicrobial functionalization

Medical textiles is one of the faster growing fields within technical textiles. This fast development can be attributed to several factors, such as: population growth and ageing, life-style changes, and increased awareness and concern about healthy living and health in general as well as increases in contagious bacterial diseases and hospital infections etc. Nowadays, the surface modification of textile fibres is considered to be the best route for obtaining advanced textile treatments [1–9]. Several functionalization methods are available for acquiring antimicrobial qualities for textile materials [3–11]. Most of these methods still employ reagents harmful to humans and the environment, and include inorganic salts, phenols and thiophenols, antibiotics, formaldehyde etc. It is for these reasons that the usage of those functionalization procedures that apply environmentally-friendly and biodegradable reagents have been increasing, such as amino polysaccharides and their derivatives with antibacterial properties. From amongst them, the more commonly used is the polysaccharide, chitosan. Chitosan is an abundant biopolymer, consisting of poly [β-(1-4)-2-amio-2-deoxy-D-glucopyranose] – which is obtained after the alkaline deacetylation of chitin, as found in the exoskeletons of crustaceans, arthropods, and mollusks, as well as the cell-walls of certain fungi [12].

Chitosan has two important structural parameters, these being the degree of deacetylation (DD) and molecular weight (MW). Its performance within physics and chemistry is determined by the influences of these two parameters on such things as solubility, enrichment ions, the mechanics of the chitosan membrane, flocculation etc. [12]. The amino groups are responsible for antimicrobial activity. In acidic solvents, the NH_2 group of chitosan becomes a cation (NH_3^+) that allows the chitosan to inhibit the growths of several bacteria, including gram-negative and gram-positive ones [13, 14]. It is for this reason that chitosan is extremely popular for medical textile development. To date, different textile fibres have undergone broad surface modifications using chitosan, in order to stimulate antimicrobial activity. Niekraszewicz [15] evaluated a chitosan-polypropylene non-woven prepared according to the wet-paper method of production. Microbiological tests showed antimicrobial activity, without any cytotoxicity detection. The usage of chitosan as an antimicrobial agent for the treatment of polypropylene non-woven for surgical covers has been researched using different types of hospital bacteria. Those textiles treated in

such a way were effective against both gram-negative and grampositive bacteria [16]. Sakai et al. [17] dissolved chitosan in water using carbonic acid gas (CO_2) *via* preparation of the gel. Cotton gauze treated with chitosan-CO_2 solution effectively inhibited Staphylococcus aureus. A very pertinent example of functionalization was a cotton fabric surface modified by chitosan that absorbed antibiotic molecules from an aqueous solution. The quantity of absorption depended on the degree of the sample's modification. The higher the degree of modification, the higher the amount of antibiotic bonded to the textile. Such cotton textile finishing enables the achievement of therapeutic new-generation dressings for the protection of surgical wounds against infections [18]. Zemljic et al presented the functionalization of viscose cellulose material with chitosan for potential applications in cellulose tampons for gynaecological use [19]. However, despite chitosan's common and widespread usage and popularity, it has certain drawbacks, especially limited solubility and, consequently, *p*H-dependent antimicrobial activity. Also, difficulties with purification also require a high-level of analytical support. Due to the nature of its conventional production from animal origins (crustaceous), it is very difficult to produce reproducible material with defined properties. A broad spectrum of derivatives can be gained from chitosan by using chemical modifications for the purpose of improving its characteristics.
These include chito-oligosaccharide [20, 21], N-(2-hydroxy) propyl-3-trimethylammonium chitosan chloride [22–24], N-p-(N-methylpyridinio)methylated chitosan chloride, and N-4-[3-(trimethyl-ammonio) propoxy] benzylated chitosan chloride [25]. Many of these derivatives contain a quaternary ammonium group in order to enhance their antimicrobial activities. The purpose of this study was to compare commercial chitosan (primary amino groups) with its derivatives (chitosan with quaternary ammonia compounds), regarding their potential usages as adsorbates for cellulose fibres, in order to develop medical textiles and devices. Functionalized cellulose fibres were characterized in terms of amino group content determination and antimicrobial activity, respectively.

EXPERIMENTAL PART

Materials and methods

Viscose fibres

Regenerated viscose cellulose fibres were obtained from Lenzing AG, Austria. The linear densities of all the fibres were 1.3 dtex and the fibre lengths were 39 mm, respectively.

Pre-treatment of viscose fibres

Conventional cleaning procedures were used in this experiment, as commonly used as pre-treatment processes during textile praxis: alkaline washing, bleaching and demineralisation. Alkaline washing is aimed at removing oils, antistatic agents etc. Bleaching decomposes those substances such as colours, pigments etc. that are hard to remove by alkaline treatment. The demineralization process is required in order to remove metal ions and to convert fibres into their *H*-form. These procedures are listed in table 1. Following this treatment, the samples were rinsed using distilled water until the conductivity of the rinsing water was less than 3 µS/cm. The processed material was air-dried and air-conditioned (T = 20°C ± ± 2°C and relative humidity = 65% ± 2%).

Chitosan and its derivatives

The chitosan used for fibre impregnation was chemically-graded, low-viscous chitosan (M = 100 000; 90% de-acetylated) from Gillet Chitosan, France. It was used without further purification. The water-soluble chitosan derivatives, N,N,N trimethylated chitosan (TMC), derived from ultra pure chitosan produced by Kytozyme (Belgium) (M = 90 000; 80% de-acetylated, a substitution degree of 80% determined by H-NMR) was used as a chitosan derivate.

Other materials

All the chemicals were purchased from Sigma Aldrich (HCl, NaOH and Na_2CO_3), Kemika Zagreb (KCl), Baker Dilut-it (KOH), Sandoz (Sandoclean PC), Tanatex (Tanatex Geo), Belinka (H_2O_2) and were also commerciall-graded.

Impregnation of fibres using both chitosan acidic solutions

Chitosan and its derivate TMC were adsorbed onto viscose fibres, respectively, by soaking the fibres (5 g) in 0.5% of 100 ml acidic chitosan solution (*p*H adjusted to 3.6 by adding 1 M hydrochloric acid) for 10 minutes, as described by YS Chung et al. (1998) [26, 27]. The fibre impregnation was performed by passing through a foulard impregnation press (W. Mathis) at a pressure of 1.6 bars. After this treatment, the fibres were dried at T = 40°C and t = 60 minutes, and further air-conditioned for 48 hours. Finally, the functionalized fibres were washed with distilled water until the conductivity of the water was less than 3 µS/cm. The processed material was air-dried and air-conditioned (T = 20°C ± 2°C and relative humidity = 65% ± 2%). The samples' notations are given in table 2.

Analytical methods

Potentiometric titration

A twin burette instrument (Mettler T-70) was used, equipped with a combined glass electrode (Mettler T DG 117). The burettes were filled with 0.1 M HCl (Merck, Titrisol) and 0.1 M KOH (Baker, Dilut-it).
All the solutions were prepared using deionized water with a very low carbonate content (< 10–6 M), which was achieved through boiling, and then subsequent cooling, within a nitrogen atmosphere.
The pre-treated viscose fibres, and those fibres functionalized within both chitosan solutions (0.5 g of oven-dry fibres), were all titrated in forward and back runs between *p*H = 3 and *p*H = 10. In order to avoid

Table 1

TREATMENT CONDITIONS FOR VISCOSE CELLULOSE FIBRES' PRE-TREATMENT PROCEDURES		
Alkaline washing	**Bleaching**	**Demineralisation**
1 g/L Na_2CO_3	6 mL/L H_2O_2	0.01 mol/L HCl
1 g/L Sandoclean PC (wetting agent, anionic)	2 mL/L Tanatex Geo (mineral stabiliser for H_2O_2 stabilisation)	
BR = 1: 20	BR* = 1 : 20	BR = 1 : 100
*p*H 10.9	*p*H 10.7	*p*H 2
t = 30 minutes	*t* = 30 minutes	*t* = 30 minutes
T = 60°C	*T* = 98°C	*T* = 22°C

*BR = bath ratio

Table 2

SAMPLES NOTATIONS	
Sample	**Treatment**
CV	Pre-treated viscose fibres: alkaline washed, bleached, demineralized
CH	Chitosan
TMC	N,N,N tri-methylated chitosan
CV-CH	Viscose fibres treated by 0.5% CH acidic solution
CV-TMC	Viscose fibres treated by 0.5% TMC acidic solution

any sticking to the electrode and jamming with the stirrer, the fibres were kept in a stainless-steel tea container. Prior to the titrations, the ionic strength was set at 0.1 M, by adding pure solid KCl (Kemika, Zagreb). The ionic strength therefore remained within 2% of the initial value upon the additions of HCl and KOH. The blank HCl–KOH titrations were performed under the same conditions as above.

The equilibrium criteria for the timed addition was set at *dE/dt* = 0.1 mV/minutes. The total amount of weak acidic groups was calculated from the difference (D*V*) in the added KOH volume between the fibre sample (V_t) and the blank (V_t, Blank), and any given *p*H.

The molar concentration *Q* relating to the overall charge of the weak ions was calculated from the charge- balance equation:

$$Q = \sum_i c_i z_i = [OH^-] - [H^+] + [Cl^-] - [K^+] \qquad (1)$$

where the square brackets and c_i denote molar concentrations of ionic species, and z_i is the charge number of the species *i*. For a detailed description of the charge calculation, see Čakara et al. [28]. All the reported values are the mean values of triplicate determinations.

Spectrophotometric C.I. Acid Orange 7 method

The adsorption of dye C.I. Acid Orange 7 (purified by recrystallization) into the reference and functionalized viscose fibres coated with both-chitosan acidic solutions, was evaluated by determining the dye concentration in the solutions (*p*H = 3.6 ± 0.5) in contact with the fibres. This was done by using on-line optical absorbance measurement, at a wavelength of maximum absorbance (484 nm). Equilibrium was reached during the 6th hours. The solutions were kept in a thermostat at (25 ± 0.5)°C and stirred with a magnetic stirrer. A Cary 50 Conc computer-controlled UV spectrometer from Varian was used for this experiment. For this adsorption kinetics experiment, 0.25 g of viscose fibre was stirred with an acidic dye solution of 4×10^{-4} M.

Kjeldahl analysis

About 1.5 g of the sample was digested with H_2S0_4 and a catalyst containing 2.8% TiO2, 3.0% $CuS0_4$ $5H_2O$, and 94.2 % K_2SO_4. The residue was treated with NaOH to liberate NH_3, which was subsequently absorbed in boric acid and titrated with HCl. All the samples were analysed, in at least triplicate, to ensure reproducibility and to exclude statistical errors. The total amount of nitrogen was calculated in mmol/kg.

Antimicrobial test

The antimicrobial properties of the functionalized fibres were evaluated by modified ASTM E2149-01 (Standard Test Method for Determining the Antimicrobial Activity of Antimicrobial Agents under Dynamic Contact Conditions), which is a quantitative antimicrobial test method performed under dynamic-contact conditions [30]. Gram-positive and Gram-negative bacteria, as well as a fungus, were used as test organisms. The antimicrobial activity was expressed as *R* = the % reduction of the organism after contact with the test specimen, compared to the number of bacterial cells surviving after contact with the control.

RESULTS AND DISCUSSIONS

Amino group content

Figure 1 shows charging isotherms vs. *p*H for those fibres functionalised with chitosan and those functionalised by TMC, respectively. From figure 1 the amount of amino groups was calculated. The variation coefficient for the potentiometer results was within the region of 2–5%.

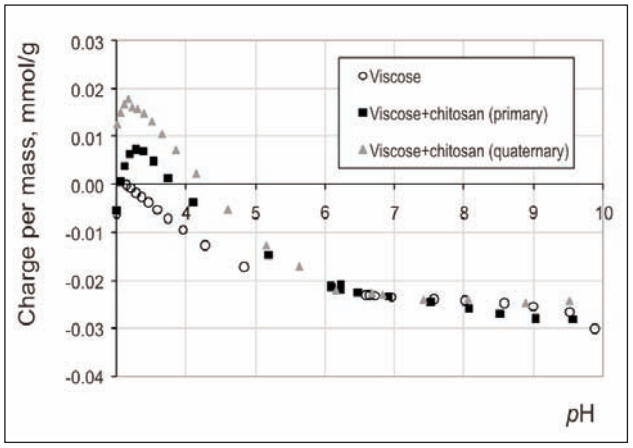

Fig. 1. Fibre-charging isotherms versus *p*H

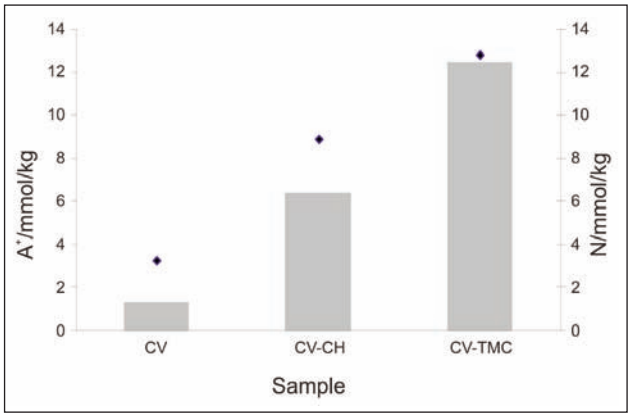

Fig. 2. Amino groups' amounts for the non-functionalized and the both- functionalized fibres determined by Acid Orange and Kjeldahl technique, respectively

In pre-treated cellulose fibres, only a negative-charge was detected (24,2 mmol/kg), which was due to those deprotonated carboxyl groups originating from the glucuronic acid of a cellulose or from oxycellulose, respectively [28]. In regard to fibre functionalization with TMC, significantly higher proportions of available amino groups were detected in comparison with those fibres functionalised by chitosan (CV-CH). The difference in amino group content was 8.2 mmol/kg of fibres which means that, in the case of fibre functionalization by the adsorption of TMC, around 120% higher amino group content was introduced into the material. Taking into account that only primary amino groups were analysed by potentiometric titration and that, in the case of TMC solution only 20% of those for the commercial chitosan solution were available, it is obvious that TMC adsorbed much better onto the fibres. This was also evident from the charge-reversal values (PZC) that, in the cases of the CV-TMC fibres had shifted much more to the right-hand side; which meant higher amounts of amino groups. However, charge-reversal occurred, in both cases, of around *p*H = 4 indicating an excess of deprotonated carboxyl groups within the fibres.

The results of the -COOH group content (see plateau level of anionic charge) for all the investigated samples clearly suggested that the adsorption of chitosan onto the fibres was dominated by physical and hydrophobic interactions. This was in accordance with previous publications [28, 31].

Figure 2 shows the results obtained using the CI Acid Orange 7 method and by Kjeldahl technique, respectively.

The obtained C.I. Acid Orange 7 results were in accordance with the potentiometric titration results, and further revealed that the adsorption of chitosan-quarternary ammonium salt (TMC) onto fibres is more efficient than the adsorption of fibres by chitosan. The amino groups' amount for those viscose fibres functionalised by TMC was 12.45 mmol/kg, whilst for those fibres functionalised by chitosan it was approx. half of the CV-TMC value. The referenced viscose fibres (pre-treated) had 1.3 mmol/kg of amino groups, which was caused by an insignificant quantity of C.I. Acid Orange 7 dye attached to the fibres due to possible hydrogen bonds or Van der Waals forces, at the beginning of experiment. This phenomenon had already been recognized in some of the previous similar experiments [19, 32]. Chitosan adsorption and introduced amino groups were also detected using conventional Kjeldahl technique. When the fibres were treated with chitosan as well as its derivatives, a higher amount of nitrogen was determined in comparison with the non-treated sample; i.e. this confirmed that chitosan was adsorbed on all those fibre surfaces treated with chitosan and its derivatives TMC. The nitrogen in the referenced fibres was obviously present due to impurities in the structure-owing nitrogen (e.g. proteins). However, it seems that these impurities were absent as dissociable functional groups (protonated amino groups). CV-CH possessed 8.9 mmol/kg of nitrogen, whilst CV-TMC around a 45% higher value, which clearly showed that the treatment of fibres by quarternized chitosan (TMC) increased the adsorption abilities of the fibres and, hence, were in agreement with the potentiometer titration and C.I. Acid Orange 7 methods' results. Moreover, all the analytical methods used during this research produced comparable trends for all the presented amino groups onto the functionalized viscose fibres and express good correlation. The obtained small deviations in the results are explained by the different natures of the techniques.

Antimicrobial activity

The results of physicochemical characterization for both-functionalized fibres clearly showed that NNN, tri-methylated chitosan had a higher affinity for adsorption onto fibres, thus these functionalized fibres possessed higher amino group contents. It is known that the amount of positive surface-charge for a material (due to protonated amino groups), affects the level of antimicrobial activity. Moreover, in those fibres functionalised by TMC, quaternary ammonium groups are presented, in addition to the primary amino groups. Polyelectrolyte titration proved (not shown here) the fact that these groups were protonated within a broad *p*H range where commercial

Table 3

PATHOGEN BACTERIA AND FUNGI REDUCTION FOR ANTIMICROBIAL-TREATED VISCOSE FIBRES				
Reduction, %				
Sample	**Pathogen bacteria**			**Pathogen fungi**
	Staphylococcus aureus	*Escherichia coli*	*Streptococcus agalactiae*	*Candida albicans*
CV	23	66	36	29
CV-CH	92	53	26	18
CV-TMC	87	80	100	92

chitosan appears to be insufficient, since in an alkaline medium (*p*H greater than 7) all the amino groups were deprotonated. Most of skin and mucosa irritation, and infection causes *p*H increase; thus when healing using these kinds of medical textiles, antimicrobial activity within a broad *p*H range is essential. The microbiological testing was performed in order to examine the impact of the amino group's amount (as a result of chitosan binding onto the fibres) on the antimicrobial properties of the functionalized viscose fibres. The viscose fibres, before and after functionalization with chitosan and its derivatives TMC, were tested by using the dynamically-shaken test, according to the ASTM E 2149-01 standard (applied for textiles). The results for the reduction (*R*, %) of the pathogenic bacteria Staphylococcus aureus, Escherichia coli, Streptococcus agalactiae and Candida albicans fungi regarding ready-made materials, are given in table 3.

It is estimated that the effectiveness of antimicrobial agents in accordance with the selected standard can be confirmed in those cases where the reduction in microorganisms is more than 75%. The results showed that raw viscose fibres did not fulfil the conditions for antimicrobial effectiveness, because the reductions of all epathogens were less than 75%. Those viscose fibres functionalized with commercial chitosan (primary amino groups) showed effective reduction against pathogenic bacteria (Staphylococcus aureus), but were ineffective against the Escherichia coli (53%), Streptococcus agalactiae (26%), and pathogenic fungus Candida albicans (18%), respectively.

Viscose fibres' functionalization with quarternized chitosan showed intense inhibition for all the tested pathogenic organisms (Staphylococcus aureus, Escherichia coli, Streptococcus agalactiae, and Candida albicans). For the pathogenic fungus Candida albicans, a 92% reduction was detected, and with Streptococcus agalactiae, 100%, respectively.

As hypothesized, the antimicrobial activities were strongly dependent upon the quantity of amino groups and on their protonation as a function of *p*H; i.e. functionalised fibres with a higher content of amino groups display a better overall reduction in pathogenic microorganisms. Thus, CV-TMC showed better antimicrobial activity. In addition, due to the fact that antimicrobial standard tests are required at *p*H = 6,8, it was obvious that around 80% (due to DS value) of the TMC amino groups were cationized and were therefore the driving force for antimicrobial activity within a broad *p*H region.

CONCLUSIONS

From the results it can be concluded that the viscose fibres' functionalization was successful with both the commercial TMC. On the basis of the results it can be concluded that the functionalization of viscose fibres using TMC is more efficient, in contrast to the functionalization of fibres with acidic chitosan solution, in the sense that it contributes more amino groups that more positively influence the growth of pathogen microorganisms. The antimicrobial activities were strongly dependent upon the quantity of amino groups and on their protonation as a function of *p*H; thus it is extremely important to analyse charging behaviour of fibres functionalised by chitosan and its derivates. Our chosen methodology is suitable for analysing the antimicrobial activities of fibres functionalised by amino polysaccharides.

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Specific components of mechatronics systems dedicated to textile and leather equipment and technology*

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REZUMAT – ABSTRACT

Componente specifice ale sistemelor mecatronice destinate echipamentelor şi tehnologiilor din industria de textile şi pielărie. Partea I

Creşterea calităţii producţiei oricărui produs textil sau din piele este influenţată direct de principalele componente ale echipamentelor implicate în tehnologiile specifice sectorului de textile şi pielărie. Elaborarea de soluţii noi în domeniul utilajelor destinate acestui sector şi creşterea calităţii celor existente impun permanente studii teoretice şi experimentale în vederea îmbunătăţirii componentelor lor specifice. Un rol principal în elaborarea unor soluţii constructive moderne revine sistemelor mecatronice implicate în structurile echipamentelor textile şi de pielărie. Lucrarea prezintă unele aspecte privind modele experimentale de sisteme mecatronice destinate controlului unor procese din sectorul de textile şi pielărie şi de echipamente specifice acestora. Studiile au avut ca scop evidenţierea legăturii dintre diferitele tipuri de componente mecatronice, respectiv aplicaţii ale acestora, şi structurile sistemelor tehnice caracteristice industriei de textile şi pielărie, precum şi influenţa lor asupra proceselor specifice acestui sector. Modelele experimentale pot oferi diferite tipuri de control, cum ar fi controlul tensiunii din fir sau al centrării fus + inel, ce reprezintă factori determinanţi în asigurarea calităţii firelor obţinute pe maşini de filat cu inele.

Cuvinte-cheie: echipament textil, sistem mecatronic, senzor, control, tehnologie, textile şi pielărie

Specific components of mechatronic systems dedicated to textile and leather equipment and technology. Part I

The increase of production quality for each textile or leather product is directly influenced by the main equipment components involved in textile and leather technologies. Development of new solutions for the equipment in this field and quality increase of the existing equipment require continuous theoretical and experimental studies in order to improve their specific components. Mechatronic systems involved in the textile and leather equipment structures have a special role in the development of modern cosntructive solutions. The paper presents some aspects concerning experimental models of mechatronic systems dedicated to the control of certain textile and leather processes and their specific equipment. The studies were focused on pointing out the connection between different types of mechatronic components and the technical system structures of the textile and leather industry, as well as their influence on the processes specific to this field. The experimental models can offer different types of control, such as control of yarn tension or ring + spindle centering, as important factors for assuring yarn quality on ring spinning machines.

Key-words: textile equipment, mechatronic system, sensor, control, technology, textile and leather

Development of new generation of textile and leather equipment is based on the integration of mechatronics subsystems structured with precise actuators and equipped with a range of ancillary components (sensors, converters) that offers the possibility to obtain the technological parameters imposed by textile and leather processes [2], [6], [9], [10].

Many textile and leather technological processes served by these machines require high energy consumption and generate environment pollutants. Also, noise and vibration are another major factors of pollution produced by textile and leather equipment. Reducing negative environmental impacts and reduce energy consumption involves rethinking textile machinery active structures using knowledge of mechatronics assembly that allows the development of new process control solutions based on elements consisting of computers, sensors, actuators and specific software that take into functions largely classical mechanical structures. Besides the high degree of reliability, mechatronics solutions allow obtaining flexible working arrangements for large complex equipment that can work in sync, offers possibilities to adapt the work regimes depending on the parameters of raw materials, environmental conditions (temperature, humidity) etc. Development of transmission infrastructure data (Internet, GSM) offers great possibilities for remote monitoring and control of processes taking place in units located anywhere in the world [7], [11], [12].

Operation computerized machinery enables this new configuration as their functions can be monitored and changed remotely. Location of supervision and control elements allow rapid identification of defects and

* **Part I** / ***Partea I***

real-time communication of information to support agencies [8], [13].
From the multitude of hardware that structure control systems of textile machines and processes, the paper presents only issues on modules for converting analog signals into digital signals.

DESIGN CRITERIA FOR CONTROL SOLUTION

The high degree of complexity of textile machinery and the large number of active elements do not allow the development of effective centralized control solutions based synchronous sequential operations performed by a single processor and therefore requires:

- implementation of active nodes consisting of autonomous processors, sensors and actuators specific subprocesses;
- development of standardized communication interfaces so that nodes can communicate with each other asynchronously to adapt local functions depending on the overall behavior;
- development of software structures with tasks distributed independent components which together could achieve on-time schemes for textile machinery and provision of open structures that can be easily adapted to different types of equipment and processes with infinite recombination functions and standardized elements;
- ensuring the interconnection of these local networks of nodes with Internet or GSM network to enable remote operation (monitoring, change the work regimes etc.);
- providing opportunities for unconventional power supply that eliminates the risk of faulty operation when accidental power interruptions;
- providing opportunities for development and upgrading software using certified resources available in cloud systems so that local resources are not required costly to develop and maintain applications. This is very important primarily because of the degree of flexibility of equipment, the utmost is done in software;
- ensuring reliability and accuracy criteria by using software libraries certificated to avoid isolated implementations, non-portable.

Analysis of the elements on which a solution can be developed based on control network active nodes that can perform the above conditions have highlighted the following:

- large number of sensors and actuators involved in textile processes require high performance computers to implement the functions of positioning, fabrication and calibration of process parameters. This requires software features calibrated as the main reason for the choice of processors available on a large scale and for which there are software tools that could develop robust applications;
- ensuring interoperability of nodes implies uniform hardware platform with minimal components that enable modular development with standardized and dedicated elements;
- ensuring compatibility with products from the same category by using development tools with broad spreading, avoiding exotic instruments developed commercially without appropriate licenses;
- ensure optimal communications data network so that nodes can be controlled/upgraded remotely effective.

Alternatives analysis of integrated systems in a single circuit (microcontroller) that can be the central element to such a node revealed the following:

- Development platforms and microcontrollers from Microchip covers a wide range of applications being available processors on 8, 16 and 32 bits. They have countless notes software applications and development tools centered around MPLAB product that can integrate a wide range of programming languages such as Microchip products, Hitec, IAR, CCS etc. A special category in range Microchip processors represent the implementation in nanoWatt XLP technology which function with extremely low power consumption. They have great advantages in mobile applications or those located in inaccessible areas and unconventional energy sources fueled. Adoption of the MIPS architecture for 32-bit architectures provides developers access to a huge stock of tools and software libraries developed in recent years for professional applications. No programming resourced cloud version are available.
- Another analyzed big producer of components for computers and communications is Freescale Semiconductor company (formerly Motorola). It has 8-16-32 bit microcontrollers and a wide range of components such as memory, interface circuits for communications, power converters, sensors etc. Range of 32 bit processors is using primarily PowerPC and ARM processor cores. With a long tradition in production of microprocessors, Freescale provides a complete range of integrated structures for industrial control, automotive, military applications etc. The entire range of microcontrollers has a programming language Codewariorr focused on using C/C++ for application development. As Microchip the products range of this company has improved constantly and included extensions for network functions such as Ethernet Interfaces, USB, Zigbee, Blutoooth etc. Development of new applications is restricted by proprietary solutions.
- Texas Instruments is another producer of integrated computer into a single circuit analysed for selecting an alternative for achieving control nodes of textile machinery. Like most companies, it produces the full range of microcontrollers covering both applications based on lower energy consumption and for processing the signals that computing power is achieved through architectures equipped with hardware structures dedicated

for fast operations (DSP). For the range of 32 bit applications processors Texas Instruments adopted ARM cores that included its own periphery, complete structures are available allowing the development of extremely wide range of applications. There are main difficulties in using these components for prototypes for which is required expensive equipment to achieve very small interconnections.

- ARM processors represent a range of products supplied by the company after a license ARM Ltd. It integrates a 32 bit RISC processor fitted with a standard set of instructions known by the term ISA (Instruction Set Architecture); these structures largely inherited experience with the implementation of range Cray supercomputers based on multiprocessor structures and multitasking operating systems. RISC machines offered since the early emergence lower energy consumption and working facilities in parallel structures. This was possible primarily due to the presence of a set of instructions that are executed in precise and uniform time quanta thus ensuring rigorous switching tasks. Perhaps the greatest achievements in the field of home computers were originally on these machines in most cases designed for military applications. Being developed in open technologies they have led standardization assemblies and anchoring process extremely large number of developers, eliminating the barriers caused by proprietary solutions providers. Such a process obviously redesigned to be integrated into a single circuit was developed by ARM which then comes manufacturing license in most manufacturers of microprocessors (NXP, Philips, Texas Instruments, Intel, Analog Device etc.). Software developers are focused on such architectures with a high degree of ruggedness, minimal power consumption and possibilities for expansion in multicore structures. Most mobile phones and the recent iPhone and iPad1/2 are made with such processors. Web servers, the main equipment that caused massive power consumption because they are constantly fed are redesigned to be implemented with ARM multiprocessor architectures.

EXPERIMENTAL MODEL FOR ANALOG-DIGITAL CONVERTER (ADC)

Figure 1 shows the block diagram of the ADC module which has the following characteristics:

- conversion by successive approximations (SAR);
- conversion speed to 1 Msps;
- external pin for reference voltage;
- four unipolar differential amplifiers Sample/Hold;
- up to four simultaneous sampling analog inputs;
- examination of the channels automatically;
- selectable trigger source for conversion;
- conversion buffer length of 16-word;
- completing selectable modes of buffers;
- four alignment options of the outcome;
- operation during CPU Sleep and Idle modes.

An application of ADC module as main component of mechatronics systems for textile equipment refers to a device that whichever one analog signal from the microcontroller analog input signal level indicates a set of 8 leds; the minimum level will light one led and the maximum level will leds. At an intermediate level will turn a set of lights at the level applied. The levels are determined by reference with level range of control parameter values from sensors of designed mechatronics systems (wire tensions, eccentricities, speed etc.).

Depending on the device variant, ADC module can have up to 13 analog inputs, designated AN0 – AN12. These analog inputs are connected through multiplexers to four amplifiers Sample/Hold, marked by CH0 – CH3. Multimplex devices corresponding to analog inputs include two sets of control bits indicated by CHySA/ChyNA for MUXA and CHySB/ChyNB for MUXB. These control bits selects a particular analog input for conversion. Unipolar differential conversions are possible on all channels using dedicated input pins. Evaluation method (scan) can be enabled channel amplifier CH0 Sample/Hold. Any subset of analog inputs (AN0 – AN12) can be selected by the user. Select pins are converted in ascending order using CH0.

The ADC module supports sampling multiple channels simultaneously using Sample/Hold to sample the entries at the same time as channel-specific conversion is performed sequentially. By default, multiple channels are sequentially sampled and converted.

ADC module is connected to a 16-word buffer length achieved by dual port RAM. Conversion result is available in four different numerical formats. ADC module has seven registers for control and status. They are:

- control registers of conversion operations AD1CON1, AD1CON2, AD1CON3;
- selection registers of pins to be connected to amplifiers Sample/Hold: AD1CHS0 and AD1CHS123;
- register to set the input pins as analog or digital inputs I/O: AD1PCFGL;
- selection register of inputs for sequential examining AD1CSSL.

CONCLUSIONS

Analog-digital conversion module for signals collected from sensors attached to textile equipment in order to ensure control specific processes was designed to be integrated into a complex information processing system as it is defined in mechatronics. Details of the operating regimes ADC and hardware problems/software specific to this module will be published in future issues of this journal.

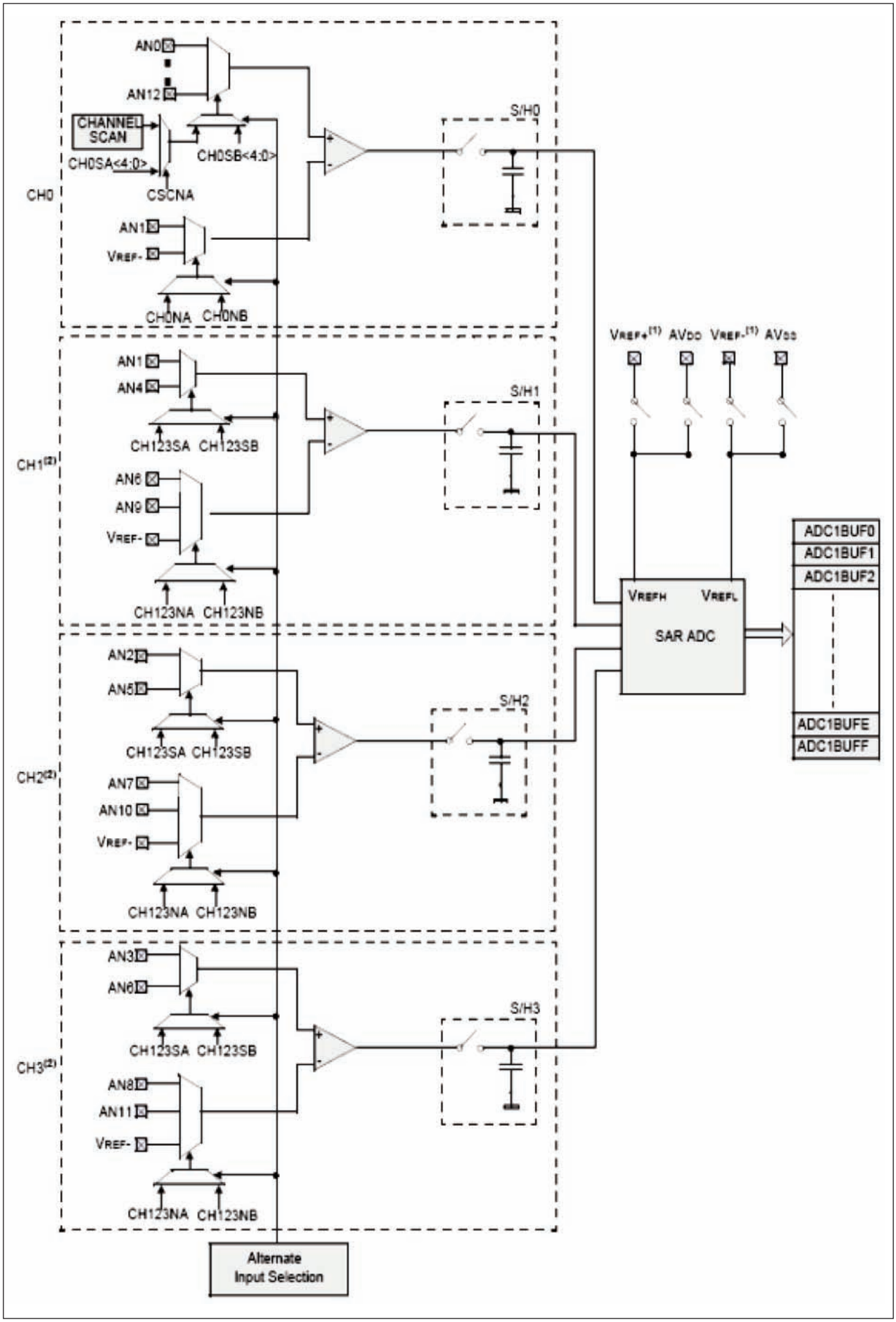

Fig. 1. Internal structure of ADC

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The effects of yarn and fabric structural parameters on the seam slippage, abrasion and pilling properties of double woven upholstery fabrics

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REZUMAT – ABSTRACT

Efectul parametrilor structurali ai firelor şi ţesăturilor duble pentru tapiţerie asupra proprietăţilor de piling, rezistenţă la abraziune şi alunecare

În lucrare au fost studiate, prin analiză de varianţă, proprietăţile ţesăturilor duble destinate tapiţeriei, respectiv rezistenţa la alunecare, rezistenţa la abraziune (Martindale) şi efectul piling, care reprezintă importante caracteristici fizice şi de performanţă ale unor astfel de ţesături. În literatura de specialitate, studiile privind efectele parametrilor structurali ai firelor şi ţesăturilor, cum ar fi compoziţia fibroasă, desimea firelor de bătătură şi tipul de legătură, cu raportare la ţesăturile duble pentru tapiţerie, au fost mai puţin aprofundate. În cadrul experimentelor, s-a observat că, în cazul ţesăturilor duble destinate tapiţeriei, rezistenţa la glisare a firelor se îmbunătăţeşte odată cu creşterea desimii firelor de bătătură şi cu reducerea raportului de legătură. În timp ce rezistenţa la abraziune şi efectul piling ale ţesăturilor duble destinate tapiţeriei ar fi trebuit să manifeste aceleaşi tendinţe ca şi rezistenţa la alunecare, datorită legăturii satin aceste proprietăţi nu sunt influenţate în mod obişnuit de setarea bătăturii. În cazul ţesăturilor duble pentru tapiţerie, cu bătătură din fire scurte de poliester, rezistenţa la alunecare a firelor în urzeală şi bătătură şi rezistenţa la abraziune au înregistrat valori mai mari decât în cazul ţesăturilor cu bătătură din fire de bumbac. La acelaşi tip de ţesături, s-a observat un comportament invers în ceea ce priveşte efectul piling.

Cuvinte-cheie: ţesături duble, tapiţerie, aluncecare, rezistenţă la abraziune, piling, proprietăţi fizice

The effects of yarn and fabric structural parameters on the seam slippage, abrasion and pilling properties of double woven upholstery fabrics

In this work, seam slippage, Martindale abrasion and pilling properties of the double woven upholstery fabrics, which are important physical and performance characteristics for such fabrics, have been investigated by analysis of variance. The effects of yarn and fabric structural parameters such as weft densities, face weave patterns and raw materials of weft yarns, which have been analyzed and reported in this work for double upholstery woven fabrics, were not studied in the references. It has been observed that the seam slippage strength of upholstery double fabrics has increased by increasing weft density and by decreasing sateen number of face weave. While the abrasion resistance and pilling degree of upholstery double fabrics have showed similar tendencies with seam slippage strength of those by the effect of sateen number of face weave, these properties have not been affected regularly by weft setting. The seam slippage strength along both warp and weft directions and the abrasion resistance of upholstery double fabrics woven with wefts of staple polyester yarn have been higher than those of upholstery double fabrics woven with cotton wefts. The opposite has been observed for the pilling degree of upholstery double fabrics.

Key-words: double fabrics, woven upholstery, seam slippage strength, abrasion behavior, pilling, physical properties

It is required by consumers that upholstery fabrics look aesthetical and are enduring. In this context, seam slippage strength, abrasion resistance and fabric propensity to fuzzing and to pilling are important properties for upholstery fabrics. Seam slippage is an openness, which is formed by slipping yarns that are in or adjacent to stitch line and a partial or complete entanglement of stitch integrity. It is observed at fabrics, woven with sliding yarn, and fabrics in which there are few interlacing. Abrasion is a decrease in the length of eaves and roughness, or a tear, slimming as a result of decline of its thickness and volume. Fibers relocate progressively during abrasion. Abrasion resistance is an endurance of fabrics surface against abrading effects under certain circumstances. On the other hand pilling is a fabric surface fault, which is observed as fiber groups or round mass of mixed and fuzzy fibers, attached to fabric surface with one or more fibers. Upholstery fabrics are rubbed against clothing or sheet fabrics, namely they are subjected to friction of fabric to fabric [1].

There are studies, which investigated the seam opening behavior of woven fabrics: Galuszynski [2] described the relationship among parameters which influence the seam opening, and claimed that the resistance to seam opening increase with increasing yarn-to-yarn friction, contact angle between threads (fabric geometry), the number of weft yarn in fabric, stitch density and yarn flexural rigidity. Gürarda [3] investigated the effects of weft density, weave type and sewing thread on the seam performance of plain and twill fabrics. She found that twill fabrics had much tendency for seam slippage than plain fabrics. And also seam slippage strength increase as weft density increased. Yıldırım [4] made a non-linear regression mathematical model to predict the seam opening

properties of a kind of sateen fabric with in two different seam opening standard test methods. He found that seam opening of the seat upholstery under both types of test conditions was seriously affected by weft density.

Furthermore, a number of studies were carried to search the abrasion property of woven fabrics: Çeven [5] investigated comparatively the effect on abrasion resistance of the presence of chenille and flock yarns, as weft yarn in the structure of woven fabrics. Fabrics with high weft density and low float length have less mass loss values after abrasion test. The fabrics manufactured with flock yarns have higher abrasion resistance than fabrics manufactured with chenille yarns. Kalaoğlu and Demir [6] investigated the chenille yarn parameters on the abrasion resistance and seam slippage of upholstery fabrics. While raw material and twist of chenille yarn affected abrasion resistance, weave affected seam slippage. Ülkü, Örtlek and Ömeroğlu [7] found that pile length and twist rate of chenille yarns, weave had influence on the abrasion resistance of upholstery fabrics. Özdemir and Çeven [8] observed that there was an improvement in abrasion resistance of chenille yarn and upholstery fabric with increasing twist, pile length, and the use of natural fibers as pile materials. Babaarslan and İlhan [9] found that the mass loss rate (%) tended to increase as pile length of chenille yarn increased. Castellar, Saurí, Martí and Manich [10] measured the abrasion behavior of thirty-nine woven fabrics made of wool, polyester and cellulosic (viscose and flax) fiber blends using the Martindale wear and abrasion tester. They found that firstly abrasion kinetics depended on fabric sett, interlacing weave and composition, secondly the most relevant fabric parameters related to the abrasion resistance were fabric composition, thickness and the weave interlacing coefficient. Pamuk and Çeken [11] researched the Martindale Abrasion properties of seven types of commonly used automotive seat cover fabrics. They found a significant difference between the fabric types both in the rate of weight and thickness. On the other hand, fabrics made from acrylic, wool and viscose yarns had less abrasion resistance than those made from polyester yarns. Kaynak and Topalbekiroğlu [12] investigated the abrasion resistance properties of seven types of woven fabrics. They observed that the weave pattern had a significant effect on the abrasion resistance property of woven fabrics. Tukey test results showed that abrasion resistance of weave types with a high number of floats and low number of interlacing was low.

The studies in literature focused on single layer woven fabrics. The aim of this study was to determine the effects of selected yarn and fabric structural parameters on the seam slippage strength, abrasion resistance and propensity to surface fuzzing and to pilling of certain double woven upholstery fabrics. In this regard, an experimental study has been carried out, the effects of the parameters have been detected firstly by graphics formed by obtained data and secondly by analysis of variance [13].

EXPERIMENTAL PART

Certain double woven upholstery fabrics planned in this study have been produced, after the seam slippage strength and abrasion resistance of these fabrics have been measured and their propensity to surface fuzzing and to pilling have been determined.

In this research 36 kinds of self-stitched double upholstery woven fabric samples, whose face weave pattern have been 5s sateen, 10s sateen, 20s sateen and back weave has been 5s sateen, have been produced in Mega Textile Industry and Trade Inc. by Dornier machine with rapier picking mechanism. Weave patterns are shown in figure 1. 150 denier of filament polyester yarn with twist of 160 tpm in *S* direction and of filament rayon yarn with twist of 500 tpm in *S* direction have been used as warp yarns. In addition to this, Ne 30/2 cotton yarn with twist of 710 tpm in *S* direction and Ne 30/2 staple fiber PES yarn with twist of 600 tpm in *S* direction have been planned as weft yarns. Warp settings of fabric samples have been 66 cm^{-1}, weft settings of fabric samples have been 32, 35 and 38 cm^{-1}.

Cotton and staple fiber polyester have been used as raw material of weft yarns so fabric samples have been produced in raw color namely ecru. Fabric samples have been applied hard finishing process and passed from RAM, which have 8 sections at the velocity of 25 m/minutes.

Fabric samples have been coded according to weft density, raw material of warp yarns and of weft yarns, face weave pattern as in table 1. The numbers in fabric codes represent weft densities, raw materials of warp yarns and of weft yarns, face weave patterns respectively. These are square unit weaves, so the number of each warp and weft yarn interlacing is equal to each other, namely the average yarn interlacing is equal to number of yarn interlacing. And also the average float length of warp yarn is equal to the average float length of weft yarn. The average float length *F* has been calculated according to Ashenhurst [14] by equation (1):

$$F_{1/2} = \frac{R_{2/1}}{t_{1/2}} \quad (1)$$

where:

$R_{2/1}$ is the weft (*2*) or warp (*1*) repeat;

$t_{2/1}$ – the number of warp or weft intersections in the weave repeat.

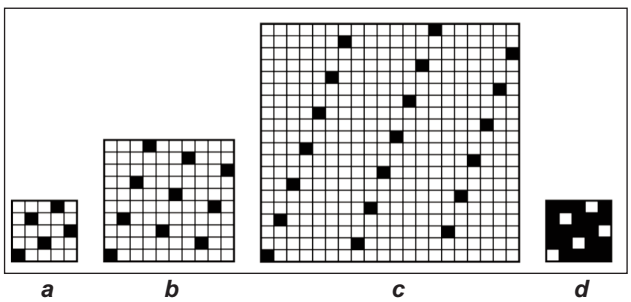

Fig. 1. Weave patterns: ***a*** – 5s sateen; ***b*** – 10s sateen; ***c*** – 20s sateen; ***d*** – 5s sateen

Table 1

THE SPECIFICATIONS OF SAMPLE FABRICS								
Fabric number	**Fabric code**	**Weft density**	**Raw material of warp yarns**	**Raw material of weft yarns**	**Face weave pattern**	**The weave interlacing coefficient**	**The average float length**	**Weight of unit area, g/m²**
1	1111	32	Polyester	Cotton	5s sateen	0,4	2,5	286,2
2	1112				10s sateen	0,2	5	279,5
3	1113				20s sateen	0,1	10	277,4
4	2111	35			5s sateen	0,4	2,5	302,1
5	2112				10s sateen	0,2	5	295,6
6	2113				20s sateen	0,1	10	293,9
7	3111	38			5s sateen	0,4	2,5	316,1
8	3112				10s sateen	0,2	5	312,0
9	3113				20s sateen	0,1	10	308,6
10	1121	32		Staple fiber polyester	5s sateen	0,4	2,5	281,7
11	1122				10s sateen	0,2	5	278,9
12	1123				20s sateen	0,1	10	279,6
13	2121	35			5s sateen	0,4	2,5	299,0
14	2122				10s sateen	0,2	5	292,2
15	2123				20s sateen	0,1	10	291,0
16	3121	38			5s sateen	0,4	2,5	311,6
17	3122				10s sateen	0,2	5	310,2
18	3123				20s sateen	0,1	10	306,0
19	1211	32	Rayon	Cotton	5s sateen	0,4	2,5	275,2
20	1212				10s sateen	0,2	5	267,7
21	1213				20s sateen	0,1	10	267,2
22	2211	35			5s sateen	0,4	2,5	288,9
23	2212				10s sateen	0,2	5	283,4
24	2213				20s sateen	0,1	10	277,8
25	3211	38			5s sateen	0,4	2,5	304,9
26	3212				10s sateen	0,2	5	297,9
27	3213				20s sateen	0,1	10	295,2
28	1221	32		Staple fiber polyester	5s sateen	0,4	2,5	268,0
29	1222				10s sateen	0,2	5	264,4
30	1223				20s sateen	0,1	10	263,9
31	2221	35			5s sateen	0,4	2,5	281,4
32	2222				10s sateen	0,2	5	277,2
33	2223				20s sateen	0,1	10	277,6
34	3221	38			5s sateen	0,4	2,5	299,8
35	3222				10s sateen	0,2	5	292,8
36	3223				20s sateen	0,1	10	290,4

Subscripts *1* and *2* are used throughout to denote warp and weft respectively. The other fabric property, the weave interlacing coefficient, defined by Galcerán [15] has been calculated by equation (2):

$$KL = \frac{i}{w_1 \cdot w_2} \quad (2)$$

where:

i is the number of interlacing points in weave repeat;

w_1 – the number of ends in weave repeat;

w_2 – the number of picks in weave repeat.

Seam slippage strength, abrasion resistance and pilling tests have been conducted on the fabrics according to the features and methods of TS11818 EN 14465 Textile Upholstery Fabrics in Physical Testing Laboratory of in-house. The fabric samples have been conditioned at standard atmosphere conditions (20 ± 2ºC, 65 ± 2% relative humidity) for 24 hours.

Tests performed

The seam slippage strength of the upholstery fabrics under static loading has been determined according to TS EN ISO 13936-1 using Multipurpose Strength Tester, Instron 4411. The values of seam slippage strength have been inferenced from force-distance graphics according to TS EN ISO 13936-1.

Nu-Martindale Abrasion Tester (James H Heal, UK) has been used to evaluate the mass loss of upholstery fabrics after abrasion in compliance with the TS EN ISO 12947-2 standards. The fabric samples have been abraded under the pressure of 12 KPa (795 ± 7 g). The weight of upholstery fabrics have been measured by a digital balance at the end of 10000 abrasion cycles, and percentages of the mass loss of upholstery fabrics have been calculated. Propensity to Surface Fuzzing and to Pilling of Upholstery Woven Fabrics has been determined according to TS EN ISO 12945-2 standards by Nu-Martindale Abrasion Tester. The fabric samples have been compared with standard photographs subjectively by 3 persons at the Physical Testing Laboratory after 2000 abrasion cycles. The evaluations have been shown as numbers and letters in compliance with TSE 11818 EN 14465 standards. While 5 and A indicate the lowest propensity to surface fuzzing and to pilling, 1 and D indicate the highest propensity to those.

RESULTS AND DISCUSSIONS

The works conducted in this scope of the study have been investigated in two separate categories: upholstery fabrics woven with polyester warps and with rayon warps. Results experimentally obtained for upholstery fabric samples have been evaluated graphically and then statistically by Multivariate Analysis of Variance (MANOVA) according the General Linear Model with SPSS 15.0 Statistical Software. Significance degrees (p), which have been obtained from MANOVA, have been compared with significance level (α) of 0.05. The effects, whose significance degrees have been lower than 0.05, have been interpreted as statistically important.

Seam slippage strength test results

It is observed in figure 2 that seam slippage strength along both warp and weft directions has increased in agreement with weft density of upholstery fabrics woven with cotton wefts. Seam slippage strength along warp direction of upholstery fabrics woven with wefts of staple polyester yarn has increased with weft density; however there is no regular change in seam slippage strength along weft direction. On the other hand, it is seen that if sateen number of face weave pattern increases, seam slippage strengths along both warp and weft directions will decrease. It is because that if the sateen number increases, the weave unit will enlarge, so the weave interlacing coefficient will decrease, as seen on table 1, namely the friction between the intersecting yarns will decrease, consequently, it will get easier for the yarns to slip due to the forces perpendicular to their axis. Seam slippage strength along both warp and weft directions of upholstery fabrics woven with cotton wefts has been higher than those of samples woven with wefts of staple polyester yarn. It probably because that the frictional forces between cotton wefts and polyester warps have been higher than those between polyester wefts and polyester warps. According to Galuszynski [16], yarn to yarn friction plays an important role in seam slippage resistance. He found that the frictional force between cotton yarns was higher than that between polyester yarns. On the other hand, seam slippage strengths of upholstery fabrics along warp direction have been higher than seam slippage strengths of those along weft direction. This probably results from the fact the warp density of upholstery fabrics is higher than weft density of those.

Results of MANOVA, which has been performed for seam slippage strength of fabrics woven with polyester warps along warp and weft directions, are shown in table 2 and table 3. It can be concluded that the effects of all parameters on seam slippage strength of fabrics woven with polyester warps along weft direction are statistically important, whereas the effects of raw material of weft yarns and face weave pattern on seam slippage strength of fabrics woven with polyester warps along warp direction are statistically important at the significance level of 0.05.

While seam slippage strength of upholstery fabrics woven with cotton wefts along weft direction has increased in accordance with weft density, there is no regular change in seam slippage strength along warp direction can be seen in figure 3. It is also observed that seam slippage strength of upholstery fabrics woven with weft of staple polyester yarn along both warp and weft directions has increased in accordance with weft density. Besides, if sateen number of face weave pattern enlarges, namely the weave interlacing coefficient decrease, seam slippage strength along weft direction will decrease. The reason explained for upholstery fabrics woven with polyester warps is valid here also. However, there is no regular change in seam slippage strength along warp direction. Seam slippage strengths along both warp and weft directions

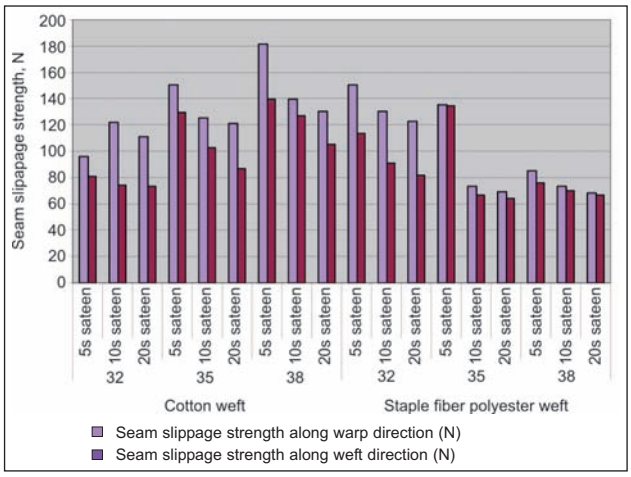

Fig. 2. Seam slippage strengths of fabric samples woven with polyester warps

of upholstery fabrics woven with cotton wefts have been higher than those of samples woven with wefts of staple polyester yarn. It probably because that the frictional forces between cotton wefts and rayon warps have been higher than those between polyester wefts and rayon warps. Galuszynski [16] found that the frictional force between cotton yarns was higher than that between polyester yarns. On the other hand, seam slippage strengths of upholstery fabrics along warp direction have been higher than seam slippage strengths of those along weft direction. This probably results from the fact the warp density of upholstery fabrics is higher than weft density of those. There has been no regular effect of raw material of weft yarns on the seam slippage strength of upholstery fabrics along both warp and weft directions.

Results of MANOVA, which has been performed for seam slippage strength of fabrics woven with rayon warps along warp and weft directions, are shown in table 4 and table 5. According to the results of MANOVA, weft density, raw materials of weft yarns and face weave pattern have affected seam slippage strength of fabrics woven with rayon warps along both warp and weft directions statistically ($\alpha = 0.05$).

Martindale abrasion test results

Mass loss after 10 000 abrasion cycles of fabric samples woven with polyester warps are shown in figure 4. The upholstery fabric woven with cotton wefts, with a weft density of 38 cm^{-1} and face weave pattern as 5s sateen, lost the lowest amount of mass, thus has the highest abrasion resistance. On the other hand, the upholstery fabric woven with cotton wefts, with a weft density of 35 cm^{-1} and face weave pattern as 20s sateen, lost the highest amount of mass, namely has the lowest abrasion resistance.

The results presented in figure 4 show that there is no regular effect of weft density on the mass loss of upholstery fabrics woven with polyester warps. The mass loss of upholstery fabrics woven with cotton wefts increased in agreement with sateen number of face weave pattern. This can be explained by the fact that if the unit size of sateen weaves increase, the average float length will increase. Thus, the fabric surface exposed to abrasion will increase. Besides, the weave interlacing coefficient, namely the density of yarn interlacing and connection with cross yarns, will decrease. So the fabric structure will loose and then abrasion resistance will decrease. However, here has been no regular change in the mass loss of upholstery fabrics woven with wefts of staple polyester yarn. On the other hand, mass losses of upholstery fabrics, except for whose face weave patterns have been 5s sateen, woven with wefts of staple polyester yarn have been lower than mass losses of upholstery fabrics woven with cotton wefts. This is due to the fact that while polyester fiber has higher abrasion resistance value, 249.21; cotton fiber has a middle-level abrasion resistance value, 32.5 [17]. Results of MANOVA, which has been performed for the mass loss of fabrics woven with polyester warps,

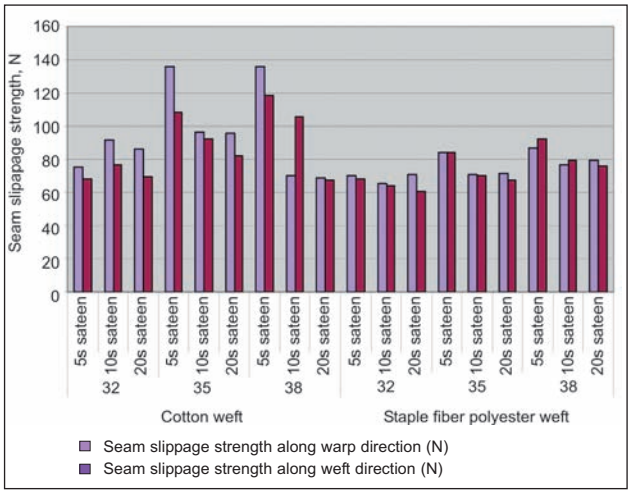

Fig. 3. Seam slippage strengths of fabric samples woven with rayon warps

Table 2

RESULTS OF MANOVA FOR SEAM SLIPPAGE STRENGTH OF FABRICS WOVEN WITH POLYESTER WARPS ALONG WARP DIRECTION

Source of variance	*F*	*p*
Weft setting	2.813	0.067
Raw material of weft yarns	64.109	0.000
Face weave pattern	22.063	0.000

Table 3

RESULTS OF MANOVA FOR SEAM SLIPPAGE STRENGTH OF FABRICS WOVEN WITH POLYESTER WARPS ALONG WEFT DIRECTION

Source of variance	*F*	*p*
Weft setting	42.398	0.000
Raw material of weft yarns	215.024	0.000
Face weave pattern	271.728	0.000

Table 4

RESULTS OF MANOVA FOR SEAM SLIPPAGE STRENGTH OF FABRICS WOVEN WITH RAYON WARPS ALONG WARP DIRECTION

Source of variance	*F*	*p*
Weft setting	26.123	0.000
Raw material of weft yarns	119.780	0.000
Face weave pattern	47.788	0.000

Table 5

RESULTS OF MANOVA FOR SEAM SLIPPAGE STRENGTH OF FABRICS WOVEN WITH RAYON WARPS ALONG WEFT DIRECTION

Source of variance	*F*	*p*
Weft setting	117.876	0.000
Raw material of weft yarns	134.935	0.000
Face weave pattern	83.886	0.000

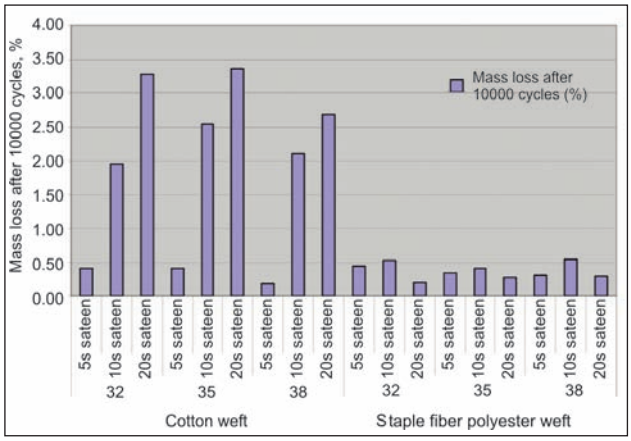

Fig. 4. Mass losses of fabric samples woven with polyester warps

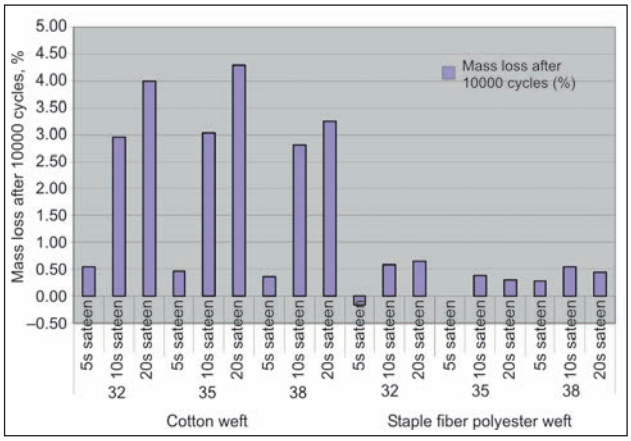

Fig. 5. Mass losses of fabric samples woven with rayon warps

Table 6

RESULTS OF MANOVA FOR MASS LOSS OF FABRICS WOVEN WITH POLYESTER WARPS		
Source of variance	***F***	***p***
Weft setting	4.145	0.020
Raw material of weft yarns	711.044	0.000
Face weave pattern	194136	0.000

Table 7

RESULTS OF MANOVA FOR MASS LOSS OF FABRICS WOVEN WITH RAYON WARPS		
Source of variance	***F***	***p***
Weft setting	0.687	0.506
Raw material of weft yarns	346.848	0.000
Face weave pattern	2.971	0.058

are shown in table 6. It can be concluded that the effects of weft density, raw material of weft yarns and face weave pattern on the mass loss of fabrics woven with polyester warps are statistically important at the significance level of 0.05.

Mass loss after 10 000 abrasion cycles of fabric samples woven with rayon warps are shown in figure 5. The upholstery fabrics woven with wefts of staple polyester yarn, with weft densities of 32 cm^{-1} and 35 cm^{-1} and face weave pattern as 5s sateen, get mass. This probably results from the fibers on the fabric surface, which are detached from abrader fabric due to wefts of staple polyester yarn. On the other hand, the upholstery fabric woven with cotton wefts, with a weft density of 35 cm^{-1} and face weave pattern as 20s sateen, lost the highest amount of mass, namely has the lowest abrasion resistance, as for the upholstery fabric woven with polyester warps. It can be explained by the facts that firstly average float length of 20s sateen weave is the highest, so yarns are abraded easily as explained for upholstery fabrics woven with polyester warps, secondly abrasion resistance value of cotton fiber is lower than that of polyester fiber [17].

It is observed in figure 5 that there is no regular effect of weft density on the mass loss of upholstery fabrics woven with rayon warps. The mass loss of upholstery fabrics woven with cotton wefts increased in agreement with sateen number of face weave pattern. The reason explained for upholstery fabrics woven with polyester warps is valid here also. However, there is no regular change in the mass loss of upholstery fabrics woven with wefts of staple polyester yarn. This can be explained by the fact that fabric surface exposed to abrasion increases with average float length, so abrasion resistance will decrease. On the other hand, mass losses of upholstery fabrics woven with wefts of staple polyester yarn have been lower than mass losses of upholstery fabrics woven with cotton wefts. The reason explained for upholstery fabrics woven with polyester warps is valid here also. Results of MANOVA, which has been performed for the mass loss of fabrics woven with rayon warps (table 7). According to the results of MANOVA, only raw materials of weft yarns has affected the mass loss of fabrics woven with rayon warps statistically ($\alpha = 0.05$).

Fabric propensity to surface fuzzing and to pilling

The results given in table 8 indicate that there is no regular effect of weft density on the propensity to surface fuzzing and to pilling of upholstery fabrics. However, the propensity to surface fuzzing and to pilling of upholstery fabrics increased in agreement with sateen number of face weave pattern. This is probably because of the facts that firstly fabric surface exposed to abrasion increases with average float length, secondly the increase in the average float length of weft yarns results in the decrease of number of crossover points with the warp yarns, namely the weave interlacing coefficient will decrease. As a result of these, fabric structure will loose so it gets easy to make fuzzy and form pills. It is also seen that the propensity to surface fuzzing and to pilling of upholstery fabrics woven with rayon warps have been

Table 8

RESULTS OF MEASUREMENTS OF SAMPLE FABRICS PROPENSITY TO SURFACE FUZZING AND TO PILLING

Fabric code	Degree of pilling after 2000 cycle	Performance level*	Fabric code	Degree of pilling after 2000 cycle	Performance level*
1111	4 - 5	A	1211	4 - 5	A
1112	4	B	1212	2 - 3	-
1113	2	-	1213	2	-
2111	4	B	2211	4 - 5	A
2112	3	D	2212	3	D
2113	2	-	2213	2 - 3	-
3111	4 - 5	A	3211	4 - 5	A
3112	3 - 4	C	3212	3	D
3113	1 - 2	-	3213	2 - 3	-
1121	3	D	1221	4 - 5	A
1122	2 - 3	-	1222	3	D
1123	1 - 2	-	1223	2 - 3	-
2121	3	D	2221	4 - 5	A
2122	2 - 3	-	2222	4	B
2123	1 - 2	-	2223	2 - 3	-
3121	3 - 4	C	3221	4 - 5	A
3122	2 - 3	-	3222	4	B
3123	1 - 2	-	3223	3 - 4	C

*According to TS 11818 EN 14465 standard

higher than those of upholstery fabrics woven with polyester warps. This results from the fact that while the twist of rayon warps is 500 tpm; twist of polyester warps is 160 tpm. The propensity to surface fuzzing and to pilling of upholstery fabrics woven with rayon warps and wefts of staple polyester yarn have been lower than those of upholstery fabrics woven with rayon warps and cotton wefts. This is due to the fact that the abrasion resistance value of polyester fiber is higher than that of cotton fiber [17]. On the other hand, the propensity to surface fuzzing and to pilling of upholstery fabrics woven with polyester warps and wefts of staple polyester yarn have been higher than those of upholstery fabrics woven with polyester warps and cotton wefts. This is due to the fact that during pilling polyester fibers hold on to fabric tightly, while cotton fibers detach from the fabric surface by breaking off after a certain time.

CONCLUSIONS

Statistical and experimental studies have been performed within the scope of this study to determine the effects of yarn and fabric structural parameters on the seam slippage strength, abrasion resistance and propensity to surface fuzzing and to pilling of double woven upholstery fabrics. The important results obtained with these analyses are summarized below:

- According to the results of MANOVA, weft setting, raw material of weft yarns and face weave patterns have been found to have significant effects on the seam slippage strength, abrasion resistance and propensity to surface fuzzing and to pilling of double woven upholstery fabrics, except for the effect of weft density on the seam slippage strength of fabrics woven with polyester warps along warp direction and abrasion resistance of fabrics woven with rayon warps.
- As expected, seam slippage strength along both warp and weft directions has increased in agreement with weft density of two types of fabrics namely; upholstery double fabrics woven with polyester warps and cotton wefts and upholstery double fabrics woven with rayon warps and wefts of staple polyester yarn.
- It has been observed that seam slippage strength of upholstery double fabrics woven with polyester warps along both warp and weft directions has decreased, whereas seam slippage strength of upholstery double fabrics woven with rayon warps only along weft direction has decreased, by increasing sateen number of face weave pattern, in other words by increasing average float length.
- Among the upholstery double fabrics woven with polyester warps, seam slippage strength along both warp and weft directions of those woven with cotton wefts has been found to be higher than those of upholstery double fabrics woven with wefts of staple polyester yarn.
- However, it has been seen that weft density has irregular effect on abrasion resistance of upholstery double fabrics.
- Abrasion resistance of upholstery double fabrics woven with cotton wefts has been observed to have a tendency to decrease by increasing the sateen number of face weave pattern, in other words by decreasing the weave interlacing coefficient,

while there has been no regular change in the abrasion resistance of upholstery double fabrics woven with wefts of staple polyester yarn.

- Contrary to seam slippage strength, abrasion resistance of upholstery double fabrics woven with wefts of staple polyester yarn has been found to be higher than that of upholstery double fabrics woven with cotton wefts.
- It has been observed that the increase in the sateen number of face weave pattern leads to lower degrees of pilling i.e. higher tendency of fibers to leave the structure of yarn. And so they will present on the surface of fabrics as pills.
- Upholstery double fabrics woven with cotton wefts have been detected to have a higher degree of pilling, i.e. a lower pilling tendency, compared to fabrics woven with wefts of staple polyester yarn.
- Furthermore, upholstery double fabrics woven with rayon warps, with twist of 500 tpm, have been found to have higher degrees of pilling, i.e. a lower pilling tendency, compared to that of upholstery double fabrics woven with polyester warps, with twist of 160 tpm.

Consequently, as weave geometry, which basically refers to woven pattern, determines the weave characteristics such as unit cell, the weave interlacing oefficient, average float length, friction between intersecting yarns and yarn settings, it has effects on the seam slippage strength, abrasion resistance and propensity to surface fuzzing and to pilling of double woven upholstery fabrics. In addition to this, as friction and abrasion characteristics of fibers, which are raw material of the yarns, and twist, determine the physical properties of yarns, they also affect properties which are mentioned above. However the effect of weft setting, which is determined by weave, yarn count and yarn type, is found to have a major effect on the seam slippage strength of double woven upholstery fabrics.

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Experimental researches on textile laminate materials

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REZUMAT – ABSTRACT

Cercetări experimentale privind materialele textile laminate

În articol sunt prezentate rezultatele experimentelor efectuate pe materiale textile laminate, utilizate la proiectarea şi construcţia muşchilor pneumatici artificiali. Aceste materiale laminate au o structură complexă, de aceea sunt incluse în clasa materialelor avansate destinate aplicaţiilor medicale, sistemelor de protecţie, aplicaţiilor aeronautice şi echipamentelor acţionate cu actuatori neconvenţionali. Cercetările experimentale au fost efectuate pentru a stabili caracteristicile mecanice ale acestor materiale textile laminate cu două straturi, precum: rezistenţa la tracţiune, alungirea maximă, gradul de alungire şi domeniul optim de lucru. Rezultatele experimentale obţinute au arătat că aceste materiale sunt adecvate pentru realizarea muşchilor pneumatici artificiali.

Cuvinte-cheie: materiale textile laminate, materiale textile avansate, muşchi pneumatici artificiali, laminare

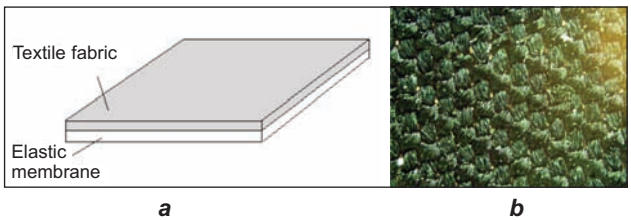
Experimental researches on laminated textile materials

This article presents the results of experiments conducted on a laminated textile material used in the design and construction of Pneumatic Artificial Muscle (PAM). This laminated material has a complex structure and is therefore considered part of the advanced materials class used in the medical applications, protective systems, aircraft applications and equipment that operate in unconventional actuators. The experimental investigations presented here are conducted in order to establish the mechanical characteristics of a laminated textile material with two layers, such as: tensile strength, maximum extension, rate of elongation and optimal working field. The experimental results obtained indicate that this laminated textile material can be used in the manufacturing of pneumatic artificial muscle.

Key-words: laminated textile materials, advanced textile materials, pneumatic artificial muscle, coating textiles

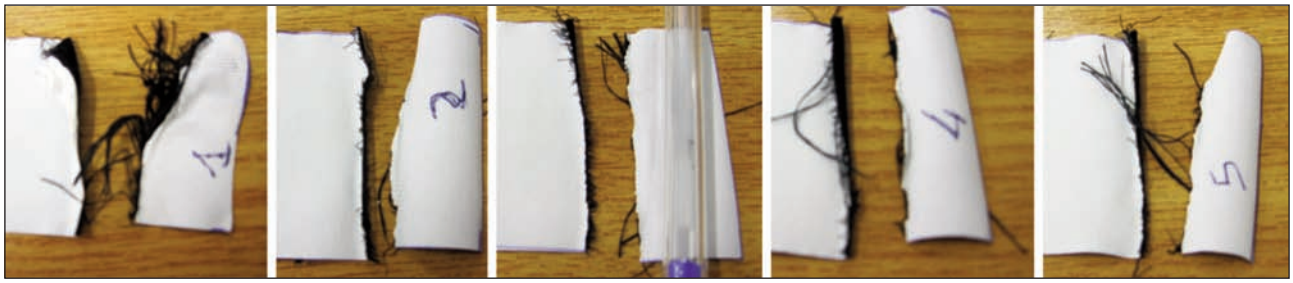
Laminate materials are known as sandwich structure, that are often made of metallic, ceramic or polymers layers; but in our case they are made of polymeric microfibers textile and a thin polyurethane membrane [8, 9]. These materials are the product of an advanced research on synthetic textile field, which are obtained due to advanced technologies and advanced polymeric materials. The main advantage of this textile laminate material is the high force-mass rate. Another important characteristic is their structure, which has a proper elasticity and impermeability to air and water [1]. Due to those characteristics, this material can be used to manufacture Pneumatic Artificial Muscles, known in scientific reports as PAM's. These types of actuators are incorporated in mechatronics systems that satisfy these requirements: small dimensions, low mass, high safety coefficient. The application fields of these nonconventional actuators are: medical equipment, outhouses, prosthesis, humanoid robots, rehabilitation equipment, active suits etc. [2, 6, 7].

In this paper, we will present a brief analysis of experimental results obtained from testing laminated fabrics in order to establish the mechanical characteristics and their optimal working field. These tests were conducted to determine whether "Superflex Mecpor" – a textile laminated material made by Mectex company, could be used to manufacture PAM. The textile material tested has a laminar structure made of 94% polyamide and only 6% elastane, with mass of 198.34 g/m^2, that has 30 fibers/cm on warp direction and 24 fibers/cm on weft direction. The first layer of this material (fig. 1 *a*) is a waving polyamide microfiber textile whose detailed imagine is presented in figure 1 *b* and a second layer a thin elastane membrane. Like any other elastic material the considerate fabric has anisotropic characteristics [9], and for this



a ***b***

Fig. 1. Superflex Mecpor – tested material:
a – laminate material structure; ***b*** – textile fabric structure



a b c d e

Fig. 2. Sample parts photos from traction test on elastic direction

reason the elastic behavior is different in correspondence with two perpendicular directions. Furthermore this material belongs to the group of high comfort materials that confer a body microclimate by means of a transpired membrane or resin embedded [www.mectex.it/mecpor.htm]. Apart from the features provided by the manufacturer, physical-mechanical parameters must be determined in accordance with international standards. In the next paragraph will be presented experimental methods and equipment used.

EXPERIMENTAL PART

Materials and methods

The experiments were conducted in order to establish the static and dynamic characteristic on elastic and rigid direction of coated material (that represent the weft and warp direction). These tests will help us to establish the optimum working range for these materials and rheological characteristic (deformation and flow science for plastic and synthetic materials) [3]. The experimental research here presented was carried on in Department of Mechanics of Politecnico di Torino – Italy. This activity was necessary to be done in order to establish the mechanical characteristics of this textile laminate materials, which are meant to be used in manufacture process of a PAM prototype. On this purpose was necessary to conduct traction and fatigue tests [3, 4].

The experiments were conducted on "MTS QTest/10" equipment connected to a PC that has an acquisition board on which was implemented dedicated software in order to obtain the data sheet. The traction equipment is a loading textile test bench is C.R.E. machine, according to ISO 2062-1972 – "Constant rate of specimen extension".

The displacement of the gripping device is adjustable and vertical, the distance between gripping devices is 250 $\pm$ 0,5 mm. This equipment has an: numerical control unit, mechanical load unit, a displacement gripping unit with constant speeds that can be establish in the digital interface, interchangeable load cell are 5, 10, 50 kN, the displacement transducer is solider with mobile clip.

Samples used in this test were 10 for traction and 10 for fatigue. In particular, 5 samples (200 mm x 50 mm) were cut in the elastic or weft direction and 5 samples (200 mm x 50 mm) in the rigid or warp direction in order to establish the orthotropic characteristic, according to the International Standards ISO 13934-1 "*Tensile properties of fabrics – Determination of maximum force and elongation at maximum force using the strip method*", because the textile fabric is the predominant material.

The testing procedure was as follows. The samples were placed and fixed in gripping device at 200 mm distance between them, ensuring that mounting surface is free of folds and the material is not prestressed. The measuring system must be set to zero, in the data acquisition program is necessary to insert the sample dimensions, the maximum loading force, range of displacement and also the loading speed (2 mm/s – the external load must be slow and progressive). During the deformation process is performed data acquisition stage and the operator must follow the tested sample.

Traction testing

From these tests it is needed to determine the yielding force, the rupture force, and the static characteristic. Also this stage is important in order to establish the loading field for fatigue test.

Traction test on elastic direction

In figure 2 are shown the elastic samples that had been tested. From these pictures it can be concluded that all the samples have been unraveled near to the gripping area, perpendicular to load direction, and the membrane remains attached on textile material.

In figure 3, are represented the load-extension characteristics of all samples. If we analyze the experimental results obtained it can be concluded that the experimental characteristics have a good repeatability, except for sample *1*. This case requires making a correlation between the images (fig. 2) and the characteristics (fig. 3). The material starts to extend up to 140 mm and a load of 170 N, suddenly it starts to breakdown until the second maximal point 180 N. This phenomenon can be seen also in figure 2 *a*, where the unraveled area does not have the same structure.

From the experimental results it was established that this material on the elastic direction has two linear deformations slopes. The first linear deformation slope begins from the origin until it riches the yielding force of 20 N that achieves an extension of 60 mm, from this point the material changes its linear deformation due to high stiffness until riches the rupture mean force of 232.35 N at a rupture mean extension of

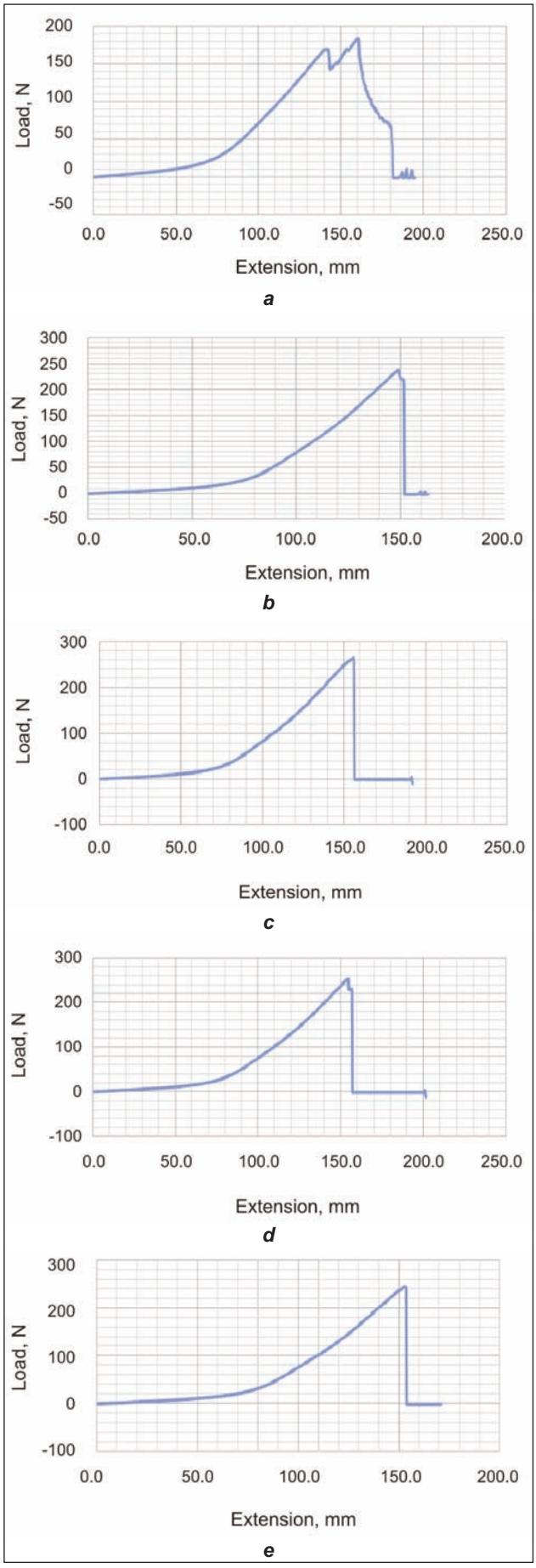


Fig. 3. Traction experimental results in elastic direction, load-extension characteristics:
a – sample *1*; ***b*** – sample *2*; ***c*** – sample *3*; ***d*** – sample *4*; ***e*** – sample *5*

146 mm that is 73.42% from sample initial length. However these materials looks like they are working together in the same time and the tensions generated between the layers are very low.

Traction test on rigid direction

In figure 4, the imagines of the tested samples, can be seen that are severely damages in all directions and also it can be noticed that this material has the tendency to ship from gripping jaws. For this reason the sample *3* slipped from the gripping device and was replaced by sample *6*.

In figure 5, are presented the load-extension characteristic of the tested samples at a speed of 2 mm/s. Analyzing the experimental results it can be concluded that load-extension characteristics on rigid direction are very different. For example sample *1*, characteristic is shown in figure 5 *a*, shows that the material behaves normally until it rich the load of 580 N, moment on witch is present a slide slope. After that the force continues to rise up until it reaches the fracture force of 600 N, where the material starts to unravel slowly, until the extension riches 53 mm. The samples *2* and *4,* shown in figure 5, have proper characteristics, but it can also be noticed that the oscillation, on rising slope and on decreasing slope, is present when the material starts to unravel. Regarding the sample *5* and *6*, the loading slope is more linear that the other cases, but the rupture is faster and it appends in two stages. The first stage represents the rupture of the resistance fibers that it's stops because have remained some fibers more elastic that will break in the second stage.

In all characteristics it can be seen an initial phase of adjustment, a repeatable linear feature, followed by slips and breaks. The mean fracture force is estimated at about 663.62 N and the maximal extension of 50.43 mm, representing 25.21% from initial length. However, the tensions generated in the textile fibers did not detach the membrane from textile material.

Fatigue testing

The fatigue test is one of the most important dynamic characteristic in order to determine the laminated material properties for a certain numbers of cycles. These tests are necessary to understand how this material behaves when it will be used to design an elastic element for PAM and where made in accordance with elastic and rigid direction, which represent the weft and warp material direction.

Fatigue tests on elastic direction

In order to decide the optimal working range, it was needed to establish the maximum load on the elastic direction (table 1). According to the traction test this limit is 200 N. Also the fatigue tests were carried out in order to determine if the textile structural integrity depends on the maximum load or the loading speed. The first test was made on sample *1*, which was performed to the maximum loading range. In this case the material started to unravel around the gripping area. For this reason, the next tests were done at

a *b* *c* *d* *e*

Fig. 4. Detail view of the tensile samples:
a – sample *1*; ***b*** – sample *2*; ***c*** – sample 3; ***d*** – sample *4*; ***e*** – sample *5*

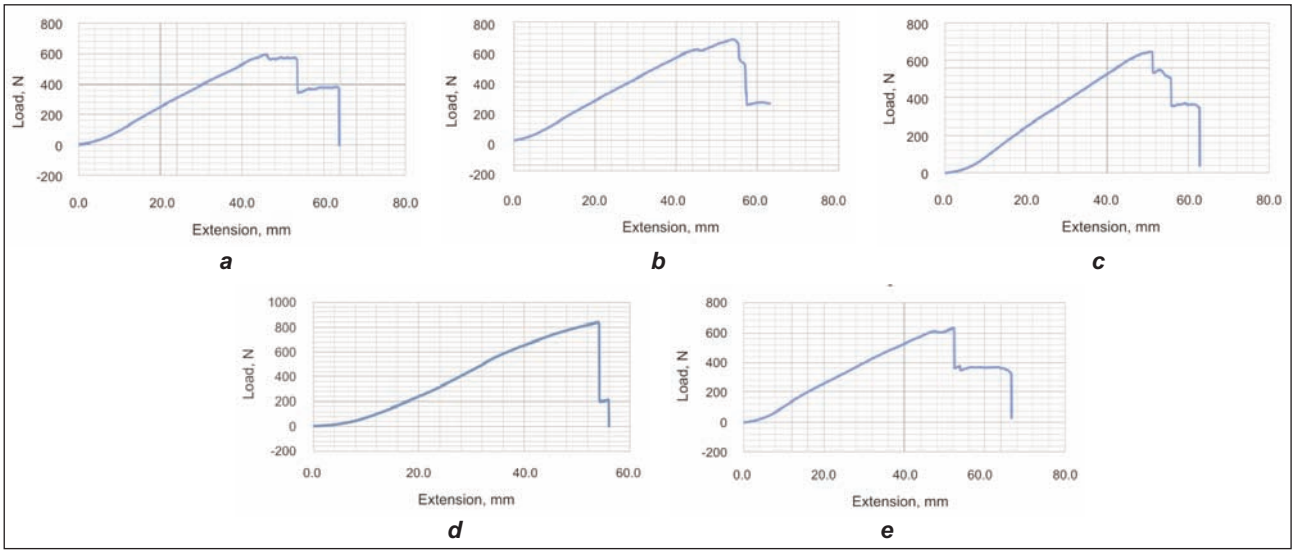


Fig. 5. Traction test experimental results in rigid direction, load – extension characteristics:
a – sample *1*; ***b*** – sample *2*; ***c*** – sample *3*; ***d*** – sample *4*; ***e*** – sample 5

Table 1

Sample no.	Number of cycles	Load range, N	Loading speed, mm/s
1	100	100 – 200	2
2	100	100 – 150	2
3	100	100 – 150	4
4	100	100 – 150	4
5	100	100 – 150	8

maximum load of 150 N. The sample *2* has no fault after testing, which means that this is the optimal loading range. Maintaining this range, it was increased the loading speed to 4 mm/s and later to 8 mm/s, representing the work frequencies of commercial PAM.
Analyzing the experimental results presented in figure 6, it can be concluded that this laminate material behaves in the same range of elongations and the loading speed has no influence on the material structure cut in elastic direction. In all cases, it can be established that the strain range of this tissue is about 7 mm at 100 cycles.

Fatigue tests on rigid direction

In this direction the experimental tests have been performed in the same way. The evaluation of the loading speeds, the maximum load and its variation are presented in table 2. The experiment results obtained on the rigid direction samples are shown in figure 7.
The chart from figure 7 *a* represents the experimental results for sample *1*, which has a loading frame from 200 to 400 N, with extensions of 2.28 mm. In this case, the membrane was damaged, especially the thin membrane that assures the air permeability of the laminated material. For this reason the loading range was changed, especially the lower value from 200 to 100 N. These values were established during experimentation. Decreasing the loading rage gives the opportunity to increase loading speed, from 2 mm/s up to 4 mm/s. In this case the test results are presented in figure 7 *b*, where extension was 3.25 mm and the material does not present any sign of deterioration.

Table 2

Sample no.	Number of cycles	Load range, N	Loading speed, mm/s
1	100	200 – 400	2
2	100	100 – 250	4
3	100	100 – 250	8
4	100	100 – 350	8

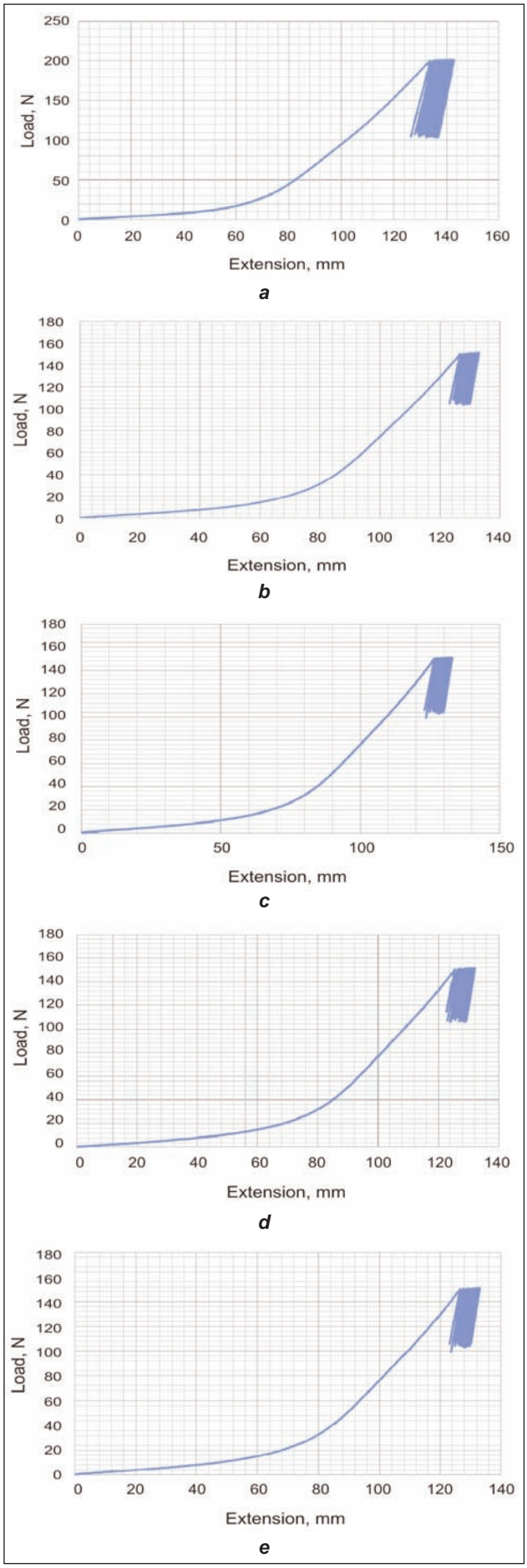


Fig. 6. Fatigue experimental results in elastic direction: ***a*** – sample *1*; ***b*** – sample *2*; ***c*** – sample *3*; ***d*** – sample *4*; ***e*** – sample *5*

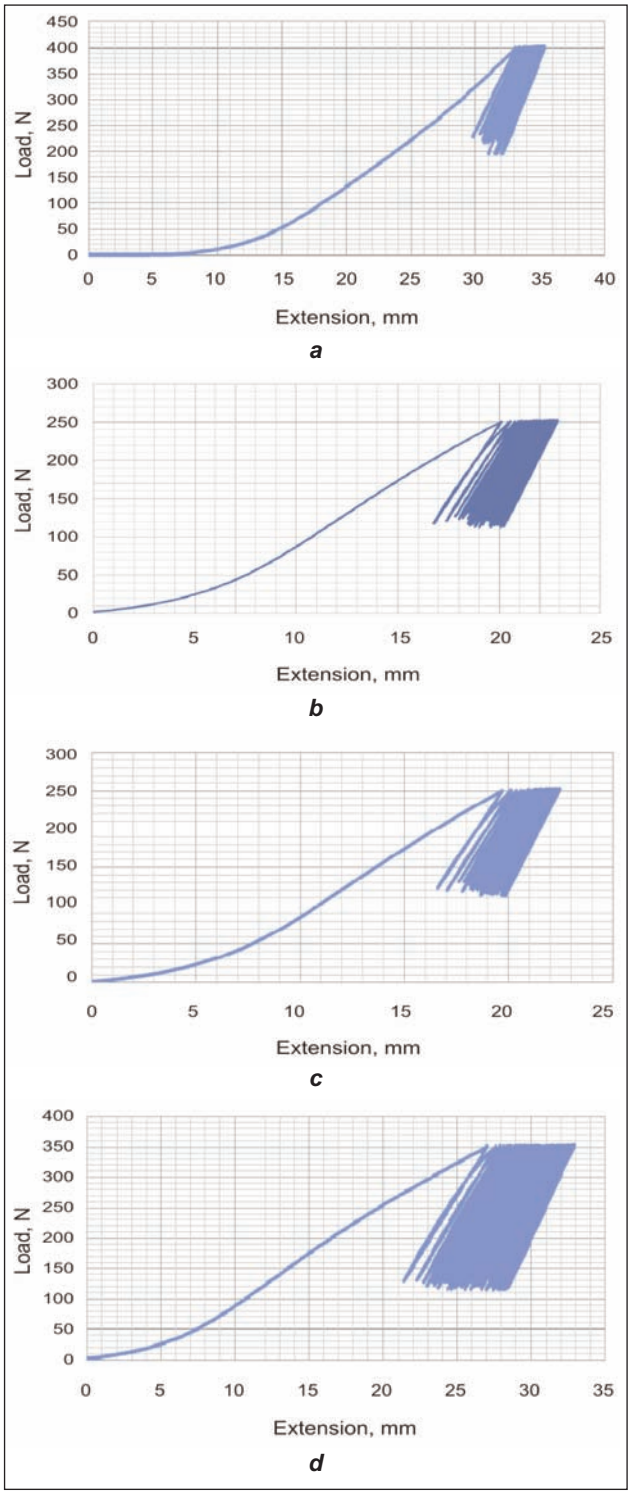


Fig. 7. Experimental fatigue test results on rigid direction: ***a*** – sample *1*; ***b*** – sample *2*; ***c*** – sample *3*; ***d*** – sample *4*

Next test was made doubling the loading speed. During testing procedure the material does not have any sign of damage, but the material extension was increased to 3.4 mm. The last test was carried out at the maximum load range of 350 N and at the same loading speed, case in which the extension was double (6.93 mm).

In conclusion, the fatigue characteristic in rigid direction depends on the maximum load values and on loading speed. In all experiments the membrane does not detach itself from the textile material, which means that these two materials are compatible.

RESULTS AND DISCUSSIONS

From the experimental traction tests, it can be established the mechanical parameters for this material on elastic and rigid direction. These characteristics can be used to establish the anisotropic properties of laminated textile material.

Figure 8 represents the material characteristic on elastic direction: the rupture force of 229 N per 50 mm width; the yielding force of 200 N, and is represented by point *C* and the maximum elongation is 73.42%. In this diagram it can also be noticed that the rigidity of this material is variable, due to initial elastic module in the first part of characteristic *OA* and the effective elasticity module on *BC* segment. The curve line describes the transition from the first elastic module (influenced by textile waive rigidity) to the second elastic module (influenced by fiber rigidity).

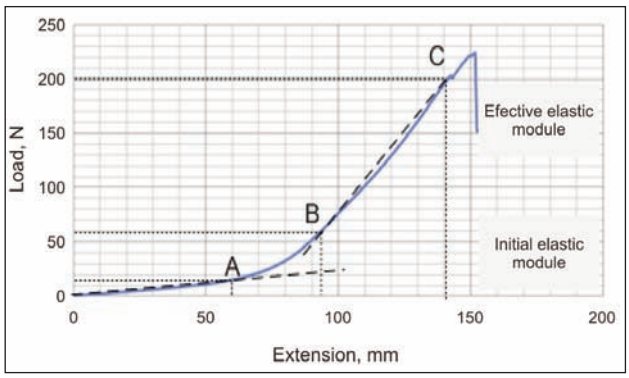


Fig. 8. Load - elongation diagram on elastic direction

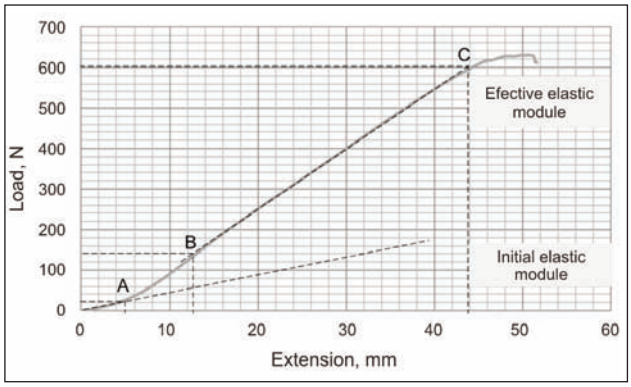


Fig. 9. Load - elongation diagram on rigid direction

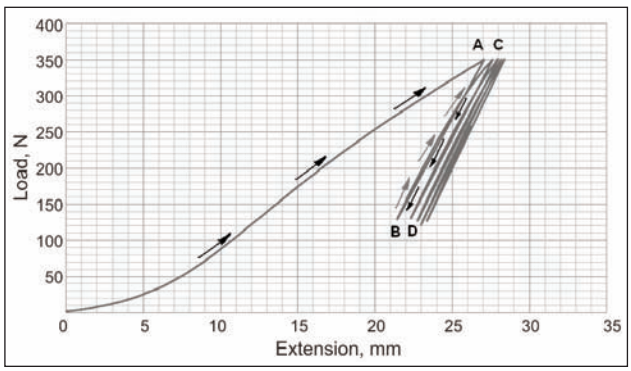


Fig. 10. Detail of fatigue test diagram in rigid direction

In the rigid direction there has been obtained the characteristic load – extension (fig. 9) where there were established the main parameters on this direction: the yielding force 663.62 N per 52 mm; rupture force 620 N and the maximum elongation is 25.21%.

Overall, the most important surface of this material is the elastic membrane, which cannot be loaded more than 200 N on elastic direction, in order to preserve structural integrity of the laminated material.

For these reasons the maximum working force of this material in both directions it is established at 200 N.

Analyzing the fatigue characteristic from figure 10, it can be stated that this material behaves like a non-linear elastic material, more exactly like an elasto-plastic material, because the loading and download curves (*OA*, *BC* and *AB*, *CD*) are not the same, which leads to irreversible deformations.

Also from this diagram it can be noticed that the material does not return to starting point when the loading force disappears and the material does not regain the initial dimensions, which means that it has a hysteresis and also a plastic deformation. This hypothesis was confirmed due to measurement made after fatigue tests, which registered a longitudinal deformation between 2 and 5 mm, measurements that were done during the next day.

The main factor affecting the fatigue characteristic is the internal damping, which has three phases, as it can be noticed in figure 10.

The first phase, the internal damping is small while the rigidity increases; the second phase, the internal rigiditychanges a little but the hardness is unchanged (smaller oscillations and more frequent) and third phase the internal damping increases until reaches the breaking point [5]. This phenomenon can be very well noticed in the fatigue diagrams on rigid direction (weft direction).

On the other hand, in the fatigue test made on elastic direction it can be noticed a good repeatability of loading curves, that finally reaches the minimum loading value, on this direction the relaxation does not influence the structure.

The stresses are concentrated where the textile material cannot change its configuration especially near the gripping area.

For this reason, during the PAM's design process and modeling, it is necessary to take into account this observation.

CONCLUSIONS

The present paper makes a brief analysis of experimental tests carried on innovative laminated materials developed with the latest technologies in the textile industry.

Due to their structure which uses the synthetic microfiber textile material in combination with elastic membrane, the testing procedures were not yet covered, but we have followed international standards for materials industry.

Like all new laminated materials, they have the objective to combine the mechanical performances of a textile material with the membrane elasticity, a successfully combination due to the fact that the integrity of this structure resists even for high loads.
This material can be framed as an elastic-viscous material with linear cold hardening with raw fissure in all directions.
The optimal working parameters are: maximum load of 200 N, maximum extension of 73.42% for a sample with an active surface of 0.01 m^2 and rate of elongation of 59.74%/minute. In conclusion it can be said that this material is very elastic and can be used successfully to design and manufacture the active chamber of a PAM, which could be implemented in rehabilitation equipment, outhouses or in active suits.

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Polyamide dyeing wastewater recycling after Fenton-like oxidative treatment

ARMAND-FLORIN BERTEA ROMEN BUTNARU

REZUMAT – ABSTRACT

Recircularea apelor uzate, provenite din vopsirea poliamidei, după decolorarea oxidativă tip Fenton

În lucrare este studiată posibilitatea recirculării apelor uzate, rezultate în procesul de vopsire a poliamidei cu coloranți acizi. În prima etapă, apele uzate de la vopsire şi cele de la spălarea ulterioară au fost decolorate folosind apă oxigenată activată cu catalizatori din răşini funcționalizate cu amine şi saturate cu Cu (II). Apele uzate astfel tratate au fost folosite în noi procese de vopsire a poliamidei, ca atare sau amestecate cu apă proaspătă. Apa oxigenată reziduală a fost îndepărtată din apele uzate înaintea vopsirii cu flotă recirculată. Au fost determinate reflectanța, diferențele de culoare, parametrii spațiului de culoare CIELAB (L, a* şi b*) şi rezistențele la spălare. S-a constatat că această variantă de decolorare oxidativă este o soluție fezabilă pentru recircularea apelor uzate, în special pentru cele cu un conținut redus de colorant. În scopul recirculării apelor uzate, provenite din procesul de vopsire, trebuie luate în considerare temperatura, pH-ul şi conținutul acestora în substanțe chimice.*

Cuvinte-cheie: ape uzate, vopsire, poliamidă, recirculare, coloranți acizi, decolorare oxidativă tip Fenton

Polyamide dyeing wastewater recycling after Fenton-like oxidative treatment

In this study, the possibility to recycle textile wastewater produced in acid polyamide dyeing was investigated. In the first stage of the research wastewater from the dyeing process and the wash-off wastewater were decolorized using hydrogen peroxide activated with catalysts from resins functionalized with amines and saturated with Cu (II). The treated wastewater was used as it is or mixed with fresh water in new polyamide dyeing processes. The residual hydrogen peroxide was removed from the wastewater prior to subsequent dyeings. Reflectance, colour difference, the colour parameters of the CIELAB colour space (L, a* and b*) and the wash fastness have been determined. It was found that oxidative discoloration is a feasible solution, especially for low dye content wastewaters. In order to recycle dyeing wastewater careful consideration has to be taken regarding the temperature, pH and chemical content.*

Key-words: wastewater, dyeing, polyamide, recycle, acid dyes, colour removal, Fenton-like system

As the natural water resources of the planet are more and more scarce and increasingly polluted, the reuse or recycle of industrial wastewater became a major investigation topic.

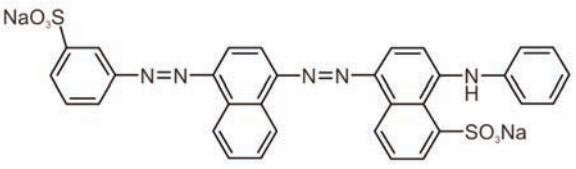
The textile industries produce high quantities of effluent with varying composition depending on the nature of the fibre and the type of the wet processes employed, water consumption varying between 3 dm^3/kg and up to 835 dm^3/kg [1]. Recycling wastewater for textile dyeing and finishing after an appropriate treatment is a solution to reduce the water consumption, and, in addition to this, has proven to result in savings in water, energy and chemicals [2, 3]. In most cases, textile wastewater is coloured, so the wastewater can be recycled in new finishing or dyeing procedures only after decolourization processes.

Many methods have been used to remove the colour of textile wastewater, among which the most studied are the membrane filtration, sorption, coagulation/flocculation, membrane and electrochemical and oxidative methods [4]. None of these methods proved to be effective in all cases, and there are supplementary difficulties in terms of cost and technological and practical considerations [5]. A method that leads to good results is the advanced oxidation, method which provides good results in removing the colour of most dyes, acid dyes included [6].

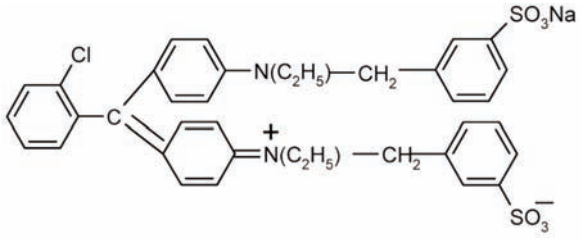
The purpose of this study was to evaluate the feasibility of the process of recycling wastewater from dyeing of polyamide in new dyeing stages, after removing colour by oxidative means. The basic goal was to arrive at a treatment system that would lead to a minimum of 90% reduction in the colour of the wastewater, and provide recycled dyebaths dyeings with a colour difference less than 1 unit comparing with the standard.

MATERIALS AND METHODS

Two half milling acid dyes have been used: Acid Blue 113 (disazo) and Acid Green 9 (triphenyl methane), both supplied by Bezema. The chemical structure of Acid Blue 113 is showed in formula (1) and chemical structure of Acid Green 9 is showed in formula (2).



(1)



(2)

These dyes have been tested without previous purification. 100% scoured polyamide fabric, weight 130 g per meter has been used for all the experiments. All dyeing processes have been performed in an Ahiba lab dyeing machine.

For the discoloration of wastewater, hydrogen peroxide solution (Merck, 30% w/w) has been used. To activate the hydrogen peroxide decomposition, catalysts from resins functionalized with amines and saturated with Cu (II) have been used. The colour removal experiments were conducted at 50ºC temperature, 60 minutes reaction time; the volume of the treated solution was 250 mL.

The colour removal degree, expressed in percentage, had been calculated from the relative decrease of absorbance, according to relation (3), where Abs_o is the absorbance of the initial solution and Abs_f is the absorbance of the final solution.

$$\%\ Colour\ Removal = \left(1 - \frac{Abs_f}{Abs_o}\right) \times 100 \qquad (3)$$

All absorbance measurements were made by a UV/Vis Camspec M501 spectrophotometer, at maximum absorbance wavelength (566 nm for Acid Blue 113 and 633 nm for Acid Green 9). The *p*H values were detected by a COLE PARMER *p*H/mV meter.

The CIELAB Colour Difference used to measure colour differences in samples was calculated by the following equation (4):

$$DE^* = [(dL^*)^2 + (da^*)^2 + (db^*)^2]^{1/2} \qquad (4)$$

A Datacolor Spectroflash SF-300 spectrophotometer has been used to measure the colour parameters and the colour of the polyamide samples dyed with recycled water. D65 – day light standard illumination condition, large area view, specular included have been used. In order to obtain a high opacity of the sample, each sample was folded twice.

The washing fastness was determined according to SR EN ISO 105 - CO6/1999.

EXPERIMENTAL PART

In the first stage of the research, synthetic wastewater has been used to analyse the colour removal process using hydrogen peroxide in the presence of Cu based Fenton-like catalyst.

In order to prepare the synthetic wastewater, 50 ppm of acid dye and 600 ppm sodium sulphate were dissolved in distilled water in a 1 dm^3 Erlenmeyer. These values are corresponding to the composition of a wastewater produced in a dyeing process at liquor ratio of 1:50 and an exhaustion of 80%.

The discoloration experiments was carried out according to the methodology that proved to offer optimal results in previous studies: 0,5 mL hydrogen peroxide 30%, activated with 0,06 g catalyst, have been added to 50 mL of wastewater samples (adjusted with NaOH to *p*H 9) and the treatment continued for 60 minutes [7]. Two treatment temperatures have been tested: 20ºC and 50ºC.

Subsequently, wastewater from real dyeing and the wash-off wastewater have been treated at 50ºC using the mentioned oxidative technology, and the treated wastewater has been recycled as it is or mixed with fresh water (1:1) in new dyeing processes of polyamide. The diagram of the dyeing process is shown in figure 1 [8], and the codification of the recycling experiments is presented in table 1.

Previous research showed that in oxidation discoloured wastewaters some residual oxygen peroxide remains, which significantly affects new dyeing processes [9]. In order to remove the residual oxygen peroxide an anionic reducing agent has been used.

New dyeing processes using treated wastewater was realised with the same dye as in the initial dyeing.

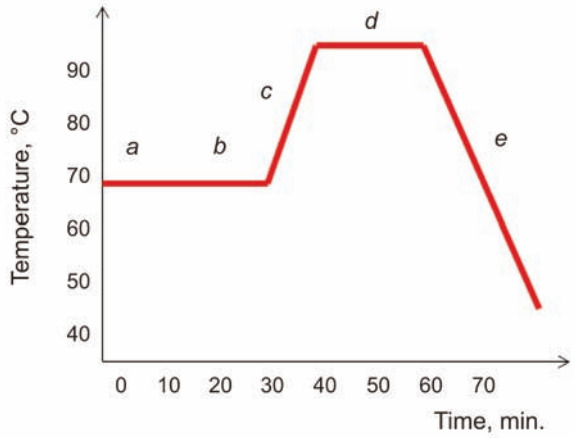


Fig. 1. The polyamide dyeing process:
a – prewetting; *b* – acid addition (3% owf - percent on weight of fabric - acetic acid); *c* – heating; *d* – setting; *e* – rinsing

Table 1

RECYCLING VARIANTS CODIFICATION

Dye concentration	Wastewater type	Recycle variant*	Number
Standard			**1**
1%	Dyeing	*a*	2
		b	3
	Rinsing	*a*	4
		b	5
3%	Dyeing	*a*	6
		b	7
	Rinsing	*a*	8
		b	9

**a* – as it is; *b* – mixed with fresh water (1:1)

Since the electrolyte (sodium sulphate) was available in the treated wastewater, the dyeing with the decolourised dyeing wastewater was carried out without any supplementary sodium sulphate and the dyeing with the dyeing treated wastewater mixed with fresh water was carried out by adding 50% of the initial sodium sulphate quantity. *p*H values of each recycled wastewater were adjusted to the starting *p*H values using acetic acid.

Reflectance, colour difference and the colour parameters of the CIELAB colour space (*L*, a** and *b**) have been determined. *L** represent the lightness of the colour and is the vertical coordinate of a three-dimensional system of colours, which has values from 0 (black) to 100 (for white), *a** is the horizontal coordinate ranging from green to red, and *b** is the horizontal coordinate that ranges from blue to yellow [10].

RESULTS AND DISCUSSIONS

The results for the discoloration of two acid dyes as obtained by the Fenton-like process are shown in figure 2. After only 30 minutes at 50ºC, up to 95% colour removal from the dye solution of both dyes was achieved, while at room temperature the colour removal degree was lesser than 70%. Therefore, is advisable to treat wastewater immediately after the dyeing process (while still warm).

For the majority of the recycling variants, the *DE** values were in the acceptable range of about 1 unit, and this proves that the decolorized wastewater did not undesirably affect the dyeing process. In the case of the 1% dyeing wastewater, for all the recycling variants the colour difference is even smaller than 1/2 unit (variants *2* to *5*). Colour difference for the samples dyed with 1% Acid Blue 113 using treated wastewater are shown in figure 3.

On the other hand, for the 3% dyeings, the colour differences are much bigger, especially for the dyeing wastewater (variants *6* and *7*), even after mixing it with fresh water. Better results have been obtained when using the treated wash-off wastewater (variants *8* and *9*).

Besides the colour difference *DE**, in order to identify colour deviations, the coordinates of the CIELAB colour space *L*, a** and *b** of the standard and the dyed samples were calculated. Is was found that the samples that showed big colour differences (variants *6* and *7*) show a significant shift towards red (bigger *a**) and yellow (bigger *b**), and the small *L** values indicate that they are darker in colour than the standard, while the samples corresponding to the other variants show only a minor shift towards green, without any colour darkening.

The brightness of the colour is generally not affected by the recirculation process (with the exception of variants *2* and *6*), where ΔC values are depicted (as Chroma denotes the colour intensity or its brightness or dullness [11]). Chroma difference for the samples dyed with 1% Acid Blue 113 using treated wastewater are shown in figure 4.

Washing fastness properties of the samples dyed with Acid Blue 113 using recycled wastewater are shown in table 2. It can be seen that all the samples have good washing fastness (even identical to the standard for the samples dyed with mixed recycled rinsing wastewater and fresh water – samples *5* and *9*).

The results obtained for the 1% Acid Green 9 dyeing processes using treated wastewater are shown in figure 5, which shows the colour difference between the dyed samples and the standard.

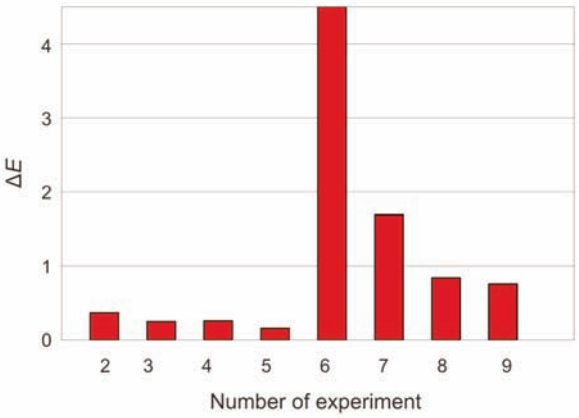


Fig. 3. Colour difference for the samples dyed with 1% Acid Blue 113 using treated wastewater

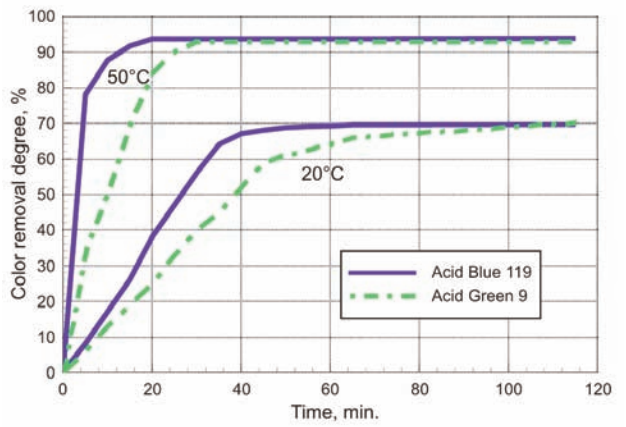


Fig. 2. Variation in time of color removal at two treatment temperatures

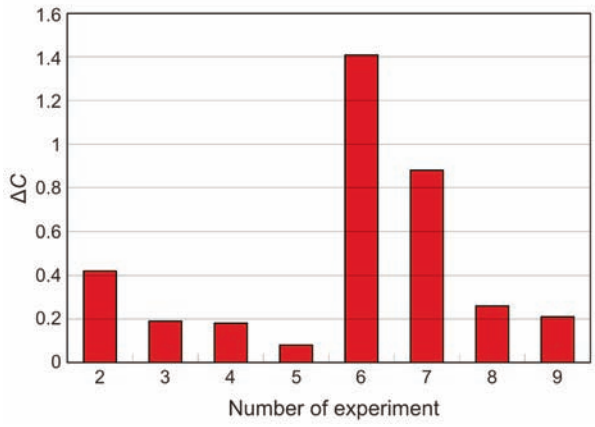


Fig. 4. Chroma difference for the samples dyed with 1% Acid Blue 113 using treated wastewater

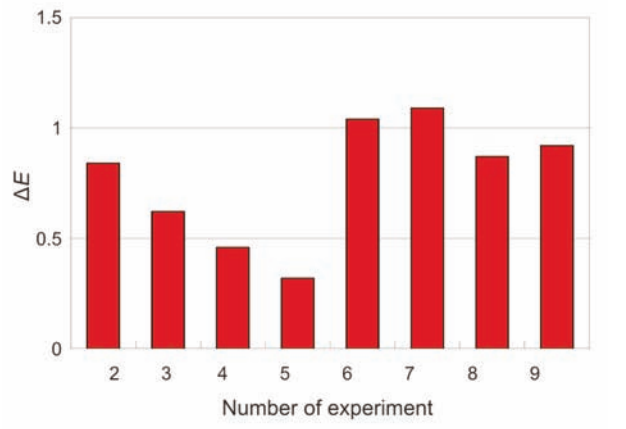


Fig. 5. Colour difference for the samples dyed with 1% Acid Green 9 using treated wastewater

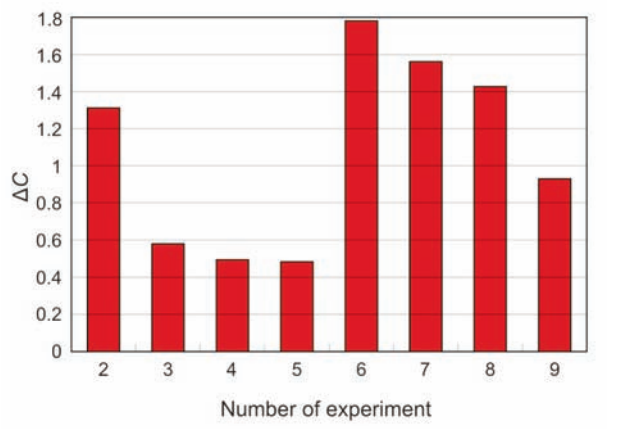


Fig. 6. Chroma difference for the samples dyed with 1% Acid Green 9 using treated wastewater

The results obtained show that, with the exception of variants *6* and *7*, the colour differences are bigger than the ones for Acid Blue 113, but they are within the accepted interval of 1 unit. As it was expected, the smaller colour difference is obtained for variant *5*, when treated wash-off wastewater mixed with fresh water has been used.

Even for the wastewater generated in the 3% dyeing the colour difference is very close to the limit of 1 unit (1,04 and 1,09 respectively), and visually there aren't any colour differences.

In order to identify the existence of colour shifts, *L**, *a** and *b** have been analysed. It was observed a slight colour shift towards red (bigger *a**) and on a smaller scale towards yellow (bigger *b**). These tendencies are more pronounced for variants *6* (*db** = = 0.3) and *7* (*da** = 1.65). It is to notice a significant decrease of *L** values, meaning that the samples dyed with recycled wastewater are darker (variants *6* and *7* lead to the bigger differences – 2.05 and 1.84 respectively), while variants *4* and *5* (wash-off recycled wastewater) show the smallest differences in lightness (0.55 and, respectively, 0.99).

Chroma difference for the samples dyed with 1% Acid Green 9 using treated wastewater are shown in figure 6. The brightness of the colour is just slightly affected by the recirculation process only for the 1% concentration dyeings, and especially when wash-off recycled wastewater was used, as it can be observed from figure 6, where DC values are represented.

Table 2 show the washing fastness properties of the samples dyed with Acid Green 9 using recycled wastewater. No modification in washing fastness can be observed, even for the 3% dyeing wastewaters (variants *6* and 7), which proves that the recycling process does not affect the wash fastness.

Table 2

WASHING FASTNESS PROPERTIES			
Acid Blue 113		**Acid Green 9**	
Number of experiment	**Washing fastness**	**Number of experiment**	**Washing fastness**
1	5/4-5/4-5	1	5/4-5/5
2	4-5/4/4-5	2	4-5/4-5/5
3	4-5/4/4-5	3	5/4-5/4-5
4	5/4/4-5	4	4-5/4-5/5
5	5/4-5/4-5	5	5/4-5/5
6	4-5/4/4	6	4-5/4/5
7	4-5/4-5/4-5	7	5/4/5
8	4-5/4/4-5	8	5/4/5
9	5/4-5/4-5	9	5/4-5/4-5

CONCLUSIONS

On the basis of the experimental data obtained, it can be concluded that the use of hydrogen peroxide activated with resins functionalized with amines and saturated with Cu (II) efficiently removes the colour of wastewater produced in dyeing processes using Acid Blue 113 and Acid Green 9.

The reuse of the oxidative discoloured wastewaters from polyamide dyeing showed small colour differences compared to standard. It was concluded that oxidative discoloration is a feasible solution, especially for low dye content wastewaters. For higher concentration wastewater, only recycling after mixing with fresh water leads to acceptable colour differences, but the brightness of the colour is affected (especially for the green dye). In all cases, the washing fastness was not affected by the recycling process.

In order to recycle dyeing wastewater careful consideration has to be taken regarding the temperature, *p*H and residual hydrogen peroxide elimination, and this is why supplementary work has to be done to evaluate the way multiple recirculation cycles affects colour (especially its brightness).

The payback on the wastewater recycling depends on the degree of constructional changes needed, and the number of times the recycling can be performed.

Acknowledgements

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INDUSTRIA TEXTILĂ ÎN LUME

UN NOU CENTRU EUROPEAN PENTRU INOVARE ÎN DOMENIUL TEXTILELOR

În octombrie 2012, în urma unei investiții de aproximativ 46 de milioane de euro, a fost deschis oficial ***Centrul European de Textile Inovative*** – ***CETI***, la Roubaix/Franța.

Crearea acestui centru s-a făcut cu sprijinul unor organizații franceze, al guvernului francez, al unor organizații guvernamentale regionale şi al Uniunii Europene.

Centrul este dotat cu echipamente ştiințifice şi de laborator, utilaje textile pentru extruziune, filare, țesere, tricotare, împletire, finisare şi producerea de nețesute consolidate prin filare în stare uscată sau filate din topitură etc.

În cadrul centrului, vor fi realizate activități de cercetare şi de producție la scară mică, se va acorda asistență în găsirea unor parteneri potriviți pentru executarea de proiecte şi se va oferi asistență tehnică. De asemenea, noul centru va încuraja cercetarea aplicată, reunind universități, laboratoare şi companii, şi combinând abilități şi resurse pentru elaborarea unor soluții inovatoare.

Cu o suprafață totală de 15 000 m^2, CETI cuprinde spații pentru ateliere, laboratoare, birouri şi săli pentru întruniri şi conferințe.

La deschidere, CETI a prezentat un raport privitor la viitorul textilelor tehnice, care a fost urmat discuții pe această temă, şi a organizat o masă rotundă în cadrul căreia s-au arătat motivele înființării centrului şi aspirațiile organizatorilor.

Technical Textiles International, octombrie 2012, p. 5

Dyeing of cotton, silk and wool with Bixa orellana in the presence of enzymes

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REZUMAT – ABSTRACT

Vopsirea bumbacului, mătăsii şi lânii cu colorant Bixa orellana, în prezența enzimelor

În lucrare sunt prezentate proprietățile țesăturilor din bumbac, mătase şi lână vopsite cu ultrasunete în comparație cu cele ale țesăturilor vopsite în mod convențional cu extract apos din semințe de Bixa orellana, utilizând enzime în locul mordanților pe bază de metal. Colorantul Bixa, aspectul țesăturilor şi compatibilitatea enzimei au fost evaluate prin analiza SEM. Pentru țesăturile analizate, s-au determinat valorile CIELab, valorile K/S şi proprietățile de rezistență a vopsirii. Enzimele – proteaza, amilaza şi celulaza – au fost special selectate, pentru a conferi stabilitate extractului şi intensitate culorii. Capacitatea tinctorială a colorantului obținut din extractul de semințe de Bixa a fost îmbunătățită prin ultrasonare. În cazul celor trei tipuri de țesături, s-a observat o creştere cu 8–10% a absorbției de colorant. Utilizarea enzimelor în locul mordanților pe bază de metal este recomandată ca metodologie de vopsire la scară industrială, fiind mai fezabilă şi mai sigură din punct de vedere ecologic.

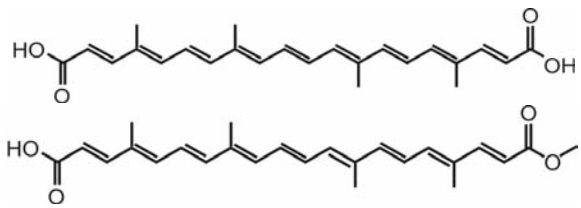
Cuvinte-cheie: enzime, vopsire naturală, semințe de Bixa orellana, ultrasunete, rentabilitate, poluare redusă

Dyeing of cotton, silk and wool with Bixa orellana in the presence of enzymes

In the present study we wish to report the properties of cotton, silk and wool fabrics subjected to ultrasound dyeing in comparison with those of fabrics conventionally dyed with the aqueous extract of Bixa orellana seeds by using enzymes in place of metal mordants. Bixa dye, fabric aspect and enzyme compatibility was substantiated by SEM. CIELab values, K/S values and fastness properties of the dyed fabrics were ascertained for the tested fabrics. Enzymes – protease, amylase and cellulase, were particularly chosen as they gave stability to the extract and the color content deepened. The dye ability of Bixa seed extract improved with the aid of ultrasonication. The enhancement of 8–10% dye up-take in the three fabrics was noted. The use of enzymes in place of metal mordant is recommended as industrial dyeing methodology, being very feasible and ecologically safe.

Key-words: enzymes, natural dyeing, Bixa orellana seeds, sonication, cost effectivity, less polluting

The colored constituents of Bixa orellana, commonly known as annatto, possess properties which find good application in textile industry. The colorant is obtained from the outer layer of seeds of this tropical tree. The chemical constituents of the colorants in annatto are a mixture of bixin, which is a monomethyl ester of a dicarboxylic carotenoid compound, and norbixin, which is a dicarboxylic derivative of same carotenoid as in bixin, presented in chemical formulas (1) and (2) [1]. Both these constituents occur in cis form, and a small percentage of both are likely changed into the more stable trans form upon heating. Bixin is a half-ester, and this property of its gives it some solubility in lipids.



Recently, Kawlekar and Mukundan [2] reported about the optimisation of dyeing conditions and fastness properties of the cotton fabric dyed with annatto imparting beautiful shades to cotton fabric, when used with mordants as well as with some inorganic salts at alkaline pH. Though fastness of the dyed fabric to light was found to be moderate in general, fastness to perspiration, washing and rubbing were found to be moderate to excellent. Thus, they reported that there is a scope to improve the fastness properties by finding the means to make the dye more stable and thus resistant to fading. Gulrajani et al. [3] used annatto seeds for dyeing of nylon and polyester fibres. They observed that though both the fibres have good affinity for this dye, the mechanism of dye adsorption, as indicated by the isotherms, is different in each case. They observed that the dye has moderate fastness to washing but poor to light.

Other properties of annatto colorants include their un-stability to oxidation, non-toxicity, and flexibility. Traditionally, annatto seeds have been used in medicines to cure wounds, skin eruptions, and burns. Annatto colorants are also used for the coloration of wool and silk. The less toxic properties of annatto

colorants make it a widely used colorant in the textile industry.
Colorants derived from the seeds of Bixa are extensively used in the food industry [4]. Their peculiar characteristic, as that which permits obtaining water-soluble and lipid-soluble colorants through small alterations of the production process, was one of the factors responsible for the success of this pigment. It can be observed that only recently, a little over five years, efforts have been dedicated to the study of the physico-chemical characteristics of the different carotenoid structures that compose this colorant. On the other hand, complementary toxicological studies of these colorants are being demanded, putting their utilization in question, and only with the conclusion of these work can their future be evaluated.

EXPERIMENTAL PART

Materials used

The cotton and silk fabrics used were scoured by treatment with a solution containing 5 g/l of sodium carbonate per 160 g of the fabric and 3 g/l of non-ionic detergent (Labolene) per 160 g of the fabric, under the boiling condition for 1 hour, after which time it was thoroughly rinsed and air dried at room temperature. A dye used is Bixa seed powder.
Enzymes used are: protease, amylase and cellulase. All other chemicals used were laboratory grade reagents.

Dyeing process

Cotton dyeing was carried out in two ways:

- One step/simultaneous dyeing – the dye, tannic acid and the enzymes (in the ratio of 20 g Bixa dye, 10 g of tannic acid and 1 g enzymes per liter for 100 g of the fabric) were all taken in one bath and the moist fabric was dipped for 3 hours at temperature 30–40°C [5–7]. The dyed fabrics were dipped in 500 ml of dye-fix solution (2 g/l) for 1 hour and then it was rinsed thoroughly in tap water and allowed to dry in open air;
- Two steps/stepwise – the tannic acid and the enzymes (in the ratio of 10 g tannic acid and 10 g enzymes in 1 litre were taken) were first treated onto taken in one bath and the moist fabric (100 g) was dipped for 3 hours at temperature 30–40°C. Dyeing was then carried out of this fabric with 20 g/l dye solution. The dyed fabrics were dipped in 500 ml of dye-fix solution (2 g/l) for 1 hour and then it was rinsed thoroughly in tap water and allowed to dry in open air.

Silk and wool dyeing were carried out in one way – no tannic treatment was required:

- One step – the dye and the enzymes (in the ratio of 20 g Bixa dye and 1 g enzymes per liter for 100 g of the fabric) were all taken in one bath and the moist fabric was dipped for 3 hours at temperature 30–40°C. The dyed fabrics were dipped in 500 ml of dye-fix solution (2 g/l) for 1 hour and then it was rinsed thoroughly in tap water and allowed to dry in open air.

Enzyme treatment

Reactivity for the different enzymes – protease, amylase, diasterase, xylase, phytase and cellulase, was checked for this natural dye with reference to the fabric. Thus cotton was treated with cellulase and Bixa, similarly protease was used for silk and amylase was used for wool for their best results. Specific enzyme reactivity was assessed for silk, wool and cotton in order to check the best combination. After treatment all samples were rinsed and squeezed and further use for dyeing.

Measurements and analysis

Color measurements

The relative color strength of dyed fabrics expressed as *K*/*S* was measured by the light reflectance technique using the Kubelka–Munk equation (3) [8, 9]:

$$K/S = (1 - R)^2/2R \quad (3)$$

where:

R is the decimal fraction of the reflectance of dyed fabric;

K/*S* – was measured for one step dyeing process as well as for two steps process.

Bixa did not show much difference in cotton dyeing in *K*/*S* values with different enzymes. However it showed distinct difference in *K*/*S* values with silk and wool using protease enzyme.
The reflectance of dyed fabrics was measured on a Premier Colorscan.

Fastness testing of dyed samples

The dyed samples were tested according to Indian standard methods [10]. Color fastness for light tested according to IS-2454-85, color fastness to rubbing according to IS-766-88, color fastness to washing according to IS-687-79 and color fastness to perspiration according to IS-971-83. Xenotest was used to test the light fastness of the dyed fabric. Wash wheel of Thermolab model was used test the washing fastness of the dyed fabric. Perspirometer of Sashmira model was used for the testing of perspiration fastness of the dyed fabric. Crock meter of Ravindra engg. Model was used for testing the rubbing fastness of the dyed fabric. Color Matching system used for the measurement of reflectance of dyed fabrics was on a Premier Colorscan.

RESULTS AND DISCUSSIONS

Silk and wool fabrics have been dyed employing extract of seeds of in absence and presence of metal mordants such as magnesium sulphate, aluminium sulphate and ferrous sulphate by Das et al. [11]. Coloration of both the fibres is found to be most effectively accomplished at *p*H 4.5 commonly in the absence and presence of such inorganic salts. Color uptake for wool is found to be more than that for silk under all the conditions studied. Similarly Devi et al. [12], showed improved procedures leading to better dye fixation on textiles for Annato dye. The optimum

proportions for dyeing with Annato on cotton has been discussed using myrobalan as mordant.
However, we wish to report a comparison on ultrasound dyeing and conventional dyeing properties of cotton, silk and wool fabrics with the aqueous extract of Bixa orellana seeds by using enzymes in place of metal mordant.

Preparation and optimization of aqueous extract of Bixa

The seeds of Bixa were found to give out color in hot water very easily. The seeds were crushed and dipped in hot boiling water to get the maximum color in 30 minutes which shows deepening of hue color. Increasing the quantity of seeds from 10 g to 20 g per 100 ml water boiled for 60 minutes is accompanied with the increase in color strength and depth in color hue.

Enzyme treatment

Reactivity for the enzymes used for different fabrics are given in table 1. After treatment all samples were rinsed and squeezed.

Table 1

ENZYMATIC TREATMENT CONDITIONS FOR DIFFERENT FABRICS

Enzyme	Treatment time, hours	Enzyme concentration, g/L	*p*H	Temp., °C
Protease/Silk	3	1.0	7.2	25
Protease/Wool	3	1.0	7.2	25
Amylase/Cotton	3	1.0	7.0	30
Cellulase/Cotton	3	1.0	7.2	25

Dye uptake

Dye uptake was studied during the course of the dyeing process for a total dyeing time of 3 hours with and without ultrasound. About 57%, 63% and 55% exhaustion of Bixa dye (cotton, silk and wool, respectively) can be achieved in 3 h dyeing time using ultrasound while compared to only 46%, 52% and 42% in absence of ultrasound in stationary condition for natural dye respectively as shown in table 2.

Table 2

REACTIVITY OF BIXA DYE WITH DIFFERENT FABRIC AND COMPATIBLE ENZYMES

Dyeing method	Cotton	Silk	Wool
Conventional	46%	52%	42%
Sonicator	57%	63%	55%

As is shown in the table 2, one step (scouring – mordanting – dyeing) ultrasound technology afford a higher dye affinity for wool, silk and cotton than the two steps processes (conventional).
Normally, the coloring maters, bixin and norbixin, being polar and acidic in nature show a higher affinity for wool and silk (having more polar groups) than for cotton which has only hydroxyl group in its structure. The use of ultrasound intensify the removal of the outermost waxy layer and intensify diffusion of dyes into fibers.

Fastness properties

The main focus of the research is the study of pre-treatment with enzyme, with the aim of improving natural dye uptake. In our previous research works [7], we have demonstrated that the enzymatic treatments could modify the fiber surface enhancing the accessibility to organic molecules in water medium, without affecting the bulk morphological characteristics and the mechanical behavior of the fibres. This research has been focused on optimum pre-treatment conditions of natural dyeing processes on natural fibers (cotton, silk and wool), such as enzyme concentration, *p*H, liquor ratio, mechanical agitation of the bath in terms of sonication, temperature and time suitable to enhance dye-bath exhaustion, dye penetration into the fiber, color fastness etc. Commercial enzymes have been used in the study.
It was observed that dyeing with Bixa seed extract gave fair to good fastness properties in conventional dyeing as well as sonicator dyeing. The table 3 shows

Table 3

CIELAB OF DIFFERENT FABRICSS

Type of fabrics		*L**	*a**	*b**	*C*	*H*	*dE*	*K/S*
Amy-Cot	untreated	72.42	10.47	29.11	30.15	60.33	--	10.32
	treated	70.40	12.95	31.61	34.16	67.68	4.90	15.22
Cell-Cot	untreated	72.14	9.48	29.01	30.18	68.00	--	11.19
	treated	70.15	10.93	30.81	32.69	70.44	2.19	13.38
Pro-Silk	untreated	77.86	7.68	40.22	42.27	75.34	--	4.26
	treated	76.51	8.34	43.59	44.38	79.12	14.21	18.47
Pro-Wool	untreated	75.38	9.24	36.87	38.01	72.35	--	32.84
	treated	73.95	10.41	38.42	39.80	74.80	8.09	40.93

Note: *L** is lightness of the color; *a** – position between red/magenta and green; *b** – position between yellow and blue; *C* – chroma; *H* – Hue angle

*L**, *a** and *b** values and can be seen that enzyme treatment shows lower value of *L** show darker shades while higher *L** values signifies lighter shades. Similarly negative a* and negative *b** represents green and blue shifting respectively for cotton, silk and wool fabrics. The results clearly show that dyeing with the extract of Bixa seed extract shows better dye uptake, even without the use of enzyme but the dye did not adhere properly.

The higher values of *L** indicate a brightness shades of colour for silk and wool meaning a better scouring and a higher uptake of dyes than for cotton. The results are confirmed by the increased values of *H* and *K/S*. *K/S* values being proportional to the dye concentration in the sample for silk and wool.

This process showed reduced dyeing time and cost effective as the seed is available in abundance. Overall, it could be used for commercial purpose, the dyed fabrics attain acceptable range in terms of fastness properties as shown through *K/S* values. In figure 1 shows *K/S* values for cotton, silk and wool treated with different enzymes.

Amylase and cellulase for cotton (shown as # 0 and 1) also indicates that the coloristic efficiency or tinctorial value of this natural dye for cotton fabric gives a pretty camel color and protease was used for wool (# 2) and silk (# 3) fabrics as shown in figure 2, which gives yellowish tinge.

Effect of ultrasound

Generally, the sonochemical activity arises mainly from acoustic cavitation in liquid media. The acoustic cavitation occurring near a solid surface will generate microjets. The microjet effect facilitates the liquid to move with a higher velocity resulting in increased diffusion of solute inside the pores of the fabric. In the case of sonication, localized temperature raise and swelling effects due to ultrasound may also improve the diffusion. The stable cavitation bubbles oscillate which is responsible for the enhanced molecular motion and stirring effect of ultrasound. In case of cotton dyeing, the effects produced due to stable cavitation may be realized at the interface of fabric and dye solution.

Many studies have shown that the use of ultrasound technology in wet processes as desizing, scouring, bleaching, mercerizing, dyeing is beneficial due to low energy and chemicals consumption, production time reduction, preservation of physical properties of textile materials, lowering overall processing costs, thereby increasing industry competitiveness.

The ultrasounds do not impair the complex structures of the enzyme molecules, but significantly improves their performance, leading to performances comparable to those obtained by traditional alkaline scouring [13]. Outside the general benefits, the ultrasounds have positive effects on dyeing by enhancing the dye up-take and allowing realtime control of color shade.

Generally, the sonochemical activity arises mainly from acoustic cavitation in liquid media. The acoustic cavitations occurring near a solid surface will generate microjets. These high speed microjets dilate the amorphous regions, increase fiber swelling in water, and diffusion of solute inside the pores of the fabric. In the case of sonication, localized temperature raises and swelling effects due to ultrasound improves the dye diffusion in the fiber, the fiber/dyebath partition coefficient. In the same time, the ultrasonic cavitation breakup of the dye agglomerate structures and could generate nanosized particles, enhancing the uptake of the dye on textile material.

The oscillating effect of the stable cavitation bubbles is responsible for the enhanced molecular motion and high stirring velocity of ultrasound. In case of cotton dyeing, the effects produced due to stable cavitation bubbles may be realized at the interface of textile material and dye solution.

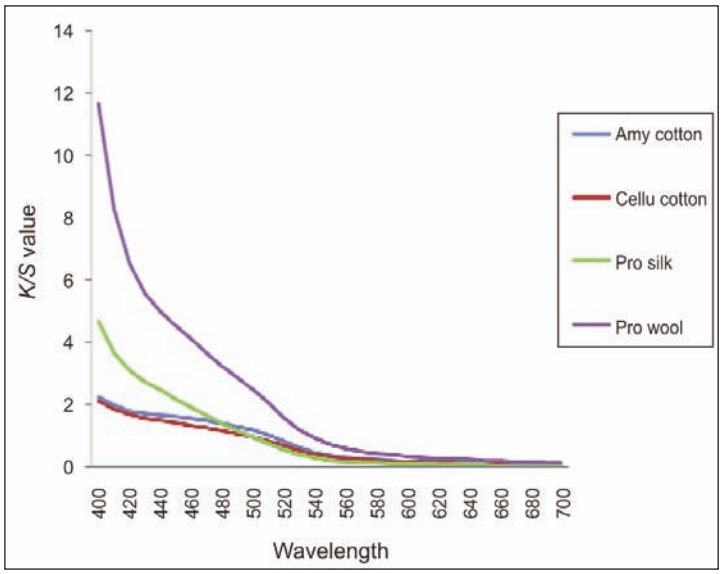


Fig. 1. Bixa dyeing on enzyme treated cotton, silk and wool

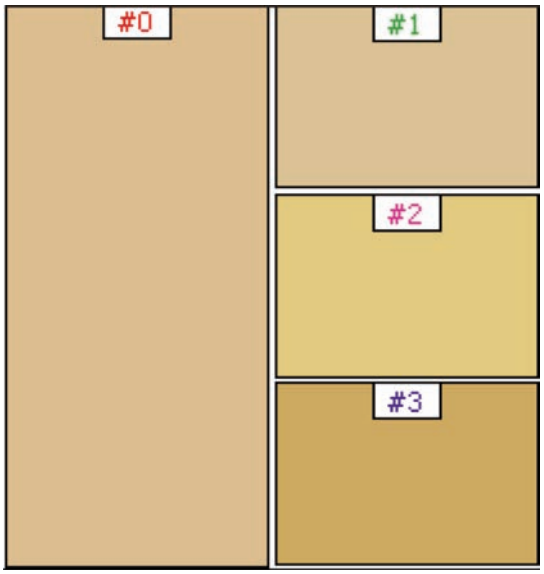
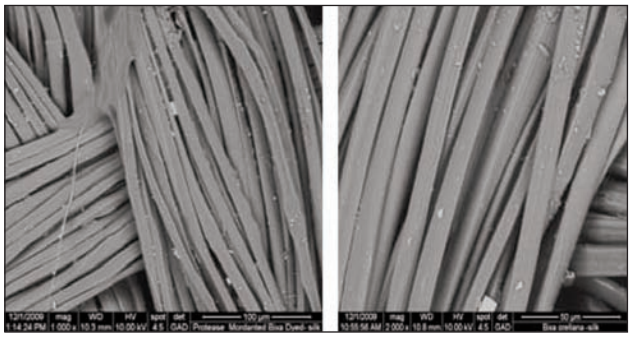


Fig. 2. Shades the color on different fabrics

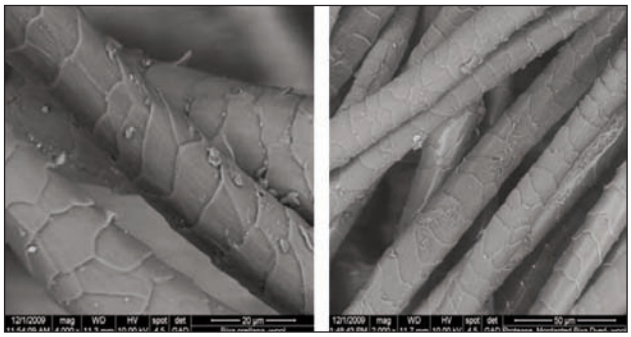
a *b* *c*

Fig. 3: ***a*** – SEM image of untreated cotton;
b – SEM image of amylase treated cotton; ***c*** – SEM image of cellulase treated cotton



a *b*

Fig. 4: ***a*** – SEM image of untreated silk;
b – SEM image of protease treated silk



a *b*

Fig. 5: ***a*** – SEM image of untreated wool;
b – SEM image of protease treated wool

SEM images of dyed fabrics

The SEM images of dyed cotton fabrics of control (without enzyme treatment) and with amylase and cellulase shown as figure 3 *a-c*.

The SEM images reveal a more aggressive effect (fibrils) of cellulase on cotton, breaking-up and peeling a part of the outer layer of the fibers, due to its inherent property to cleave ß-1,4-glycosidic bonds in cellulose. A better cleaning effect is observed in the case of amylase (a high number of impurities are removed from cotton surface) than in the case of cellulase. These effects are confirmed by the lower brightness (*L** is 70.15) and the higher redness (*H* is 70.44 compared with 67.68 of cotton sample treated with amylase). The amylase treatment has no detrimental effect of fibers, removing the impurities from the fibers, producing a smooth, clean surface and improving the dyeing uniformity. Protease penetrates the exterior layer of fibers through cracks or micropores, allowing the removal of sericine and consequently a better contact between the dyes molecules and the fibre.

The SEM images of dyed silk fabrics without and with enzyme treatment show a more uniform fiber surface with less impurities in the case of protease treatment (fig. 4 *b*) than in the case of untreated silk (fig. 4 *a*).

The SEM images of dyed wool fabrics of control (without enzyme treatment) and with protease shown as figure 5 *a-b* very clearly show that the dye molecule remain at the surface of fibers in the untreated figure 5 *a,* while in figure 6 *b* the dye is well impregnated. The applied enzymatic ultrasound treatment does not damage the fiber structure, confirming the investigations of numerous authors [14–18] that protease diffuses into the wool fibre and causes no significant modifications on the outer fibre surface.

Fastness properties of dyed fabrics

The rating of fastness of fabrics dyed with Bixa are shown in tables 4-6. The cotton fabrics dyed in one step process have an improved fastness to washing, alkaline perspiration, dry rubbing and light. In the case of materials dyed in the presence of cellulase, the fastness properties showed an improvement of one scale in light fastness than the cotton fabrics dyed in presence of amylase. The wool and silk fabrics show better results than cotton due to the high affinity of polar carotenoid dyes with protein structures.

CONCLUSIONS

A comparison on ultrasound dyeing and conventional dyeing properties of cotton, silk and wool fabrics with the aqueous extract of Bixa orellana seeds by using enzymes in place of metal mordant was carried out. Aqueous extract of Annatto yield yellowish to brown shades on cotton silk and wool fabrics with

Table 4

FASTNESS PROPERTIES OF DYED COTTON FABRICS UNDER CONVENTIONAL AND ULTRASONIC CONDITIONS OF AMYLASE AND CELLULASE ENZYMES AND BIXA DYE

Dyeing methods	Wash – perspiration – rubbing – light					
	WF	Per_{acidic}	Per_{basic}	Rub_{dry}	Rub_{wet}	*LF*
Conv./Amylase	3–4	4	3–4	2–3	2-3	4
Ultra/Amylase	4	4	4	3-4	3-4	4-5
Conv./Cellulase	4	4	3–4	3-4	3-4	4
Ultra/Cellulase	4-5	4	4	4	3-4	5

Note: *WF* is wash fastness; *LF* – light fastness

Table 5

FASTNESS PROPERTIES OF DYED SILK FABRICS UNDER CONVENTIONAL AND ULTRASONIC CONDITIONS OF PROTEASE ENZYME AND BIXA DYE

Dyeing methods	Wash – perspiration – rubbing – light					
	WF	Per_{acidic}	Per_{basic}	Rub_{dry}	Rub_{wet}	*LF*
Conventional	4	4	3 - 4	3 - 4	3 - 4	4
Ultrasonic	4 - 5	4 - 5	4 - 5	5	5	5

Note: *WF* is wash fastness; *LF* – light fastness

Table 6

FASTNESS PROPERTIES OF DYED WOOL FABRICS UNDER CONVENTIONAL AND ULTRASONIC CONDITIONS OF PROTEASE ENZYME AND BIXA DYE

Dyeing methods	Wash – perspiration – rubbing – light					
	WF	Per_{acidic}	Per_{basic}	Rub_{dry}	Rub_{wet}	*LF*
Conventional	4	4	4	3 - 4	3 - 4	4
Ultrasonic	5	4 - 5	4	3 - 4	3 - 4	4

Note: *WF* is wash fastness; *LF* – light fastness

good fastness properties. The color strength (*K/S* values) are good particularly cotton by cellulase enzyme treatment and wool & silk by protease treatment. The cotton fabrics dyed in one step process have an improved fastness to washing, alkaline perspiration, dry rubbing and light. In the case of materials dyed in the presence of cellulase, the fastness properties showed an improvement of one scale in light fastness than the cotton fabrics dyed in presence of amylase. The wool and silk fabrics show better results than cotton due to the high affinity of polar carotenoid dyes with protein structures. The dye has good scope in the commercial dyeing of cotton silk, wool fabric for garment industry. Work is in progress so that this dye can be popularized as a cheap source of natural dye. The use of enzyme in place of metal mordant has been proposed as a very feasible ecologically safe methodology for the dyeing industry.

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INDUSTRIA TEXTILĂ ÎN LUME

TREVIRA MOISTURE CONTROL – O NOUĂ FIBRĂ PENTRU ÎMBRĂCĂMINTE

Catastrofele climatice din ultima vreme au cauzat un deficit în furnizarea fibrelor naturale, ceea ce a creat dificultăți mai ales pe piața lânii, aflată în continuă ascensiune. Aceşti factori au adus, din nou, în prim-plan fibrele artificiale şi, în special, poliesterul. Venind în întâmpinarea acestei tendințe, producătorul de fibre ***Trevira*** a dezvoltat o nouă fibră destinată confecționării îmbrăcămintei de înaltă calitate.

Noua fibră, *Trevira Moisture Control,* este reciclabilă şi oferă purtătorilor de îmbrăcăminte, realizată din ea, un confort sporit în purtare. Țesăturile obținute din această fibră prezintă caracteristicile specifice textilelor Trevira, inclusiv proprietăți foarte bune antipiling, rezistență a culorii, rezilință şi întreținere uşoară. În plus, noile fibre conferă un aspect semimat plăcut, un tuşeu moale şi un management optim al umidității.

Cercetătorii de la ***Institutul Hohenstein*** au confirmat faptul că un costum realizat din 100% Trevira Moisture Control prezintă caracteristici de aspect şi confort identice cu cele ale unui costum clasic, realizat din lână 100%.

Sursa: *wwwtextile-network.com*

Materii prime

NOI DEZVOLTĂRI ÎN DOMENIUL NANOFIBRELOR

Oamenii de ştiinţă de la ***Universitatea din Bayreuth*** şi ***RWTH*** – Aachen, Germania, au elaborat o metodă nouă de producere a nanofibrelor pentru medii filtrante, fără costuri prea mari.
Cu ocazia celei de-a 51-a ediţii a *Conferinţei Fibrelor Artificiale,* desfăşurate la Dornbirn – Austria, în perioada 19 – 21 septembrie 2012, dr. Hans-Werner Schmidt arăta că autoasamblarea supramoleculară a unor compuşi moleculari cu greutate redusă reprezintă cheia acestei inovaţii. Spre deosebire de tehnicile utilizate până în prezent, cum ar fi filarea din topitură şi electrofilarea, acest proces este foarte rapid şi simplu şi poate fi uşor extins cu uşurinţă pentru producţia industrială. Principiul chimiei supramoleculare este preluat din natură, unde există multe exemple de aplicaţii ale proceselor structurale şi ale mecanismelor de reacţie, în scopul organizării unui plan foarte complex, cu eficienţă ridicată. Un exemplu îl constituie structura dublu helicală a acizilor nucleici care formează acidul dezoxiribonucleic (ADN). Structura şi forma sa rezultă din interacţiunile supramoleculare. În mod similar, este posibilă obţinerea unor nanoobiecte constând din structuri supramoleculare, având controlul moleculelor componente, pe baza unor interacţiuni fizice, cum ar fi legăturile de hidrogen, într-un lichid.
În condiţii simlare, poate fi obţinută o reţea de nanofibre tridimensionale în solvent. Prin scufundarea unui mediu filtrant nețesut într-o soluţie de 1,3,5 - benzoltrisamiden este posibilă „umplerea“ acestuia cu nanofibre. Nanofibrele sunt perfect integrate în interiorul ţesăturii, fără să migreze în aer. Rezultatul este obţinerea unui nețesut filtrant din nanofibre foarte omogen. S-a constatat că nanofibrele din acest mediu posedă o rezistenţă mecanică foarte ridicată, dar şi o greutate redusă, ceea ce ar putea îmbunătăţi în mod semnificativ eficienţa filtrelor realizate din ele.
Experimentările în domeniul acestei noi tehnologii vor continua, întrucât se doreşte studierea posibilităţii folosirii unor solvenţi pe bază de apă.

Smart*textiles and nanotechnology, noiembrie 2012, p. 4*

Noi tehnologii

TEHNOLOGII ANTIMICROBIENE PE BAZĂ DE ARGINT

Compania producătoare de materiale termoplastice ***RTP Co***, cu sediul în Winona/Minnesota, S.U.A., şi-a extins linia de producţie a compuşilor antimicrobieni şi aditivilor, pentru a include noile produse bazate pe tehnologia *BactiBlock*, realizată de compania ***NanoBioMatters,*** cu sediul în Valencia, Spania.
Testele de eficacitate au dovedit că BactiBlock – un aditiv antimicrobian pe bază de argilă funcţionalizată cu argint, obţinută din surse naturale – este compatibil cu o varietate de sisteme de răşini termoplastice, inclusiv ABS, nylon 6 şi 6/6, polipropilenă, polietilenă, elastomeri termoplastici olefinici şi alcool etilvinilic. Este în curs de desfăşurare şi evaluarea altor tipuri de polimeri.
În aces sens, Jean Sirois – director general al Diviziei de Coloranţi a RTP Co a declarat: *„Aditivii şi compuşii obişnuiţi, realizaţi pe baza tehnologiei BactiBlock, se adaugă sortimentului existent de produse antimicrobiene, care, datorită transportatorului său unic din argilă, oferă un sistem de livrare foarte eficient şi durabil pentru ionii de argint, în scopul asigurării unei protecţii eficiente împotriva degradării a articolelor din material plastic“.* BactiBlock este foarte economic în comparaţie cu alte soluţii antimicrobiene pe bază de argint.
Produsele în care sunt încorporaţi aditivi BactiBlock sunt disponibile la nivel global, fiind produse în reţeaua RTP din toată lumea, cu facilităţi de producţie în America de Nord, Europa şi Asia. Aceste produse pot fi vopsite integral şi sunt uşor de dispersat şi de procesat la temperaturile normale ale polimerilor. Nivelurile de încărcare variază în funcţie de cerere şi pot fi adaptate mediilor finale, pentru a menţine eficienţa dorită. Tehnologia BactiBlock este înregistrată la Agenţia de Protecţie a Mediului, EPA, din S.U.A.
Cererea de soluţii antimicrobiene pe bază de argint, cum ar fi BactiBlock, este în creştere, atât datorită eficienţei lor în protejarea polimerilor împotriva degradării, cât şi siguranţei la contactul cu corpul uman. Aditivii antimicrobieni pot fi folosiţi pentru a combate dezvoltarea microbilor, bacteriilor, ciupercilor/mucegaiurilor şi algelor, care, lăsate necontrolate, pot provoca decolorarea sau degradarea polimerilor.
Aditivii antimicrobieni pot fi încorporaţi – prin turnare, injecţie sau extrudare – în produse destinate unor domenii diverse, precum: mobilier de exterior, echipamente pentru sport şi recreere, asistenţă medicală, echipamente de birou, bunuri de uz casnic, articole de îngrijire personală etc.

Smart*textiles and nanotechnology, august 2012, p. 8*

TRATAMENT DE SUPRAFAŢĂ APLICAT POLIOLEFINELOR

O demonstraţie de succes a unei tehnologii emergente a deschis calea spre un nou tratament aplicat fibrelor sensibile la căldură, de tipul poliolefinelor.
Noua tehnologie de tratare reactivă a suprafeţei, elaborată de ***Alexium International Group Ltd.***, din

Perth/ Australia, poate fi aplicată fibrelor poliolefinice, pentru a le conferi o serie de funcţii, cum ar fi proprietăţi antimicrobiene şi ignifuge, sau pentru a spori aderenţa materiei prime.
Piaţa europeană a produselor textile poliolefinice este estimată la o valoare anuală de 2,5 miliarde de dolari S.U.A., iar principalele aplicaţii ale acestora sunt textilele tehnice şi covoarele. Cu toate acestea, extinderea domeniilor de utilizare a acestora este îngreunată de o barieră tehnică reprezentată de incapacitatea de a conferi funcţionalitate suprafeţei acestor fibre inerte. De exemplu, aderenţa scăzută între fibre şi răşina din matricea materialelor compozite reprezintă o cauză curentă de defecţiune mecanică. Tratamentele economice aplicate în scopul îmbunătăţirii noii tehnologii de tratare a suprafeţei se bazează mai degrabă pe energia furnizată de microunde, decât pe căldură, iar faptul că fibrele poliolefinice sunt transparente face ca acestea să nu fie direct încălzite în timpul tratamentului (fig. 1).

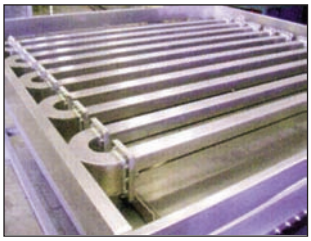

Fig. 1

După ce a demonstrat faptul că peliculizarea aplicată pe suprafaţa fibrei nu provoacă degradarea termică a acesteia, compania s-a implicat în dezvoltarea unor proiecte comerciale pilot privind aplicarea de substanţele antimicrobiene, promotori de aderenţă sau agenţi FR.
În acest sens, directorul Departamentului de Tehnologia Informaţiei, Bob Brookings, a declarat: „*Noi anticipam faptul că noul procedeu RST va oferi o gamă complet nouă de aplicaţii ale acestor ţesături tehnice*“.
Stefan Susta, director al Departamentului de Operaţiuni şi director executiv al companiei, a adăugat: *„Alegem cu atenţie obiectivele şi lucrăm cu diverşi lideri de piaţă în aplicarea unor peliculizări, ce au drept scop extinderea domeniilor de utilizare a ţesăturilor poliolefinice şi adăugarea unor valori noi domeniilor deja existente“.*

Technical Textiles International, octombrie 2012, p. 7

Nanotehnologii

PROIECTUL *TECHNOTOX* DESPRE COMPORTAMENTUL NANO

În prezent, nanoparticulele sunt utilizate pentru a crea produse cu funcţionalităţi noi, de exemplu materiale care resping praful sau materiale cu efect antibacterian. Ca în cazul oricărei tehnologii noi, şi aceasta trebuie să prezinte siguranţă. De aceea, atât pentru producători, cât şi pentru utilizatori, este important ca nanoparticulele, pe parcursul întregului lor ciclu de viaţă, să fie inofensive pentru oameni şi mediu.
În decembrie 2010, proiectul de cercetare *TechnoTox* a fost lansat, în Germania, de ***ITV Denkendorf***, ***Institutul Hohenstein pentru Inovaţie Textilă*** şi diverse companii industriale, cu scopul de a realiza o evaluare a situaţiilor de risc a nanoproduselor textile. Proiectul este sponsorizat de statul german din Baden – Württemberg.
În anul 2011, Consiliul German al Mediului (SRU) a publicat următoarea declaraţie despre utilizarea nanomaterialelor: *„Până în prezent nu s-a demonstrat ştiinţific că nanomaterialele, aşa cum sunt produse şi utilizate astăzi, ar conduce la distrugerea mediului şi a sănătăţii“.*
Totuşi, deoarece se prognozează o extindere a domeniilor de utilizare a textilelor nanofuncţionalizate, aceste noi posibile utilizări nefiind încă luate în considerare în evaluările anterioare ale riscului, se impune folosirea unor metode de testare eficientă a efectelor nanomaterialelor şi a proprietăţilor lor toxicologice.
În proiectul TechnoTox sunt prezentate date privind comportamentul, retenţia şi efectul biologic al materialelor realizate din nanofibre în funcţie de condiţiile de mediu şi este realizată o evaluare a riscului în situaţiile date. Pe parcursul desfăşurării proiectului, au fost dezvoltate noi metode de analiză a potenţialului de risc uman şi ecotoxicologic al nanoparticulelor utilizate în diverse medii relevante.
Toţi participanţii la proiect doresc să-şi promoveze realizările în domeniul nanotextilelor şi să demonstreze siguranţa utilizării acestora dintr-o perspectivă de risc bazată pe o metodologie de testare complementară.
Abordarea este bazată pe soluţii complementare, în care studiile cu privire la expunerea materialelor fizice sunt cuplate direct cu studii biologice asupra efectelor utilizării acestora. Rezultatele studiilor sunt grupate şi evaluate pentru orice corespondenţă de efecte. Determinarea proprietăţilor particulelor şi a efectele lor asupra produselor realizate asigură o evaluare exhaustivă a riscurilor materialelor din fibre nanofuncţionalizate destinate bunurilor de larg consum.

Smart*textiles and nanotechnology, august 2012, p. 8*

NANOFIBRE COMPOZITE UTILIZATE ÎN MEDICINA REGENERATIVĂ

Mijloacele bioinginereşti de înlocuire a tendoanelor, ligamentelor, meniscului genunchiului şi a altor ţesuturi necesită refacerea arhitecturii tridimensionale a acestor ţesuturi.
Ţesuturile fibroase pe bază de colagen din corpul uman au o structură ordonată, care le conferă robusteţe, pentru a putea suporta o sarcină mecanică extremă. Tratamentul conservator aplicat în cazul

rupturilor de ligament încrucişat anterior şi de menisc ale genunchiului, al leziunilor centurii scapulare şi al rupturilor tendonului lui Achile implică utilizarea unor suporturi realizate din nanofibre, care pot ghida țesutul astfel încât acesta să se dezvolte într-o structură ordonată. Din păcate, utilizarea pe scară largă a acestor fibre în ortopedie a fost redusă, din cauza faptului că, atunci când fibrele sunt prea strâns asamblate, procesul de colonizare a suportului cu celule este îngreunat.

În prezent, în cadrul ***Universității din Pennsylvania***, din Philadelphia, este dezvoltată şi aplicată o nouă tehnologie, în care suporturile din nanofibre compozite oferă o structură suficient de flexibilă pentru a coloniza celulele fără niciun fel de impediment, putând crea astfel un nou țesut.

„Aceste nanofibre sunt fibre mici, cu *un potențial imens*, care *poate fi dezvoltat prin includerea unui element temporar*, *cu menținerea aceluiaşi spațiu*“ – afirmă Dr. Robert L. Mauck, profesor de chirurgie ortopedică şi bioinginerie la Şcoala de Medicină Perelman, de la Universitatea din Pennsylvania.

Echipa de cercetători a realizat, prin electrofilare, compozite care conțin două tipuri de fibre – un polimer cu degradare lentă şi un polimer solubil în apă, acestea putând fi eliminate selectiv, pentru a mări sau pentru a micşora spațiul dintre fibre. Fibrele sunt obținute pe baza unor soluții de încărcare electrică a polimerilor dizolvați. Soluțiile se agită pentru a ține ca un spray fin de fibre, care cad, precum zăpada, pe un tambur rotativ şi, apoi, se reunesc formând o țesătură elastică.

Materialul textil astfel obținut poate fi utilizat în diverse aplicații medicale, fiind colonizat ulterior cu celule sau implantat direct – ca un plasture – în țesutul afectat.

Creşterea procentului de fibre de dizolvare sporeşte capacitatea celulei-gazdă de a coloniza plasa de nanofibre şi, în final, formează un adevărat țesut tridimensional. În ciuda eliminării a peste 50% din fibrele inițiale, restul suportului are o arhitectură suficient de rezistentă pentru a alinia celulele şi a forma o matrice extracelulară, extrem de organizată, datorită prezenței celulelor producătoare de colagen. Acest lucru a condus, la rândul său, la realizarea unui material biologic cu proprietăți de tracțiune similare, din punct de vedere al mecanicii țesutului, cu cele ale țesutului ce formează meniscul genunchiului.

„Această nouă abordare transformă ceea ce a reprezentat odată un fenomen interesant în domeniul biomaterialelor – celulele de pe suprafața straturilor de nanofibre – într-o metodă prin care se pot forma țesuturi funcționale, tridimensionale“ – afirma Dr. Robert L. Mauck.

Realizarea noilor materiale constituie un pas înainte în prelucrarea țesuturilor fibroase portante, care îşi vor găsi aplicații, pe scară largă, în medicina regenerativă.

În prezent, Dr. Robert L. Mauck şi echipa sa testează aceste materiale noi pe un model de animal mare, pentru repararea meniscului şi alte aplicații ortopedice.

Smarttextiles and nanotechnology, septembrie 2012, p. 7

Modă

NOI SORTIMENTE DE CIORAPI PENTRU SEZONUL DE TOAMNĂ

Pentru zilele răcoroase de toamnă a fost creat un nou sortiment de ciorapi-pantalon, cu o notă florală, în stil Art Déco.

Acest ciorap elegant este realizat pe un fond al structurii tricotului cu deschideri relativ mari şi cu un model floral din trandafiri (fig. 1). Dispuse la distanță mică între ele, florile se împletesc una în jurul celeilalte formând lujeri care înfăşoară delicat piciorul – un detaliu care reține privirea şi al cărui efect este intensificat prin combinația de culori. Un maro cu o nuanță de lila a fost combinat cu un galben pal, creând un efect contrastant de culoare.

Pentru a susține modelul floral curgător, dar mai ales pentru a conferi un confort sporit în purtare, acest articol de îmbrăcăminte atrăgător a fost creat fără cusătură, într-o singură piesă – de la vârf până la linia taliei.

Fig. 1

Ciorapul-pantalon a fost realizat pe maşini de tricotat tip raşel, cu două fonturi, de la Karl Mayer. Maşina a fost reglată pentru finețea E 24 – în tehnica offset, dar care creează o finețe E 12 – cu ochiul liber.

Pentru structura de fond s-a folosit o poliamidă texturată, iar pentru liniile modelului un fir filat cu miez. Acest fir este realizat din două componente – un miez din elastan şi un înveliş din poliester sau poliamidă.

Combinația inspirată de fire conferă articolelor de ciorapi tricotați din urzeală un tuşeu moale şi călduros şi asigură o libertate optimă de mişcare, dar şi o bună aderență a ciorapului la picior.

Maşinile de tricotat din seria DJ şi RDPJ, cu sistem automat de acționare a barei cu pasete şi de control Multispeed KAMCOS® sunt perfecte pentru producerea de articole fără cusătură, mulate pe corp.

Pentru obținerea unui aspect elegant, pe lângă marea varietate de modele, se pot alege şi diferite desimi ale ochiurilor. Alături de proiectarea detaliată a tiparelor, acest lucru este important pentru a asigura o potrivire perfectă a articolului cu forma corpului.

Kettenwirk Textilinformationen Praxis, martie 2012, p.18