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Sumar

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Effect of hydrothermal processing on the structure and properties of wool fibers

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XING TAO ZHANG

REZUMAT – ABSTRACT

Efectul prelucrării hidrotermice asupra structurii și proprietăților fibrelor de lână

În lucrare sunt studiate fibrele de lână tratate cu apă fierbinte sub presiune, prin metoda hidrotermică. Sunt analizate efectele timpului de reacție, la diverse temperaturi, asupra unor proprietăți ale fibrelor de lână, cum ar fi: tensiunea, frecarea, ondularea, indicele de galben și indicele de alb. Schimbarea structurală este investigată prin microscopie electronică de scanare cu emisie în câmp, spectroscopie în infraroșu cu transformată Fourier, analiză termogravimetrică și difracție de raze X. Rezultatele arată că, în urma tratamentului hidrotermic, apare o oarecare micșorare a valorilor tenacității și alungirii la rupere ale fibrelor de lână. De asemenea, se constată o reducere a eficienței frecării statice și a celei cinetice. Numărul de ondulări crește ușor, însă gradul de ondulare, procentajul de recuperare a ondulării și cel de ondulație reziduală scad. Indicele de galben crește, în timp ce indicele de alb scade. Procesul hidrotermic are un efect redus asupra stabilității termice, morfologiei şi structurii fibrelor de lână. Gradul de cristalinitate și gradul de orientare scad.

Cuvinte-cheie: fibre de lână, prelucrare hidrotermică, structură, proprietate

Effect of hydrothermal processing on the structure and properties of wool fibres

In this paper, wool fibres are treated with hot pressure water by a hydrothermal method. The effects of reaction time at different temperatures on the properties of wool fibres like tension, friction, crimp, yellowness, and whiteness are studied. The structural change is characterized by field emission scanning electron microscopy, Fourier transform infrared spectroscopy, thermal gravimetric analysis, and X-ray diffraction techniques. The results indicate that after hydrothermal treatment, the tenacity and elongation at break of wool fibres decrease to some extent. Both static and kinetic friction effectiveness decrease. The crimp number slightly increases, but the crimp percentage, crimp recovery rate, and residual percentage crimp decrease. The yellowness index increases, whereas the whiteness index decreases. The hydrothermal process has a little effect on the morphology, structure, and thermal stability of wool fibres. The degree of crystallinity and degree of orientation decrease.

Key-words: wool fibres, hydrothermal processing, structure, property

Many different approaches, such as sol-gel, in-suit synthesis, sputtering, and ion change, have been used for the modification of textile fibres or fabrics. Obviously, the aforementioned methods for stabilization of nanoparticles on the textiles are not prone to withstand repetitive washing. In fact, some essential steps like functional treatment, neutralization, washing, drying, curing, and final treatment are involved. These processes are not only hazardous for the environment but also costly and time-consuming owning to the incorporation of many chemicals. Moreover, most of them result in a series of deteriorations in colour, softness, abrasion resistance, and mechanical properties of textiles [1], [2]. In recent years, in order to endow textile materials with multifunctional properties such as self-cleaning, wettability, UV protection, and antimicrobial action, titanium dioxide has been extensively utilized in the textile industry due to its high photo-catalytic activity, biocompatibility, non-toxicity, and physicochemical stability [3], [4].

The hydrothermal technique has been employed as a unique method to produce the functional materials because of the adaptability, homogeneity, and environmentally benign property, particularly for deposition of various compounds on metals, polymers, ceramics etc. [5]. The chemical modification of wool fibres is usually carried out at a relatively low temperature (< 90°C), or at high temperature for short time (> 100°C). It has been reported that wool fibres are treated with pure water for various periods of time at specified temperatures. The chemical changes associated with the degradation of wool keratin have been systematically studied, including thiol and disulphide contents, sulphur contents, lanthionine contents of hydrolyzates, "bromine-oxidizable sulphur" contents of hydrolyzates, "labile sulphur" contents, amino-acid analysis, nitrogen contents, and yellowness indices [6], [7]. It is proven that the hydrothermal treatment causes the hydrolysis of some peptide bonds in wool fibres. The hydrolysis rate of peptide bonds increases with the increase of the treatment temperature and time. Furthermore, the crystal residues in wool can be converted to lanthionyl residues. Some reactive sulphur-containing residues are also formed. It has been confirmed that after treatments of long duration or at high temperatures, some peptides, e.g. lysyl, aspartyl, prolyl, threonyl, seryl, and so on, are progressively hydrolysed to lower molecular-weight peptides, free amino acids, and other degradation products [8]. A continued heating will lead to the degradation of wool keratin, which becomes more severe with increasing water content, and a concomitant weakening of the fibres [9], [10]. It is worth pointing out that the presence of internal lipids can significantly reduce the damage to fibre cells during hydrothermal treatments [11]. Also, the fibre surface is modified both chemically and morphologically by treatment with water at elevated temperatures [12]. Additionally, the colour change of wool fibres can be minimized by reducing the entry regain of the wool and limiting the period for which the wool is associated with boiling water [13].

In our early work, wool fabric has been modified with tetrabutyl titanate under hydrothermal conditions, and then dyed using C.I. Reactive Blue 69 with an aim to improve the anti-felting ability and colour fastness [14]. However, the processing conditions used are different from the previous studies and the properties of friction, crimp, thermal stability, and orientation of wool fibres are seldom discussed. Therefore, in this study, the effect of hot pressure water on the structure and properties of wool fibres need to be elucidated. The hydrothermal processing has been optimized so that the degradation of wool fibres does not reach an unacceptable level.

EXPERIMENTAL PART

Materials and chemicals

The merino wool fibres with a spinning count of 66 s, as well as nonionic surfactant W 900, were obtained from the local textile mill. The linear density of wool fibres was 3.2 ± 0.1 dtex and the average diameter was about 20 μm. The reagent-grade chemicals, such as sodium carbonate, acetone, and anhydrous ethanol, were used as received. Deionized water was used throughout this experiment.

Hydrothermal treatment of wool fibres

About 3.0 g of raw wool fibres was scoured in 200 ml of mixed solution containing 2.0 g/l of sodium carbonate and 0.5% of nonionic surfactant W900 at 50°C for 15 minutes, and successively degreased with a 100 ml of acetone and anhydrous ethanol solution at 50°C for 10 minutes, respectively, and then washed thrice in deionized water at 40°C for 10 minutes and dried at 60°C for 8 hours. The pre-treated wool fibres were directly added into 80 ml of deionized water at room temperature, and then transferred to a PTFE sealed can with a capacity of 100 ml, which was put into the stainless steel autoclave. The autoclave was placed in a furnace and run at a speed of 60 rotations/minute. The temperatures were raised to 100°C, 110°C, 120°C, and 130°C at a heating rate of 2.5°C/minute, respectively. After a certain time of 1 hour, 2 hours and 3 hours, the treated wool fibres were taken out and subsequently washed with the acetone, ethanol, and deionized water at 50°C for 10 minutes, respectively, and finally dried at room temperature.

Testing method

The tensile properties of fibre samples were measured on an YG001N electromechanical test instrument in the reference with GB/T 13835.5-2009. The gauge length was 10 mm and the constant extension rate was 10 mm/minute. The pretension was 0.1 cN. More than 300 wool fibres were measured to ensure a 95% confidence level. The friction properties of fibre samples were evaluated using an Y151 single fibre friction instrument based on the capstan method. The coefficients of static and kinetic friction (a fibre over a metal rod) were calculated [15]. The crimp properties of fibre samples were assessed on an YG362B fibre crimp elasticity tester in accordance with GB/T 13835.9-2009. The average value of 50 tests was reported for the friction and crimp properties, respectively. The volume density of wool fibre samples was tested using a ZMD-2 automated digital density meter according to GB/T 1463-2005. The average value of ten tests was reported. The whiteness and yellowness values of fibre samples were evaluated using a Datacolor SF300 Plus colorimeter (D65/10°) according to ASTM E-313. All tests were carried out at a temperature of 20°C and a relative humidity of 67%.

Characterization

The surface morphologies of wool fibres before and after treatments were observed using field emission scanning electron microscope (FESEM, JEOL JSM-6700). Fourier transform infrared spectroscopy (FT-IR) measurements were conducted using a FT-IR 7600 spectrophotometer (Lambda Scientific Systems, Inc.). The spectra were recorded in the range of 400–4 000 cm^{-1} with a resolution of 4 cm^{-1} as the KBr pellets. The thermogravimetric and differential thermaler. The percentage weight change with respect to temperature was evaluated from 40°C to 600°C with a 10°C/minute heating rate under nitrogen atmosphere. The X-ray diffraction (XRD) patterns of fibre samples were obtained by using Cu $K\alpha_1$ radiation (λ = 0.154056 nm), using a 7 000 S diffractometer at 40 kV and 40 mA with the angle of 2 θ from 5° to 80° at a scan speed of 8 deg/minute. According to Segal's empirical method [15], the degree of crystallinity of wool fibres was calculated by equation (1).

$$Crystallinity = \frac{I_{9^\circ} - I_{14^\circ}}{I_{9^\circ}} \times 100\% \qquad (1)$$

where:

I_{9° is the maximum intensity of diffraction at 2 θ = 9°;

I_{14° – the intensity of the amorphous diffraction at 2 θ = 14°.

The degree of orientation *y* of fibre samples was also measured using the above instrument (40 kV, 30 mA) in the range of 0–360° at a scanning speed of 60 deg/minute, and was calculated by equation (2).

$$y = \frac{360 - \sum h_i}{360} \quad (2)$$

where:

h_i is the full width at half maximum (FWHM) of the *i* the diffraction peak.

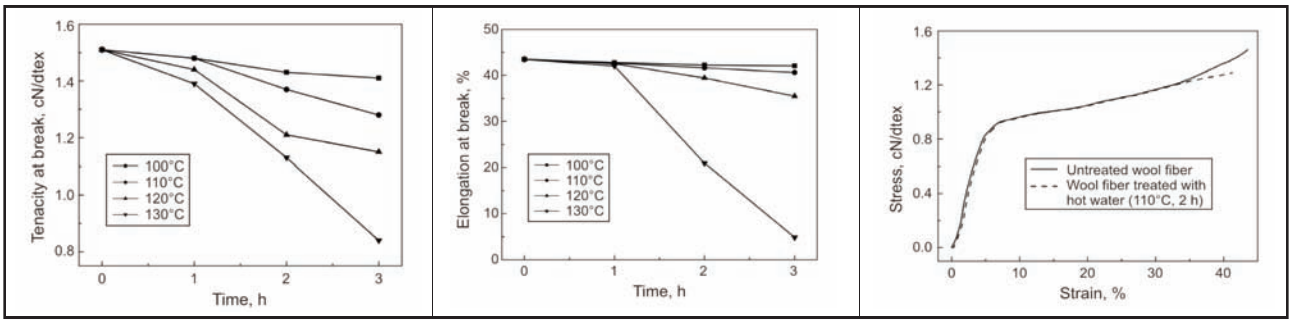
RESULTS AND DISCUSSIONS

Tensile properties

The results of tensile tests of wool fibres before and after treatments are exhibited in figure 1. It is seen that after hydrothermal process the tenacity at break (coefficient of variation from 27.6% to 36.5%) of wool fibres decreases with increasing reaction time. The corresponding elongation at break (coefficient of variation from 30.5% to 37.8%) also follows the same rule. Obviously, the high temperature results from the sharp reductions of tenacity and elongation at break of wool fibres. The hydrothermal treatment causes the hydrolysis of some peptide bonds (the fission of the C-N bonds amide groups of some glutaminyl and asparaginyl residues) in wool, which results in the decrease of the breaking load of a single fibre. The proportion of peptide bonds in wool made dialyzable by water treatment markedly increases with increasing treatment temperature and time [6]. It is also noted that the typical stress-strain curves are almost overlapped for the untreated and treated wool fibres. However, the tenacity and elongation at break of wool fibres are reduced to some degree. The hydrothermal treatment has little effect on Young's modulus (the slope of the stress-strain curve in the low strain region). The reason is due to the fact that maintaining wool in the presence of hot water allows the formation of a network of secondary bonds and a greater force is required to extend the fibre. In addition, the aggregation and cleavage of the main chain of the proteins contribute to the tensile properties of wool fibres [10]. If wool fibres are extended in water, the stress-strain characteristics are certainly related to any degradation, so that such fibres exhibit a weakened Hookean region [9].

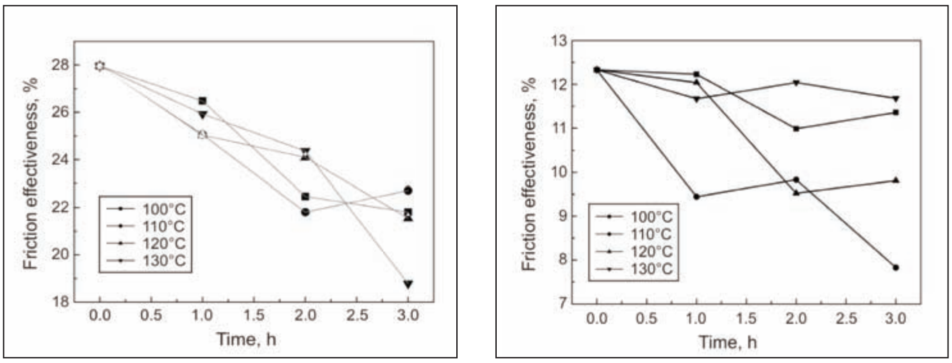
Friction properties

The effects of reaction time at different temperatures on the static and kinetic friction effectiveness of wool fibres are revealed in figure 2. It is found that the static and kinetic friction effectiveness of the treated wool fibres decreases to some extent after hydrothermal treatment in comparison with the untreated samples. As the reaction time increases, the friction effectiveness for both static and kinetic tests generally decreases. The influence of reaction time on the static friction is more significant than that on the kinetic friction. After wool fibres are treated with hot pressure water, the degree of scale protrusion of wool fibres is enhanced. The edge of scale may be damaged with the increase of the reaction time, and become rough. The static and kinetic friction coefficients against –



a *b* *c*

Fig. 1. Effects of reaction time at different temperatures on:
a – tenacity; *b* – elongation at break; *c* – stress-strain curves of wool fibers



a *b*

Fig. 2. Effects of reaction time at different temperatures on the:
a – static friction effects of wool fibers; *b* – kinetic friction effects of wool fibers

and with – scale directions increase to some extent. Thus the friction effectiveness decreases. The effect of reaction temperature on the kinetic friction is more prominent with respect to the static friction. The change of the friction behaviour is attributed to the morphological changes of wool fibres [11]. Moreover, the removal of surface-bound lipid makes an impact on the chemical nature of the fibre surfaces [12]. Considering the properties of tension and friction of wool fibres, the optimum reaction temperature is set at 110°C and the reaction time is 2 hours.

Crimp properties

The hot pressure water induces the change of structural conformation of relaxed wool fibres. The results of crimp tests of wool fibres before and after treatments (110°C, 2 hours) are compared in table 1. After hydrothermal treatment, the crimp number of wool fibre increases slightly from 7.0 to 7.5 per 25 cm. However, the crimp percentage, crimp recovery rate, and residual percentage crimp decrease to some degree. The crimp properties of wool fibre are hence affected by the hydrothermal treatment, but not too seriously. This is because the water at higher temperature can enhance the relaxation processes within the wool fibres [9].

Table 1

THE RESULTS OF CRIMP PROPERTIES OF WOOL FIBERS BEFORE AND AFTER TREATMENTS

Wool fibers	Crimp number, 25 cm	Crimp percentage, %	Crimp recovery rate, %	Residual percentage crimp, %
Untreated	7.0	6.76	96.3	6.61
Treated (110°C, 2 hours)	7.5	4.22	92.4	3.95

Density, yellowness, and whiteness indices

The experimental results show that after treatment with hot pressure water at 110°C for 2 hours, the volume density of wool fibres does not change and is kept at 1.31 g/cm^3. However, the yellowness index increases distinctly from 16.6 to 30.1. The whiteness index of the untreated wool fibre is 0.38 while it is –0.01 for the treated wool fibre. The results confirm other studies on the hydrothermal yellowing of wool fabric [13]. The yellowing of wool fibres is associated with the degradation of crystal residues, perhaps coupled with the hydrolysis of certain peptide bonds [6].

SEM

The SEM images of wool fibres before and after treatments are shown in figure 3. It is clear that the surface of the untreated wool fibre is very clean. The scales having the sharp edges protrude outward from the body. After treatment with hot water at high pressure (110°C, 2 hours), the appearance of wool fibre changes slightly. Some deposition can be found, which is ascribed to the amorphous material extracted from wool fibres. The fringe part of wool scale is destroyed to a slightly lesser extent. The outer edge of the scale becomes irregular. This is due to the fact that treatment of wool fibres in boiling water results in the hydrolytic damage to the cuticle or cortical cell membrane complex, the loss of surface integrity, and an increase in cuticle cell thickness [11].

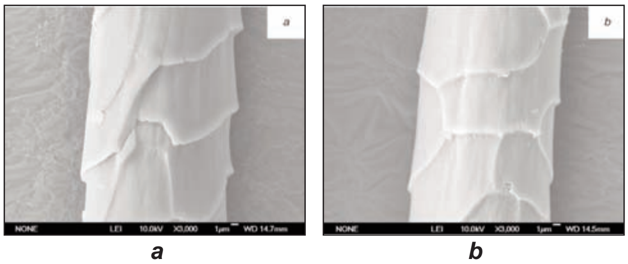


Fig. 3. SEM images of wool fibers:
a – before treatments; *b* – after treatments (110°C, 2 hours)

FT-IR

The FT-IR spectra of wool fibres before and after treatments are represented in figure 4. After treatment, the N-H band of wool fibres decreases from 3 430 cm^{-1} to 3 425 cm^{-1}. This is attributed to the absorbed water caused by hydrothermal process. The peaks at 2 932 cm^{-1} (CH_2 asymmetric stretching) and 2 877 cm^{-1} (CH_3 symmetric stretching) are reduced to 2 927 cm^{-1} and 2 875 cm^{-1}, respectively. The amide I band is shifted from 1 636 cm^{-1} (C=O stretching) to 1 634 cm^{-1}. The peaks at 1 126 cm^{-1} (C-O stretching) and 568 cm^{-1} (N-H deformation) increase to 1 129 cm^{-1} and 588 cm^{-1}, respectively. Furthermore, the disulfide bond centred at 526 cm^{-1} disappears. However, the other bands occurred at 1 708 cm^{-1} (carbonyl group), 1 509 cm^{-1} (amide II, N-H bending), 1 450 cm^{-1} (CH_3 asymmetric bending), 1 388 cm^{-1} (CH_3 deformation), 1 234 cm^{-1} (amide III, C-N stretching), 1 076 cm^{-1} (C-O stretching), and 933 cm^{-1} (C-O stretching) have no obvious change [17]. Hence, it is concluded that the molecules of hot water can penetrate the interior of wool fibres under high pressure conditions.

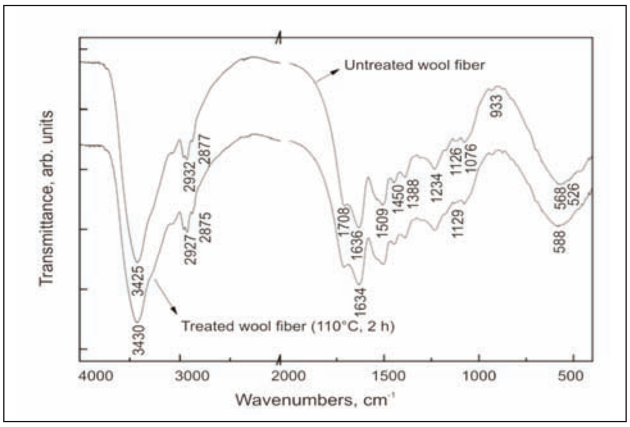
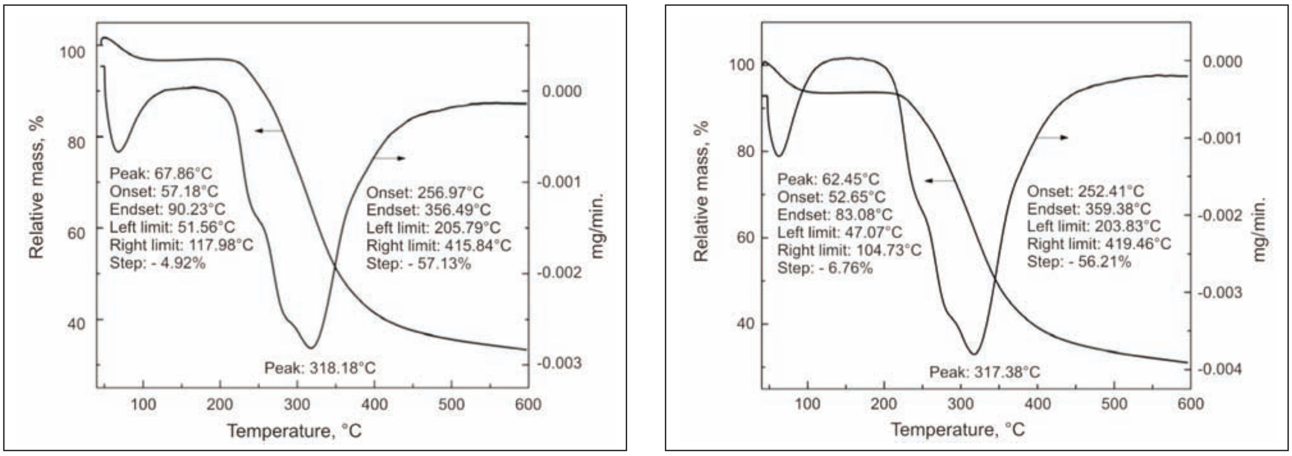


Fig. 4. FT-IR spectra of wool fibers before and after treatments



a *b*

Fig. 5. TG and DTA curves of wool fibers:
a – before treatments; *b* – after treatments (110°C, 2 hours)

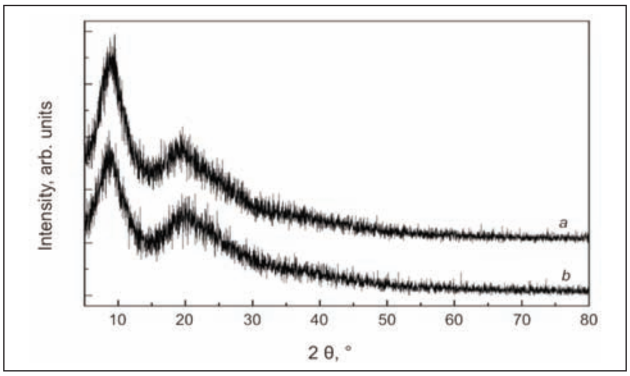


Fig. 6. X-ray patterns of wool fibers:
a – before treatments; *b* – after treatments
(110°C, 2 hours)

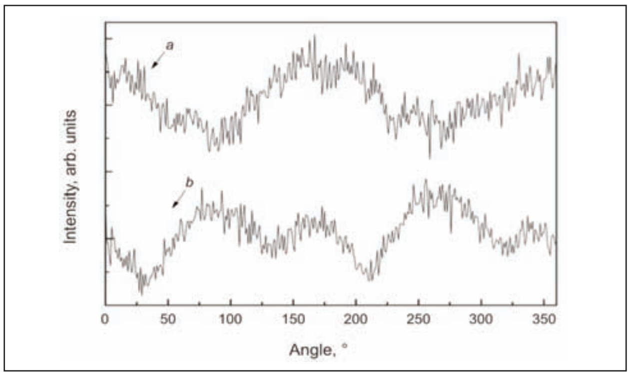


Fig. 7. Orientation curves of wool fibers:
a – before treatments; *b* – after treatments
(110°C, 2 hours)

TG/DTA

The TG/DTA curves of wool fibres before and after treatments are illustrated in figure 5. There are two stages in the thermal decomposition process for both samples. After treatment, the onset and endset decomposition temperatures for the first weight loss step are shifted from 57.2°C to 52.7°C and from 90.2°C to 83.1°C, respectively, which are ascribed to the removal of surface absorbed water or the residual water molecules. The endothermic peaks are reduced from 67.9°C to 62.4°C. The corresponding mass losses increase from 4.92% to 6.76%. The second weight loss step that appeared in 200–500°C range consists approximately in three periods. That is ascribed to the decomposition of amino acid. The main onset decomposition temperature decreases from 257.0°C to 252.4°C, while the main end set decomposition temperature increases from 356.5°C to 359.4°C. The strong endothermic peak changes slightly from 318.2°C to 317.4°C. The corresponding mass losses are reduced from 57.1% (205.8 – 415.8°C) to 56.2% (203.8 – 419.5°C). So, the hydrothermal treatment has a small effect on the thermal properties of wool fibres.

XRD

The XRD patterns of wool fibres before and after treatments are displayed in figure 6. It is noticed that the typical diffraction peaks for both samples are observed at around 2 θ = 9° and 20°, which belong to the characteristic positions of wool fibres. The degree of crystallinity is slightly reduced from 63.7% to 61.5% after hydrothermal treatment. The orientation curves of wool fibres before and after treatments are revealed in figure 7. The test results show that when wool fibres are treated with hot pressure water, the degree of orientation is significantly reduced from 69.3% to 45.8%. This is because the chemical cross-linkage mostly occurred in the amorphous region or in the surface of the crystal region is broken under the hydrothermal conditions. Parts of protein chains of wool fibres are changed from alpha-helix to beta-sheet structure [18], [19]. Meanwhile, the protein macromolecular chains deviate from the axis of wool fibre to a certain degree.

CONCLUSIONS

Wool fibres are treated with hot pressure water under hydrothermal conditions. The influences of reaction time and reaction temperature on the tension, friction, crimp, yellowness, and whiteness properties are investigated. After hydrothermal treatment, tension tests show that the tenacity and elongation at break of wool fibres decrease to some degree. Friction tests show that both static and kinetic friction effectiveness decreases. Crimp, yellowness, and whiteness measurements show that the crimp number slightly increases, but the crimp percentage, crimp recovery rate, and residual percentage crimp decrease. The yellowness index increases, but the whiteness index decreases. SEM, FT-IR, TG, and XRD results show that the hydrothermal process has a small effect on the morphology, structure, and thermal stability of wool fibres. The degree of crystallinity and degree of orientation decrease.

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Enzymatic finishing of blended textile materials from cottonised flax and cotton fibers

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REZUMAT – ABSTRACT

Finisarea enzimatică a materialelor textile, realizate din amestecuri de fibre din bumbac și in cotonizat

Dezvoltarea sustenabilă impune înlocuirea tehnologiilor poluante cu tehnologii ecologice. În lucrare sunt prezentate tratamentele enzimatice aplicate materialelor textile obținute din amestecul a două fibre celulozice, respectiv bumbac și in cotonizat. Materialele supuse proceselor de biocurățare și biolustruire prezintă proprietăți mai bune, comparativ cu mostrele din același material, tratate prin procedee clasice.

Cuvinte-cheie: biocurățare, biolustruire, țesături din amestec de in/bumbac, ultrasunete, tehnologii ecologice

Enzymatic finishing of blended textile materials from cottonised flax and cotton fibers

The sustainable development imposes the substitution of polluting technologies with ecological procedures. Thus, enzymatic treatments for textile fabrics composed of two cellulosic fibers: cotton and flax are presented. The worked-up materials obtained after bio-scouring and bio-polishing have better properties compared with the similar textile sample treated by classical chemical procedures.

Key-words: bio-scouring, bio-polishing, flax/cotton blended fabrics, ultrasounds, ecological technologies

The textile finishing is a process generating pollution [1] – [3]. A number of steps of textile materials finishing refer to wet processes. Because of the quantity and toxicity of generated [4] waste waters, a number of environmental and waste-related indicators have been developed [5].

The sustainable development imposes the replacement of classical finishing procedure with new non-polluting processes. An alternative to chemical finishing of textiles is the bio-finishing using enzymes [6], [7].

The chemical treatments with softener in ultrasound conditions have been successfully applied in our previous work for natural fibers (cottonised flax) and the bio-finishing procedures have been applied by our research group in a number of cases for natural fiber materials like: cotton, cotton/PES or cotton/flax using enzymes [8] – [16].

The previously obtained promising results have been a motivation for developing new bio-technologies for textile materials.

EXPERIMENTAL PART

Materials used

Textile fabrics from different cotton and cottonised flax blended yarns have been worked up. The composition of these materials is presented in table 1.

The enzymes used for the two finishing processes have been:

- ***Bio-scouring:***
  - A commercial product, Sera Zyme C-PE (Roglyr Eco 183), based on 5-15% Pectate Lyase (E.C.4.2.2.2), was used. The activity of the enzyme was determined [12] on polygalacturonate as substrate by the method described in literature [17] also used in a previous work [12], measuring the absorbance at 235 nm, characteristic for the CHO group. The resulted activity value was of 39 U/g commercial enzymes;
- ***Bio-polishing:***
  - Cellusoft L, a 1,4 β-endoglucanase produced by Novozym;
  - Cellulase rich in endoglucanase activity appears to be better suited for bio-finishing [17];
  - Complexing agent Heptol ESW, a phosphonate acting as sequestrant, supplied by CHT-Germany, with a special binding capacity toward metal ions, like calcium;
  - Tensioactive compound Sulfolen 148 (S-148, alkyl polyglicol ether), alkali stable, used for the pre-treatment and bleaching of cellulose fibers, supplied by Rotta GmbH;

Table 1

No.	Fabric	Composition cottonised linen/cotton, %	Weight per unit area, g/m^2	Applications
1	Cecilia I	42/58	205	Apparel industry
2	Cecilia II	40/60	235	Apparel industry
3	Dochia	35/65	140	Apparel and furniture industries

– Buffer solution 0.1 M of *p*H 4.7 prepared from 0.5 M monosodium phosphate and 0.5 M disodium phosphate;
– Sodium hydroxide, of ≥ 99% purity purchased from Merck & Co, Germany;
– Softner II 8 % [8].

Equipment used

Ultra-sound bath, type ELMA – TI-H-10, volume of 10 L, with thermostat at working frequency of 35 kHz. Washing machine Zanussi with multiprogramming, variable temperature from 20 to 90ºC, and centrifugation speed 1 500 rot./min.

The bio-scouring procedure

The textile fabrics have been treated with a water solution (liquor-to-fabric ratio 10/1 in mL/g) containing:

- Roglyr Eco 183 – 1.5% over fibers (o.w.f);
- Sulfolen 148 – 2% from total bath;
- Heptol ESW 1mL/L;
- Buffer *p*H 4.7 – 10% from total bath.

The mixture was kept in the ultrasound bath for 1 hour at 55ºC. The textile material was then separated. By treatment with water at 80-90ºC (liquor-to-fabric ratio 10/1 in mL/g) the residual enzyme was deactivated. The textile material was separated again and washed with water at 20ºC, the same ratio as before, for 10 minute. The obtained material was subjected to the bio-polishing operation.

The bio-polishing procedure

The textile fabrics were enzymatically treated for reducing the tendency to pill. The treatment conditions were the following:

- Liquor/textile material 10/1;
- Enzyme content 1.5% (o.w.f);
- Buffer solution *p*H 4.7 10% from total bath;
- Time 10 minutes;
- Temperature 55ºC.

The textile sample was then separated, washed and neutralized with a solution, liquor/textile 10/1 (mL/g) containing sodium hydroxide (1 g/L) at 80–90ºC, during 10 minutes. Finally the blended textile material (cotton/cottonised flax) was treated for 10 minutes at 20° with a solution of softener (Softener II 8% o.w.f.) [8] and dried by centrifugation and aeration.

RESULTS AND DISCUSSIONS

The replacement of the classical pollution technologies with bio-treatment using enzymes as catalyst for textile finishing constitutes a trend in the textile industry development. In this work we present the results of enzymatic technology application on a blended textile made from cotton and cottonised flax fibers. The first process performed on the cottonised flax/cotton fabrics was the bio-scouring performed by treatment with enzymes for the elimination of the undesired polysaccharides, especially pectin. These impurities are fragmented in smaller water soluble oligosaccharides, by a pectate-lyase (E.C.4.2.2.2), namely Sera Zyme C-PE (Roglyr Eco 183). The fragmentation with such enzyme proceeds by an elimination reaction (fig. 1).

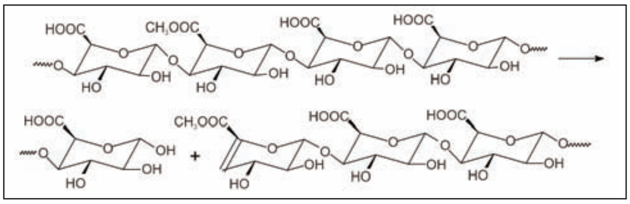

Fig. 1. Pectin fragmentation by elimination reaction

Besides the enzyme, a number of auxiliaries have been used for helping the process. Heptol ESW is a sequestering agent, environmentally friendly, coordinating with the calcium ions present into the pectin structure and consequently destroying the pectin net and favoring the enzyme access. Sulfolen 148 is a biodegradable tensioactive agent, helping the access of the reaction solution inside the fabrics.

The process was developed in mild conditions (1 hour at 55ºC). The yield was improved by the ultrasound intervention [15].

By comparison, the conventional scouring is usually performed at 100ºC, using sodium hydroxide and hydrogen peroxide, in the presence of a surfactant for textile material penetration [7], [18]. Besides the pollution connected to chemicals and higher energy needed, sodium hydroxide, being a non-specific reagent may partially destroy the textile material.

Most of the materials used in fabric contained cellulosic fibers, such as cotton, flax, ramie, viscose and lyocell had a tendency for "fuzz" formation (short fibers protruding from the surface) as well as "pilling" (fluffy/loosened fuzz attached to the surface). The prevention or elimination of fuzz and pilling is necessary for improving the textile material quality [19]. The process may be performed with cellulases being known as bio-polishing. During the last decade, treatments with cellulases for digesting protruding fibers have been widely used [20], [21].

The cellulases rich in endoglucanase activity appear to be better suited for bio-finishing [19]. Endoglucanases hydrolyze accessible intramolecular β-1,4-glucosidic bonds of cellulose chains randomly to produce new chain ends (fig. 2).

The fragmentation progress in time gives as final products glucose and cellobiose (water soluble compounds) [21], [22]. Thus, the bio-polishing process has been performed with a commercial product, Cellusoft L containing 1-4-β-endoglucanase. According to Petit and coworkers [23] the commercial product contains 7.8% protein.

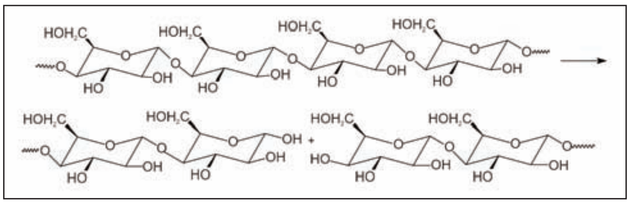

Fig. 2. Hydrolytic fragmentation of cellulose polymer chain by 1,4-β-endoglucanases

The enzyme hydrolyzed the small fibers of the textile composite material surface, making it better as regards aspect as well as smoother touch. The bio-treated materials have been compared with the chemical treated similar samples through the following criteria:

- Structure:
  - wideness, cm;
  - thickness, mm;
  - mass, g/m^{-2};
  - warp/weft density, threads dm^{-1}.
- Functional aspects:
  - breaking forces and elongations on the warp/weft directions as well as at un angle of 45 and 135º, N;
  - tearing forces wrap/weft, N;
  - warp/weft slippage, N;
  - surface abrasion resistance, cycles;
  - pilling, notes.
  - UPF protection, % and notes);
- Comfort of garments produced from these materials:
  - air permeability, L/m^{2}s;
  - water permeability, %;
  - porosity of treated fabrics, calculated according to formula (1) [24]:

$$Porosity\ (\Phi) = 1 - \frac{bulk\ volume}{medium\ volume} = 1 - \frac{\rho_b}{\rho_s} \qquad (1)$$

where:
ρ_s is medium density (fiber);
ρ_b – bulk density (woven fabric).

Fabric density can be calculated by formula (2):

$$\rho_b = \frac{fabric\ weight\ \ (g/cm^2)}{thickness\ \ (cm)} \qquad (2)$$

Maximum absorption capacity, C_m, is the weight ratio of the absorbed liquid over the dry solid medium weight and can be calculated by formula (3):

$$C_m = \frac{\rho_l}{\rho_s} \frac{\Phi}{1 - \Phi} \qquad (3)$$

where:
ρ_l is the liquid density.

Table 2 presents porosity values for classical and enzymatic treated fabrics.

The experimental determinations have been performed in standard conditions according the standardized methods indicated in table 3. By comparing the experimental data obtained after the enzymatic and the classical treatments some remarks could be emphasized.

Concerning structure of the fabric after chemical and bio-treatments:

- the enzymatic treatments reduced the width of the textile fabrics by 6.8 – 8.3%;
- the weight of all studied fabrics is higher for the enzyme treated ones by 11.1 – 14.3% indicating a lower loss of textile material during bio-treatments;
- all the enzyme treated materials have higher values for the thickness making them better as regards insulating and protection properties [24].

Regarding the functional properties a higher mechanical resistance to breaking and tearing forces was observed for the enzyme treated fabrics explained by the higher yarn density noticed in these material cases. An enhanced abrasion resistance is observed for all the enzyme treated materials. The pilling properties are comparable for the fabrics treated with chemicals or enzymes. Generally, the UPF protection is better for the enzyme treated materials.

The air permeability is diminished for the bio-treated materials making them better insulators. Meanwhile the water permeability is higher for these materials making them better for further water treatments during finishing processes. Also, porosity values and calculated maximum liquid absorption capacity, C_m

Table 2

Fabric type	Cecilia I classic	Cecilia I enzymatic	Efficiency enzymatic versus classic treatment Cecilia I, %	Cecilia II classic	Cecilia II enzymatic	Efficiency enzymatic versus classic treatment Cecilia II, %	Dochia classic	Dochia enzymatic	Efficiency enzymatic versus classic treatment Dochia, %
Fabric weight, g/m^2	223	253	-	252	293	-	132	154	-
Fabric thickness, mm	0.621	0.846	-	0.786	1.111	-	0.486	0.662	-
Fiber density, ρ_s, g/cm^3	1.54[a]	1.54[a]	-	1.54[a]	1.54[a]	-	1.54[a]	1.54[a]	-
Bulk density, ρ_b, g/cm^3	0.36	0.30	16.67	0.32	0.26	18.75	0.28	0.23	17.85
Porosity, Φ	0.766	0.805	4.84	0.791	0.828	4.47	0.821	0.848	3.18
Calculated maximum liquid absorption capacity, C_m	2.14	2.69	20.45	2.47	3.14	21.34	2.99	3.65	18.08

[a] – from reference [17]

Table 3

Fabrics properties	Cecilia I classic	Cecilia I enzy-matic	Cecilia II classic	Cecilia II enzy-matic	Dochia classic	Dochia enzy-matic	Method standard (ISO)
Structural data							
Width, cm	144	133.6	139.9	130.4	148.2	135.9	SR EN 1773:2002
Weight, g/m^2	223	253	252	293	132	154	SR EN 12127:2003
Thickness, mm	0.621	0.846	0.786	1.111	0.486	0.662	SR EN ISO 5084:2001
Warp density, threads/dm	308	340	320	358	340	360	SR EN 1049-2:2000
Weft density, threads/dm	174	226	210	228	174	226	SR EN 1049-2:2000
Functional data							
Breaking strength warp direction, N	669	714	600	698	392	386	SR EN ISO 13934-1:2002
Breaking strength weft direction, N	344	366	420	492	202	270	SR EN ISO 13934-1:2002
Breaking strength warp yarns, N	4.34	4.20	3.75	3.90	2.31	2.14	SR EN ISO 2062:2010 METHOD B
Breaking strength weft yarns, N	3.95	3.24	4.00	4.32	2.32	2.39	SR EN ISO 2062:2010 Method B
Elongation on warp direction, %	18.2	24.5	13.0	20.0	9.4	16.1	SR EN ISO 13934-1:2002
Elongation on weft direction, %	15.3	20.5	16.5	24.5	12.0	19.2	SR EN ISO 13934-1:2002
Tearing strength wrap, N	49.9	46.6	78.9	102.1	24.3	19.5	SR EN ISO 13937-3:2002
Tearing strength weft, N	18.4	17.84	32.2	28.8	13,7	13.79	SR EN ISO 13937-3:2002
Warp yarn slippage, N	410	385	226	312	269	257	SR 7523:1994
Weft yarn slippage, N	330	329	315	363	196	235	SR 7523:1994
Surface abrasion resistance, cycles	12 942	21 003	12 109	17 518	9 794	11 178	SR EN ISO 12947-2:2002
Pilling after 125 cycles	4-5	4-5	4-5	4-5	4-5	4-5	SR EN ISO 12945-2:2002
Pilling after 500 cycles	4-5	4-5	4-5	4-5	4-5	4-5	SR EN ISO 12945-2:2002
Pilling after 1 000 cycles	3-4	3-4	3-4	3-4	3-4	3-4	SR EN ISO 12945-2:2002
Pilling after 2 000 cycles	2-3	3-4	2-3	2-3	2-3	2-3	SR EN ISO 12945-2:2002
Pilling after 7 000 cycles	2-3	2-3	2-3	2-3	2-3	2-3	SR EN ISO 12945-2:2002
UPF protection, %	20	50+	40	50+	50+	25	SR EN 13758-1+A1:2007; SR EN 13758-2 + A1:2007
UPF protection, notes	Good	Excellent	Excellent	Excellent	Excellent	Very good	SR EN 13758-1+A1:2007; SR EN 13758-2 + A1:2007
Comfort data							
Air permeability, L/m^{2}s	509.4	170.6	440.7	183.2	926.6	433.3	SR EN ISO 9237:1999
Water vapors permeability, %	35.9	41.1	38.2	37	43.2	38.2	SR 9005:1979

values of enzymatic treated fabrics are higher than the values of classical treated fabrics for all types of fabrics. The behavior towards IR radiation showed also a better insulating quality in the case of bio-treated fabrics.

CONCLUSIONS

A number of blended materials from cotton and cottonised linen have been worked up with chemicals and enzymes in ultrasound conditions. The analysis of the obtained fabrics properties revealed a comparable or better quality for the bio-treated textile fabrics which make the ultrasonic enzymatic treatments a good solution in agreement with a sustainable development.

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Softener impact on environment friendly low and zero formaldehyde cross-linker performance for cotton

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REZUMAT – ABSTRACT

Impactul emolienţilor asupra performanţei agenţilor de reticulare cu conținut redus de formaldehidă sau fără formaldehidă, prietenoşi mediului, în cazul țesăturilor din bumbac

A fost evaluată performanța a trei agenți de reticulare – cu conținut ridicat de formaldehidă, cu conținut redus de formaldehidă și fără formaldehidă – aplicați pe țesături din bumbac. Materialele textile tratate cu agenți de reticulare cu conținut ridicat de formaldehidă și cu efecte toxice au prezentat proprietăți superioare ale revenirii din șifonare, comparativ cu alte produse. Adăugarea emolienților a sporit în mod semnificativ performanța de întreținere ușoară, conferită de agenții de reticulare prietenoși mediului. În plus, încorporarea unui amestec de emolienți și agenți de reticulare a dus la o îmbunătățire semnificativă a altor proprietăți ale materialului, cum ar fi: rezistență la rupere mărită, tușeu moale, frecare redusă între fire.

Cuvinte-cheie: agent de reticulare, formaldehidă, revenire din șifonare, emolient

Softener impact on environment friendly low and zero formaldehyde cross-linker performance for cotton

The performances of three easy care cross-linkers based on high, low and zero formaldehyde was assessed on cotton fabric. Toxic, high formaldehyde cross-linker treated fabric exhibited superior crease recovery properties than the alternatives. Incorporation of softeners significantly enhanced the easy care performance of the environment friendly cross-linkers. In addition, incorporation of softeners with cross-linkers also significantly improved the tear strength retention, and softness of the fabric which was reflected by reduced inter yarn friction.

Key-words: cross-linker, formaldehyde, crease recovery, softener

Cotton is the most widely used natural fiber in textiles due to its overall superior performance over other fibers. Unfortunately, creasing, especially after laundering is the main limitation of cotton fabric [1], but the introduction of cross-linkers such as formaldehyde based finishing reagents which introduce inter-polymer bonds and associated dimensional stability can control the problem. Formaldehyde is relatively cheap and an effective cross-linking agent, but it is also an irritant [2] – [4], mutagenic in certain bacterial and animal species, but most alarming of all, it is classified as a confirmed human carcinogen [5]. Hence, strict limits have been imposed in large number of countries and rigorous steps have been taken to introduce effective and extremely low or zero formaldehyde durable press (DP) finishing agents [6] – [7]. However, efficient zero formaldehyde cross-linker are either costly, like 1,3-dimethyl-4,5-dihydroxyethylene urea (DMeDHEU) [8] and 1,2,3,4 butane tetra carboxylic acid (BTCA) [9] – [10] or have other problems like yellowing in case of citric acid [11]. Modification of dimethyloldihydroxyethyleneurea (DMDHEU), especially for extremely low formaldehyde, causes a significant decrease in the effectiveness of the cross-linker [12]. Although dihydroxyethyleneurea (DHEU) is formaldehyde free, it is less effective than DMDHEU and modification is needed to overcome significant loss of whiteness and poor resistance to chlorine damage, which cause a further decrease in easy care performance.

Softeners are principally applied to textiles for improving handle, drape, tear strength and sewing properties. The handle of the fabric becomes harsh due to the removal of natural oils and waxes during pretreatment. There will be even more severe loss in the natural softness and tear strength of the fabric when a cross-linker is applied for better crease recovery [13]. So it is common practice to use a softener after or with the cross-linker application to retain the point of sale appeal.

Softeners and especially silicone softeners even without a fiber cross-linker impart a slight increase in the easy care performance [14]. However, reports are lacking onto the use of softeners or combinations of softeners as additives to significantly enhance the performance of extremely low or zero formaldehyde cross-linkers and additionally overcome the physical property loss of the fabric which is mainly due to the cross-linker.

In this research performance of low as well as zero formaldehyde cross-linkers with different softener combinations in one bath was analyzed.

EXPERIMENTAL PART

Materials and chemicals

100% mercerized cotton fabric was purchased from Phoenix Calico Ltd., UK. DMDHEU was kindly donated by Devan PPT. Knittex 7636 – a pre catalyzed modified DMDHEU, Knittex FFRC – a pre catalyzed modified DHEU, Ultratex SI – a silicone softener based on polydimethyl siloxane chemistry, and Sapamine FPG – a fatty acid amide softener, were supplied by Huntsman.

Methods used

Cotton fabric wet pick up was adjusted to 80% for all the recipes. Drying of the treated fabric was conducted at 85ºC for 5 minutes and cured at 160ºC for 3 minutes in the case of DMDHEU, 150ºC for 5 minutes in the case of the modified DMDHEU and 130ºC for 3 minutes in the case of DHEU as recommended by the manufacturer. If softener was used without a cross-linker, then it was padded at 80% wet pick up and dried at 130ºC for 3 minutes.
Samples were conditioned before performing any testing. Crease recovery angle was measured by using standard method of BS EN 22313:1992, ISO 2318:1972. BS EN ISO 13934-1:1999 method was adopted to analyze the tensile strength of the treated fabric. The BS EN ISO 13937-1:2000 method was used to determine the tear value of the treated fabric. Whiteness was measured by following the AATCC test method 110, CIE whiteness index (WI). Spectra Flash 600 instrument was used for this purpose. Durable press rating of the fabric after washing was given by using the AATCC 124-1996 method.
The Kawabata Evaluation System (KES) was used to measure the inter yarn friction (2HG5). Abrasion resistance was measured according to BS EN ISO 12947-2:1998 by the Martindale method.

RESULTS AND DISCUSSIONS

High formaldehyde based cross-linker, DMDHEU, imparted the highest crease recovery angle of 257º when used at the level of 120 g/L (table 1). There was gradual increase in the easy care and DP rating, by raising the level of DMDHEU. Although, DMDHEU has toxic issues with its application, nonetheless, its excellent easy care performance set the standards for other cross-linkers. Modified DMDHEU is an extremely low formaldehyde cross-linker and fulfilled the requirement of Oeko Tex 100. However, it was much less effective in improving the easy care properties of cotton. The DHEU treatment disappointingly only imparted a small increase in the dry crease recovery angle upon increasing the concentration up to 100 g/L and then, there was a slight decrease in crease recovery by increasing the concentration. It shows that although it is formaldehyde free, its performance needs to be improved.
The silicone softener imparted slight improvements in crease recovery up to 30 g/L and then remained constant (table 2). The fatty acid amide softener also imparted a slight improvement in crease recovery up to 15 g/L, where crease recovery angle of only 161º was obtained and then started to go down. However, the fatty acid amide softener was less effective on its own as compared to the silicone softener when applied to cotton to improve its easy care properties. However, crease recovery performance was further improved by using a combination of silicone and fatty acid amide softeners. The optimum amount of silicone and fatty acid amide softeners was 10 g/L and

Table 1

IMPACT OF CROSS-LINKERS ON THE EASY CARE PERFORMANCE OF THE COTTON					
Cross-linker, g/L		**Dry crease recovery angle**, º	**Durable press rating**		**Whiteness index**, WID
			1 wash	***5 washes***	
DMDHEU*	0	127	1.8	1.5	73.6
	60	211	2.4	2.2	72.9
	80	243	3.0	2.5	71.4
	100	251	3.5	3.4	70.7
	120	257	3.6	3.5	70.2
Modified DMDHEU	30	152	2.3	2.2	73.4
	60	180	2.4	2.3	72.4
	80	193	3.2	3.0	72.0
	100	205	3.3	3.1	71.9
Modified DHEU	30	142	3	2.5	72
	60	159	3	2.5	71.2
	80	172	3.1	2.6	70.6
	100	175	3.2	2.6	69.8
	120	174	3.2	2.8	69.1

* $MgCl_2 \cdot 6H_20$ was used as catalyst and mole ratio between DMDHEU and catalyst was 0.2

Table 2

IMPACT OF SOFTENERS ON THE EASY CARE PERFORMANCE OF THE COTTON					
Softener, g/L		Dry crease recovery angle, °	Durable rating press		Whiteness index, WID
			1 wash	*5 washes*	
Silicone softener	0	127	1.8	1.5	73.6
	10	160	1.8	1.7	66.1
	20	167	1.8	1.7	66.0
	30	176	1.9	1.8	65.9
	40	178	2.0	1.9	65.9
Fatty acid amide softener	10	153	1.8	1.7	69.2
	15	161	1.8	1.7	69.0
	20	158	1.8	1.7	68.8
	30	155	1.8	1.7	68.3
	40	154	1.8	1.7	68.0
Fatty acid amide + Silicone					
15	5	171	1.8	1.7	68.2
15	10	182	1.9	1.8	66.0
15	20	175	1.9	1.8	65.1
10	10	176	1.9	1.8	65.4
20	10	177	1.9	1.8	65.2

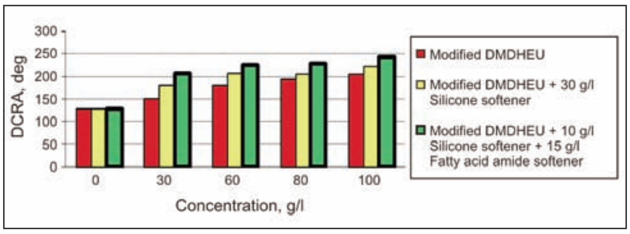


Fig. 1. Impact of different softeners with modified DMDHEU on the easy care properties of the cotton

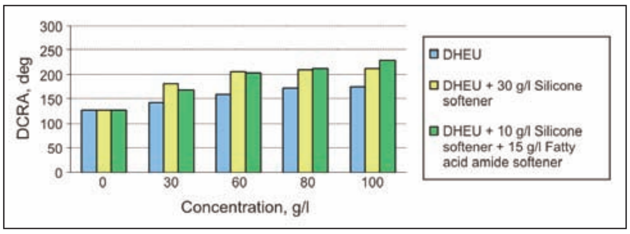


Fig. 2. Impact of different softeners with DHEU on the easy care performance of the cotton

15 g/L respectively, where crease recovery angle of 182° was achieved (table 2).

Excellent crease recovery angle of 248° was obtained by using 100 g/L of the modified DMDHEU cross-linker in combination with 10 g/L of the silicone softener and 15 g/L of the fatty acid amide softener. The crease recovery enhanced as the amount of the cross-linker raised and at 100 g/L of cross-linker, a significant improvement was imparted (fig. 1).

Although DHEU alone offered a relatively poor crease recovery performance, but when used in combination with the silicone softener, it imparted a significant improvement (fig. 2).

In addition, a combination of 10 g/L of silicone softener and 15 g/L of fatty acid amide softener with DHEU exhibited a slightly better crease recovery angle of 182° than 30 g/L of silicone softener alone.

It is quite clear that the easy care crease properties of DMDHEU treated fabric was much better on its own than modified DMDHEU and DHEU treated cotton. However, when modified DMDHEU and DHEU were used with a mixture of silicone and fatty acid amide softeners, then the difference between the three cross-linkers was much less (fig. 3).

It can also be concluded that DHEU treated fabric imparted better and improved performance with softeners as compared to the modified DMDHEU.

Although DMDHEU treated cotton exhibited excellent crease recovery properties, but it resulted in the significant decrease of the tensile strength. By increasing the concentration of the formaldehyde cross-linker, there is a gradual strength loss of 30% up to 60 g/L, and then it slowed down (fig. 4).

The modified DMDHEU treated fabric exhibited better tensile strength retention properties than DMDHEU. Surprisingly, the formaldehyde free cross-linker initially showed improvements in tensile strength as compared to the untreated sample up to the level of 60 g/L and after that level there was slight decrease. However, it was still very close to the untreated sample.

There was significant decrease of 80% in the abrasion resistance retention when DMDHEU was used at the level of 100 g/L (fig. 5), while modified DMDHEU demonstrated double the abrasion resistance retention as compared to DMDHEU treated cotton. However, when silicone and fatty acid amide softeners were used with modified DMDHEU, then there

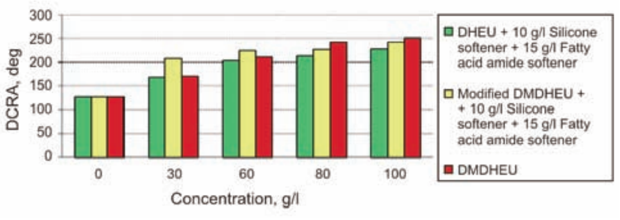


Fig. 3. Comparison of cotton treated with DMDHEU, modified DMDHEU and DHEU with different softeners on crease recovery performance

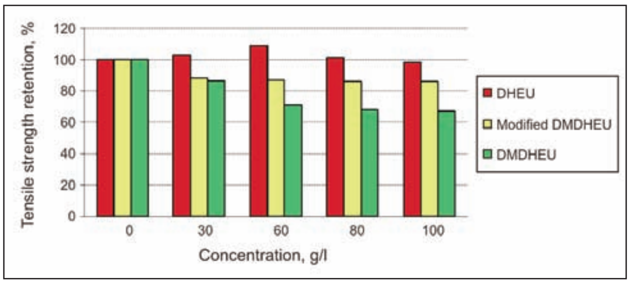


Fig. 4. Tensile strength retention of DMDHEU, modified DMDHEU and DHEU treated cotton fabric

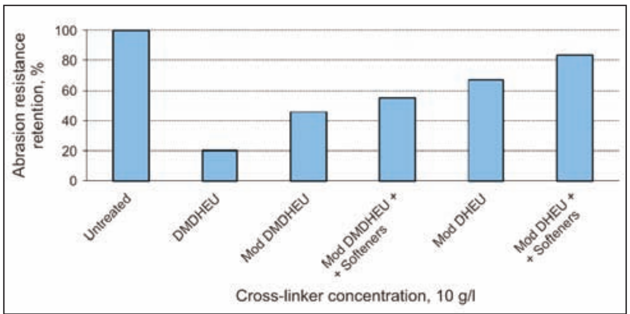


Fig. 5. Abrasion resistance retention of DMDHEU, modified DMDHEU and DHEU treated cotton fabric

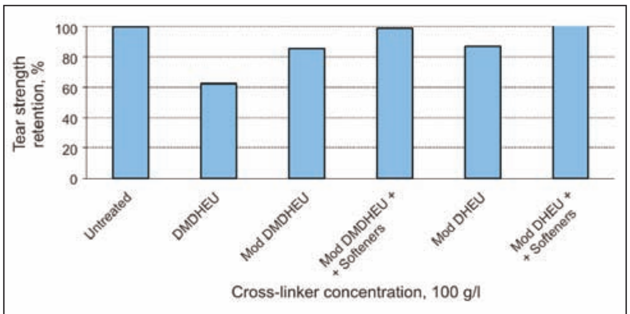


Fig. 6. Tear strength retention of DMDHEU, modified DMDHEU and DHEU treated cotton fabric

was a 10% increase in abrasion resistance. Although, modified DHEU itself demonstrated good retention of abrasion resistance, there was further increase of 20% in abrasion resistance retention when a combination of silicone and fatty acid amide softeners were used with DHEU. It can also be concluded that by using the same combination of softeners, DHEU exhibited better retention of abrasion resistance than modified DMDHEU (fig. 5).

DMDHEU cross-linker treatment on cotton fabric imparted a typical deleterious effect on the tear retention of the fabric and 38% of the tear strength of the fabric was lost. Modified DMDHEU and modified DHEU demonstrated superior retention of tear strength and there was a loss of 14% and 13% in tear strength, respectively. Tear strength retention of the fabric was further improved by incorporation of silicone and fatty acid amide softeners with modified DMDHEU, and there was only a loss of 1% in the tear strength. However, the best results were obtained when a combination of softeners was used with the modified DHEU, and the tear strength of the fabric was increased by 11% as compared to the untreated fabric (fig. 6).

Inter yarn friction values (2HG5) measured by sophisticated KES-F is an excellent mean to measure the "feel" of the fabric objectively. It is quite clear that by raising the level of modified DMDHEU, gradual increase in the inter yarn friction was observed (fig. 7), as these agents introduced cross links between cellulose chains and enhanced the fiber rigidity. However, when a combination of silicone 10 g/L and fatty acid amide 15 g/L was used, there was a significant decrease in the inter yarn friction. Although, when the modified DMDHEU concentration was increased with a fixed amount of softeners, there was a slight increase in the inter yarn friction. However, even at 100 g/L of the cross-linker, the inter yarn friction value was half of the untreated fabric.

DHEU alone and with a combination of softeners also exhibited similar levels of inter yarn friction as those of modified DMDHEU (fig. 8). So it can be concluded that it is the presence of the softener which can significantly influence the inter yarn friction rather than the cross-linker.

Enhancement of the softness of the fabric after the application of softeners was further evident from SEM figures. Untreated fabric SEM image was clean as demonstrated by figure 9 *a*. There was also not much activity on the fabric surface when treated with

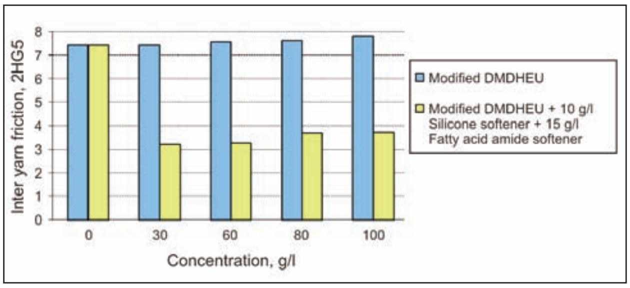


Fig. 7. Effect of modified DMDHEU alone and with softeners on inter yarn friction of treated cotton fabric

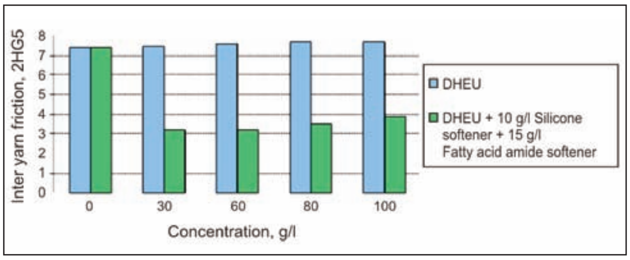
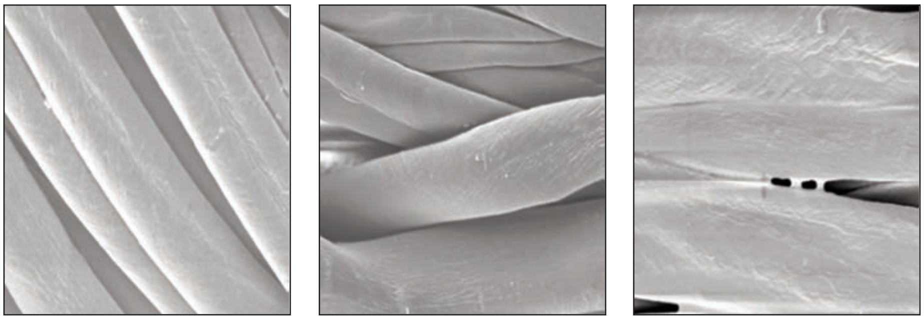


Fig. 8. Effect of DHEU alone and with softeners on inter yarn friction of treated cotton fabric



a *b* *c*

Fig. 9. SEM of cotton fabric finished with cross-linker and softener:
a – untreated; *b* – DHEU cross-linker treated; *c* – DHEU cross-linker and softener

DHEU cross-linker as cross-linker made intra fiber bonding (fig. 9 *b*). However, when cotton fabric was treated with cross-linker and softener there was clear coating on the surface and between the fibers as evident in figure 9 *c*, which can explain the significant improvement in the softness of the fabric after softener application.

CONCLUSIONS

It was possible to obtain good crease recovery properties with a low or zero formaldehyde cross-linker by incorporating softeners into the recipe. A small quantity of softener was needed and it was applied with the cross-linker in one bath, so that there were no extra processing costs. Modified DMDHEU and DHEU exhibited good crease recovery when used with 30 g/L of silicone softener. However, a combination of softeners, 10 g/L silicone and 15 g/L fatty acid amide softeners, imparted a significant increase in crease recovery when used with modified DMDHEU and DHEU. Softeners not only improved the crease recovery properties, but also had a beneficial effect on tear strength and handle of the cross-linked fabrics.

When a combination of softeners was used with modified DMDHEU and DHEU, inter yarn friction was reduced. Significant strength loss was observed with DMDHEU, but overcame by the use of modified DMDHEU and DHEU. There was also a significant improvement in abrasion resistance when a low or zero formaldehyde cross-linker was used as compared to DMDHE.

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DOCUMENTARE

Aparate de măsură

INSTRUMENT DE TESTARE A FIBRELOR DE BUMBAC

Fiind un produs natural, firele de bumbac prezintă o mare variabilitate, de aceea testarea corectă a bumbacului este foarte importantă. Una dintre problemele cu care se confruntă filatorii de bumbac este dată de prezența nopeurilor. Numărul mare de nopeuri cauzează apariția defectelor și, ulterior, a petelor albe, ușor de observat pe fire.

Rezolvarea acestei probleme, înaintea procesului de filare, necesită informații precise privitoare la prezența nopeurilor.

Uster Afis Pro 2, realizat de ***Uster Technologies AG***, din Elveția, furnizează informații exacte privind numărul total al nopeurilor, dimensiunea nopeurilor și maturitatea fibrei. Filatorii sunt interesați în mod special de maturitatea fibrei. Pentru aceasta, este necesară identificarea și separarea conținutului de fibre imature. În procesul de filare, fibrele imature cauzează rupturi, fibre excesiv de scurte și chiar un procentaj mare de deșeuri.

În timpul filării, aceste probleme duc, inevitabil, la un număr mare de defecte ale firelor și la un randament scăzut al materiei prime.

În etapele ulterioare de prelucrare, nopeurile create de fibrele imature vor apărea ca pete albe în țesătura vopsită, ducând la o calitate scăzută a materialului și la micșorarea profitului.

Datele furnizate de sistemul Uster Afis Pro 2 dintr-o secvență de teste ale materialului – de la bataj la laminarea finală – asigură monitorizarea permanentă și informații precise asupra nopeurilor, în special al celor din fibră, dar și date privitoare la maturitatea fibrei, procentul de fibre imature și conținutul de fibre scurte.

Prin urmare, riscul producerii unor fire de calitate inferioară, cu impact asupra profitabilității companiilor, poate fi evitat prin optimizarea amestecului de fibre și realizarea unui reglaj corespunzător al setărilor mașinii.

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Investigating the usage possibility of metal mono carboxylates(metal naphthenates) as antibacterial agent in textile applications

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REZUMAT – ABSTRACT

Studierea posibilității utilizării monocarboxilaţilor metalici (naftenaţilor metalici) ca agenţi antibacterieni în aplicaţiile textile

Obiectivul acestui studiu este acela de a produce o substanță care să poată fi utilizată ca agent antibacterian pentru textilele din subproduse petroliere, cu scopul de a extinde posibilitățile de utilizare a petrolului și a subproduselor petroliere în domeniul textilelor. În acest scop, au fost produși compuși complecși ai cuprului, cobaltului, nichelului, zincului și sodiului. Acești compuși au fost aplicați pe o țesătură din bumbac, prin procesul de fulardare, iar activitatea antibacteriană a acestora a fost evaluată în conformitate cu metoda difuziei în agar AATCC 147. În urma experimentelor, s-a demonstrat că cele mai bune rezultate au fost obținute în cazul bacteriilor gram-pozitive și gram-negative, cu monocarboxilați ai cuprului. Activitatea acestora a fost încă prezentă după 3 cicluri de spălare, în cazul bacteriilor Staphylococcus aureus și Escherichia coli, și după 10 cicluri de spălare, în cazul Bacillus subtilis și Klebsiella pneumoniae.

Cuvinte-cheie: monocarboxilați metalici, naftenat, bumbac, antibacterian

Investigating the usage possibility of metal mono carboxylates (metal naphthenates) as antibacterial agent in textile applications

In this study, it was aimed to produce a substance, which could be used as an antibacterial agent in textiles, from petroleum sub-products in order to extend the use of petroleum and petroleum sub-products in textiles. For this aim, complex compounds with copper, cobalt, nickel, zinc, and sodium, were produced. Then, those compounds were applied onto cotton fabric by padding process, and their antibacterial activities were evaluated according to the AATCC 147 agar diffusion test method. As a result of the trials, it was determined that best results were obtained against both gram-positive and gram-negative bacteria with copper mono carboxylate. Its activity was still present against Staphylococcus aureus and Escherichia coli after 3 washings and against Bacillus subtilis and Klebsiella pneumoniae after 10 washings.

Key-words: metal mono carboxylate, naphthenate, cotton, antibacterial

Textile products provide the suitable temperature, moisture, and nutrients for the microorganisms to live and reproduce due to their structures and places of use [1]. Uncontrolled reproduction of microorganisms on textile products results in many undesirable effects, such as colour change in textile products, bad smell, worse handle property, and negative effects on performance aspects. When it is investigated in terms of human health, it might cause infections to spread, especially at hospitals, by carrying microorganisms. This situation makes antimicrobial textile products be produced and used more [2] – [6]. Microorganisms living in textile products might harm both the product itself and its user. Antimicrobial textile products help to minimize or clear the negative effects of microorganisms. This group of products is used to prevent microorganism to infect, keep the infections under control, and prevent microorganism-based bad smell, staining, colour change, and quality loss [7].

Antimicrobial fabrics, which are among functional textile products, have a considerable market share not only in daily use areas but also in exclusive areas. Today, increasing the security and safety level of buildings, transportation systems, and vehicles and preventing microorganisms from reproducing are among the basic needs of people living in economically and socially developed countries. Antimicrobial fabrics are commonly used in daily life, as well as for hygiene purposes at hospitals everywhere, especially in developed countries. For instance, textile products, which are used in different areas, such as hotel textiles, baby clothes, furnishing fabrics, kitchen mops, towels, napkins, sports clothes, hunting clothes and socks are required to have antimicrobial effect. Antimicrobial effect ought to be permanent in antimicrobial effect-added products [8] – [10].

Petroleum, one of the most precious subsurface raw materials in the world, provides great benefits to humankind not only in technology, but in many areas. Usage areas of materials, which are produced as sub-product from petroleum, are progressively increasing. Although some of the sub-products are of little importance, some could be evaluated and sold after an additional process and expense [9]. Paraffin, gas, maintenance oil, grease, asphalt, and many other sub-products are produced from petroleum. Additionally, products such as ethane, butane, propane, paraffin oil, diesel fuel, diesel oil, and paraffin wax are among the petroleum sub-products. The less known petroleum products have surprising usage areas. There is petroleum wax (paraffin wax) in wax and polish, and perfumes, cosmetics, and even some materials that prevent cheese from decaying are made of petroleum oil. There are other petroleum oils in insecticides. Ethylene, the substance that is used to ripen tomatoes, artificial silk, and acetone, the substance that is used for nail polishing, are made of gases, which are derived from the treating process. Main chemicals, used while producing artificial rubber, plastics, and liquid detergents are also petroleum products. Petroleum and petroleum sub-products, which have usage aspects in many areas, are not commonly used in textiles [11].

This study was planned to produce a substance, which could be used as an antibacterial agent in textiles, from petroleum sub-products in order to extend the use of petroleum and petroleum sub-products in textiles. For this aim, complex compounds with copper, cobalt, nickel, zinc, and sodium, were produced. Then, those compounds were applied onto cotton fabric by padding process, and their antibacterial activities were evaluated. By this way, a waste material is provided with a new usage area.

MATERIALS AND METHODS

All experiments were carried out with 100% cotton woven fabric. In the study, usage possibility of various metal cyclohexane mono carboxylates (naphthenates) as an antibacterial agent in the textile industry was investigated.

In the first stage of the study synthesis and characterization of antibacterial agents were carried out. For this aim, copper, cobalt, nickel, zinc, and sodium cyclohexane mono carboxylate compounds having blue, pink, green and white colours obtained from the reaction of naphthenic acid and metal salts [11], [12]. Synthesis reaction of metal naphthenates was as follows [13].

$$RCOOH + NaOH \rightarrow RCOONa + H_2O \quad (1)$$

$$2RCOONa + MeX \rightarrow Me(RCOO)_2 + Na_2X \quad (2)$$

where:

R is cyclohexane;

Me – metal (copper, cobalt, nickel or zinc);

X – salt anion (chloride or sulphate).

For the actualization of the reaction, firstly cyclohexane mono carboxylic acid was dissolved in the organic solvent and sodium salt of cyclohexane mono carboxylate was constituted by adding sodium hydroxide [13], [14]. By this way sodium cyclohexane mono carboxylate was obtained. For synthesis of other metal cyclohexane mono carboxylates, after obtaining sodium cyclohexane mono carboxylate, this compound was put into reaction with various metal salts. The synthesis of the agents is explained below.

Synthesis of agents

Thermometer, condenser and dropping funnel were placed at the necks of a three necked flask, as shown in figure 1. Then the solution of the cyclohexane mono carboxylic acid in 10% (v) diethyl ether, which is calculated stoichiometrically, was put into the flask, and the solution of 10% (v) NaOH was put into the dropping funnel. The temperature was raised to 40–45°C by running the magnetic stirrer, and the solution was stirred while dropping NaOH from dropping funnel for 60 minutes. The *p*H of the medium was adjusted between 7 and 8. Afterwards, the calculated amount of 10% (v) solution of copper sulphate pentahydrate, zinc chloride dihydrate, cobalt chloride hexahydrate or nickel chloride hexahydrate were used. The heater was turned on, the dropping funnel was opened and the mixture was stirred by running electromagnetic stirrer (Hot-Plate 300°C, 15 cm, circular M15 type) at room temperature for 1 hour, then this solution was held for 24 hours. Afterwards the solution obtained was put into the extraction flask and liquid phase was separated from the organic phase. After removal of solvent from the organic phase, metal cyclohexane mono carboxylate compounds were obtained [13].

After the synthesis of various metal cyclohexane mono carboxylates were completed, liquor containing 40 g/L metal mono carboxylate compound, 1 g/L dispersing agent (Denpol HT-Denge Kimya), 1 g/L wetting agent (Denwet PB 100-Denge Kimya) and acetic acid (for adjusting *p*H to 5) was prepared by stirring at 40-45ºC for 10-15 minutes in the ultrasonic bath (Baysonic). Application recipe was determined by taking the pre-test results into consideration. Then cotton fabrics were impregnated with these liquors and dried with tenter frame dryer at 85ºC for 4 minutes.

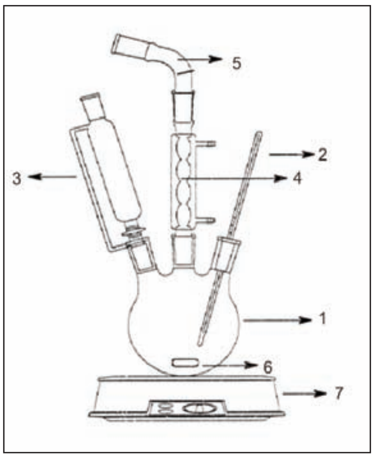
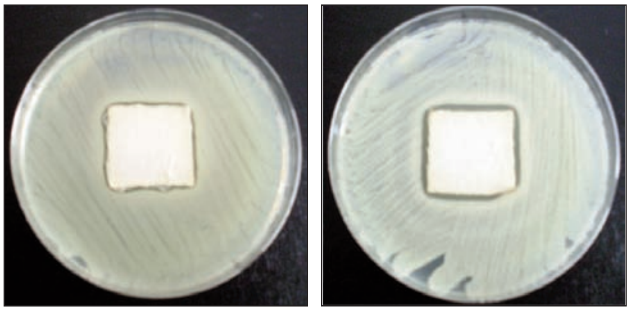


Fig. 1. Extraction equipment of the cyclohexane mono carboxylates:
1 – three-neck glass flask; 2 – thermometer;
3 – dropping funnel, *4* – condenser; *5* – $CaCl_2$ tube;
6 – magnet; *7* – magnetic stirrer

Then, antibacterial tests were applied to the fabrics before washing and after 3-5-10 washings according to AATCC 147 agar diffusion test method. As gram-negative bacteria *Escherichia coli* (ATCC25922) and *Klebsiella pneumoniae* (ATCC13883), as gram-positive bacteria *Bacillus subtilis* (NRRL NRS744) and *Staphylococcus aureus* (ATCC29213) were used.

RESULTS AND DISCUSSIONS

Antibacterial test results according to AATCC 147 standards of metal cyclohexane mono carboxylate compounds applied cotton fabrics before washing and after 3–5–10 washings are presented in table 1. As shown in table 1, no antibacterial activity against gram-positive bacteria were obtained with cobalt, nickel, and sodium mono carboxylates, even before washing. Zinc mono carboxylate displayed antibacterial activity against *Bacillus subtilis* and *Escherichia coli* before washing but lost its efficiency completely after washing. These results related to the synthesized products can be interpreted as limited importance for use in textiles. On the other hand, the most striking results in terms of textile applications were obtained with copper mono carboxylate. Copper mono carboxylate displayed antibacterial activity against *Bacillus subtilis* and *Escherichia coli* before washing. Its antibacterial activity maintained against *Staphylococcus aureus* after 3 washings (fig. 2) and against *Bacillus subtilis* after 10 washings (fig. 3).
Antibacterial activities of 6 different metal mono carboxylates against gram-negative bacteria on cotton fabric before washing and after 3-5-10 washings are presented in table 2.
Examination of table 2 reveals that cobalt, nickel, and sodium mono carboxylates showed no antibacterial



a *b*

Fig. 2. Appearance of antibacterial activity of copper mono carboxylate applied fabric against *Staphylococcus aureus*: *a* – before washing; *b* – after 3 washings

Table 1

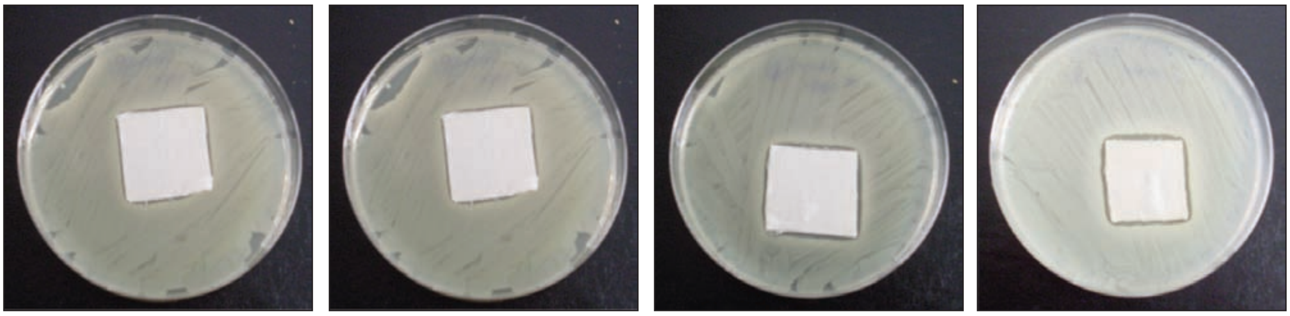
INHIBITION ZONE DIAMETERS OF SYNTHESIZED METAL MONO CARBOXYLATES AGAINST GRAM-POSITIVE BACTERIA MEASURED ON COTTON FABRIC BEFORE WASHING AND AFTER 3–5–10 WASHINGS

Metal	Bacteria	Before washing	After 3 washings	After 5 washings	After 10 washings
Copper	*B. subtilis*	1 mm	1 mm	1 mm	1 mm
	S. aureus	1 mm	1 mm	-	-
Cobalt	*B. subtilis*	-	-	-	-
	S. aureus	-	-	-	-
Nickel	*B. subtilis*	-	-	-	-
	S. aureus	-	-	-	-
Zinc	*B. subtilis*	3 mm	-	-	-
	S. aureus	1 mm	-	-	-
Sodium	*B. subtilis*	-	-	-	-
	S. aureus	-	-	-	-

Table 2

INHIBITION ZONE DIAMETERS OF SYNTHESIZED METAL MONO CARBOXYLATES AGAINST GRAM-NEGATIVE BACTERIA MEASURED ON COTTON FABRIC BEFORE WASHING AND AFTER 3–5–10 WASHINGS

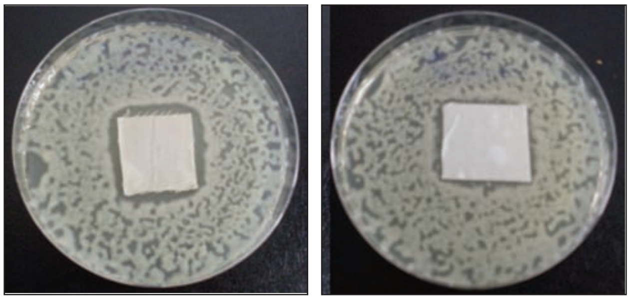
Metal	Bacteria	Before washing	After 3 washings	After 5 washings	After 10 washings
Copper	*E. coli*	2 mm	1 mm	-	-
	K. pneumoniae	2 mm	2 mm	1 mm	1 mm
Cobalt	*E. coli*	-	-	-	-
	K. pneumoniae	-	-	-	-
Nickel	*E. coli*	-	-	-	-
	K. pneumoniae	-	-	-	-
Zinc	*E. coli*	2 mm	-	-	-
	K. pneumoniae	-	-	-	-
Sodium	*E. coli*	-	-	-	-
	K. pneumoniae	-	-	-	-



a *b* *c* *d*

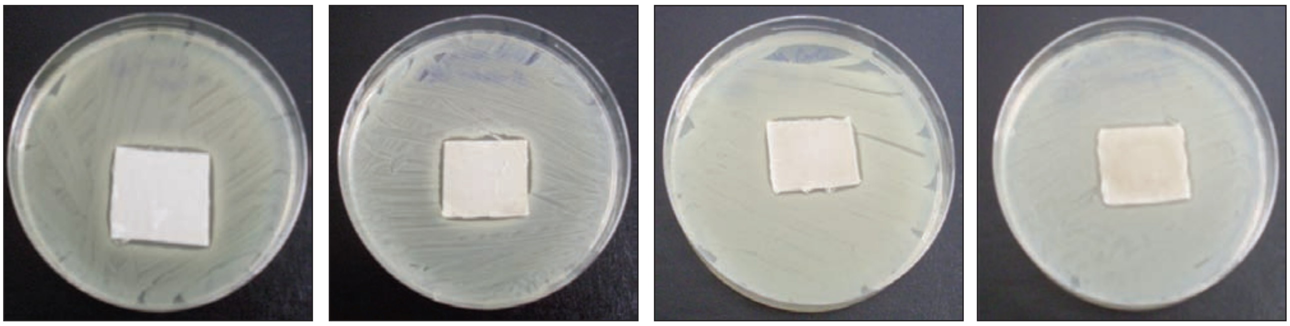
Fig. 3. Appearance of antibacterial activity of copper mono carboxylate applied fabric against *Bacillus subtilis:*
a – before washing; *b* – after 3 washings; *c* – after 5 washings; *d* – after 10 washings

activity against gram-negative bacteria on cotton fabric even before washing. Zinc mono carboxylate displayed antibacterial activity only against *Escherichia coli* before washing but lost its efficiency completely after washing. Therefore, it can be said that these products are not suitable for use in textiles. Again copper mono carboxylates showed the most valuable results in terms of textile applications, similarly as determined against gram positive bacteria. Antibacterial activity was obtained against *Escherichia coli* and *Klebsiella pneumoniae* by copper mono carboxylate before washing. The antibacterial activities against *Escherichia coli* (fig. 4) and *Klebsiella pneumoniae* (fig. 5) were lost after 3 and 10 washings respectively.



a *b*

Fig. 4. Appearance of antibacterial activity of copper mono carboxylate applied fabric against *Escherichia coli:*
a – before washing; *b* – after 3 washings



a *b* *c* *d*

Fig. 5. Appearance of antibacterial activity of copper mono carboxylate applied fabric against *Klebsiella pneumoniae:*
a – before washing; *b* – after 3 washings; *c* – after 5 washings; *d* – after 10 washings

CONCLUSIONS

In this research, metal mono carboxylates, except nickel, cobalt, and sodium mono carboxylates, were shown to display antibacterial activity against various bacteria on cotton fabric. However, the antibacterial activities are required to be lasting even after repeated washings for textile applications. For this reason, it can be concluded that the best results against gram-positive and gram-negative bacteria were obtained by using copper mono carboxylates. Its activity was lasting against *Staphylococcus aureus* and *Escherichia coli* after 3 washings and against *Bacillus subtilis* and *Klebsiella pneumoniae* after 10 washings.

By taking into consideration that environmentally friendly production and recycling are gradually getting more crucial, it can be said that copper mono carboxylate which is obtained from waste recycling, seems to be promising in antibacterial applications.

ACKNOWLEDGEMENTS

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ESD PROTECTIVE EQUIPMENT

Investigation of two-layer knitted structures with conductive fibres content*

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REZUMAT – ABSTRACT

Echipamente de protecție EDS.
Investigarea structurilor tricotate bistrat, cu conținut de fibre conductive*

Provocările industriei textile la nivel național și european trebuie să facă față atât cererii reduse de produse/sisteme textile de pe piețele tradiționale, cât și creșterii oportunităților de piață în economiile convergente. Răspunsul la aceste provocări sunt textilele tehnice ale căror piață va înregistra, până în anul 2016, o creștere anuală de circa 23%. Echipamentele de protecție sunt o alternativă pentru dezvoltarea durabilă a agenților economici de profil și pentru protecția sănătății omului. Această lucrare prezintă cercetările privind poziționarea firelor în structuri tricotate bistrat, destinate realizării echipamentelor de protecție pentru controlul descărcarii electrostatice (ESD). Lucrarea este de interes pentru mediul de afaceri, cercetare și învățământ.

Cuvinte-cheie: fire, caracteristici performante, tricoturi bistrat, inovare

ESD protective equipment.
Investigation of two-layer knitted structures with conductive fibres content*

The textile industry at both national and EU level must cope with challenges such as the reduction of demand of textile products/systems on traditional markets and an increase of market opportunities in converging economies. Technical textiles are a response to these challenges, and their market is expected to register an annual growth of approx. 23% by 2016. Protective equipment represents an alternative for the sustainable development of companies and for human health protection. This paper presents research regarding the position of knitted yarns in two-layer structures for the development of protective equipment that controls electrostatic discharge (ESD). The paper is of interest for business – research – education environments.

Key-words: high-performance characteristics, yarn, two-layer knitted structures, innovation

The approach to textiles manufacturing has changed from a technical to a consumer oriented one and as such it implies strategic thinking and New Product Development (NPD). This new process can be achieved by following five mandatory steps [www.arenasolutions.com/...new-product-development-npd], namely: concept definition (directions and barriers, technical and financial limitations), idea (as a result of brainstorming), design (focused on technical details that guarantee the idea will be implemented), testing (to check if the product meets the objectives set and whether it requires additional changes), launch (when a new product is introduced/NPI). The insertion of “invisible functionalities” in textile structures, the use of functional elements as part of the whole garment are just a few tools which define “freedom” of creation in the field of textiles.

To formulate a new conceptual model for approaching intelligent design one needs to define the interdependencies of converging elements/NPD processes, trends, lifestyle, preferences, converging criteria, new knowledge etc. In this context, protective and safety equipment constitutes a strategic direction for the development and competitiveness of economic activities. The electrical, chemical and mechanical properties of conductive textiles are crucial for intelligent textiles [1], [2].

EXPERIMENTAL PART

The electrostatic discharge (ESD) can be defined as a sudden transfer of electrostatic charge between two objects of different potentials. Accumulation of charges on an object is the result of various causes – friction, corona or induction charging [3], [4].

The ESD prevention may include the use of suitable insulating materials, of conductive filaments or of protective clothing for workers in electrical installations, of conductive wrist and leg straps to prevent the accumulation of high voltage in the workers’ body, antistatic floor mats or conductive materials that carry

* Partea I/ *First part*

electrical charges away from the work surface, and humidity control [5].

Typically, the electrostatic discharges are accompanied by significant high frequency disturbances that can damage the systems and data transmissions, can cause damage to electrical and electronic circuits (especially partially or totally irreversible breakdowns in semiconductor junctions) [6].

The protective and static control clothing is intended to minimize the effects of static electricity or loads on a person or on his/her clothing. Woven or knitted multifunctional textile substrates are used to this purpose. The protective equipment's physical and mechanical performance requirements and comfort exigencies impose: optimal use of conductive and non-conductive yarns in various textile structures, advanced processing technology and fashion design.

Functional characteristics are defined as follows [7], [8]:

- resistivity 10^4–10^5 $\Omega \cdot m$;
- distribution of electrostatic charges (EN 1149-1. *Protective clothing – Electrostatic properties*. Part 1: Test methods for the measurement of surface resistivity);
- provision of instantaneous discharge (EN 1149-3. *Protective clothing – Electrostatic properties*. Part 3: Test methods for the measurement of charge decay);
- anti-static properties are preserved even after 100 wash cycles.

An ESD protective garment should ideally have the following functions [9]:

- the protective garment should effectively shield the electric field originating from the insulating parts of the operators' normal clothing;
- the protective garment should prevent direct discharges from the operators' normal clothing;
- the protective garment should not itself cause similar problems. That is, it should not generate electrostatic field external to the garment and it should not be a potential source of direct electrostatic discharges.

To ensure the performance of a maximum safety and comfort activity, a two-layer structure with different electrostatic behaviour has been chosen. The outer layer is mainly dissipative, providing protection against short circuit and limiting the amount of electrostatic energy that can be dissipated to the work environment, while the inner layer is mainly conductive, ensuring controlled drainage of static electricity. An additional requirement for the inner layer is to ensure the user's comfort.

Two-layer knit variants were made with plaited structures, with parallel evolution of two or more yarns with strictly determined relative position as a result of their submission at different angles (plaiting yarn *V* at an angle smaller than ground yarn *F*). The most used knitted structures are jersey and rib structure. In case of jersey structure, the plaiting yarn *V* appears on the foreground on the front and the ground yarn *F*, on the foreground on the back of the fabric. In case of rib structure due to alternating of front-back wales-both the plaiting yarn (at front aspect stitches) and the ground yarn (at rear aspect stitches) will be present on the foreground, on each side of the fabric.

As a result of a functional selection stage the following configuration of the two-layer structure was set:

- inner layer:
  - conductor type 2–75% cotton + 25% hemitropic yarn (*Nm* 34/1 carbon coated polyester);
- outer layer:
  - conductor type 3 – bi-component multifilament yarn, type Nega-Stat P210, dtex 112 f 12, with trilobal carbon core, that when extruded with polyester, it extends to the surface thus increasing the conductivity;
  - conductor type 4 – bi-component multifilament yarn, obtained by core sheath, Nega-Stat type P190, 156 dtex f 24, with trilobal carbon core;
  - conductor type 5, filaments of nylon surface saturated with carbon film.

Nega-Stat® is introduced into textile materials to provide protection against a range of risks and hazards caused by static electricity in industrial end-use situations.

Knitting is made on knitting machines having yarn thread guides with special construction that provide the yarns with different deposition angles under needle head, so that in the stitch forming stage the plaiting yarn *V*, in our case a conductive yarn, remains on the front of fabric, while the ground yarn *F*, in our case the fabric yarn will stay on the back of the fabric. Both types of fabric were made on Stoll knitting machines, from SC Tanex SRL, with a possibility to have a differentiated adjustment of yarn tension so as to ensure the correct plaiting of the fabric (table 1).

Yarn used:

- ground yarn – *Nm* 50/3, 100% cotton;
- ground yarn – *Nm* 30/2, 100% wool;
- plaiting yarn – conductive yarn type 2: 75% cotton + 25% epitropic yarn (*Nm* 34/1 carbon coated polyester);
- plaiting yarn – conductive yarn type 3, respectively bi-component multifilament yarn with core-sheath structure, Nega-Stat P210, 112 dtex 12 f, polyester filament with trilobal core and carbon outer layer;
- plaiting yarn – conductive type 4, respectively Nega-Stat P190, 155 dtex, 24 f, polyester filament with trilobal carbon inner core;
- plaiting yarn – conductive type 5, nylon filament superficially saturated with carbon particles.

Reduced elasticity of the used yarns imposed low speed knitting of 0.65 m/s., and the plaiting of knitted fabric was performed in two fabric textures.

RESULTS AND DISCUSSIONS

Protective equipment must satisfy the requirements imposed by:

- specific work conditions, by maintaining normal working capacity, ensuring comfort and security conditions in order to optimally execute specific activities;

Table 1

SAMPLES RESULTED AFTER EXPERIMENTS				
Sample no.	**Structure**	**F_1/Front**	**F_2/Rear**	**Conductive yarn percentage**
1	Plaited jersey	one cotton yarn + + one yarn type 2	one cotton yarn + + three yarns type 4	6%
2	Plaited rib		one cotton yarn + + two yarns type 3	5%
3	Plaited jersey		one cotton yarn + + two yarns type 3	5%
4	Plaited rib		one cotton yarn + + three yarns type 3	6%
5	Plaited jersey		one cotton yarn + + one yarns type 3	4%
6	Plaited jersey		one cotton yarn + + two yarns type 4	6%
7	Plaited jersey		one cotton yarn + + one yarns type 4	5%
8	Plaited rib		one cotton yarn + + one yarns type 4	4.5%
9	Plaited rib		one cotton yarn + + two yarns type 4	6%
10	Plaited jersey		one cotton yarn + + one yarn type 5	4.5%
11	Plaited jersey		one cotton yarn + + two yarns type 5	6%
12	Plaited jersey		one cotton yarn + + three yarns type 5	7.5%
13	Plaited rib		one cotton yarn + + three yarns type 5	7.5%
14	Plaited rib		one cotton yarn + + two yarns type 5	6%
15	Plaited rib		one cotton yarn + + one yarn type 5	4.5%
16	Plaited rib	one wool yarn + + one yarn type 2	one wool yarn + + one yarn type 5	4.5%
17	Plaited rib		one wool yarn + + two yarns type 5	6%
18	Plaited rib		one wool yarn + + two yarns type 5	7.5%
19	Plaited jersey		one wool yarn + + three yarns type 5	7.5%
20	Plaited jersey		one wool yarn + + two yarns type 5	6%
21	Plaited jersey		one wool yarn + + one yarn type 5	4.5%

– body protection against the action of unfavourable specific work place factors.

In order to characterize the 21 knitted fabric samples, complete sets of tests were conducted for the following parameters: weight (g/m^2), density (wales/10 cm, rows/10 cm), thickness (mm), air permeability (l/m^2/s), water vapour permeability (%), thermal conductivity (mW/mK), thermal resistance (m^{2}KW), shielding factor (*S*), discharge time (s) (table 2 and table 3).

In order to realize an experimental model, the electrical resistance for sample no. 7 has been determined at Fraunhofer Research Institution for Modular Solid-State Technologies, Germany.

Electrical measurements on textiles

The measured resistance of the processed textile R_f was in the range of a few MΩ/cm, which represents a proper and useful value [10].

Figure 1 shows the analysed textile sample. The magnified image on the right reveals the structure of

Table 2

CHARACTERISTICS OF KNITTED SAMPLES								
Sample no.	**Two-layer structure**	**Type of yarns combination**	**F_1 yarns combination**	**F_2 yarns combination**	**Weight,** g/m^2	**Density**		**Thickness,** mm
						rows/ 10 cm	wales/ 10 cm	
5	plaited jersey	cotton yarns combination	one cotton yarn + one yarn type 2	one cotton yarn + + one yarn type 3	470	43	92	1.58
3				one cotton yarn + + two yarns type 3	495	41	90	1.63
7				one cotton yarn + + one yarn type 4	487	44	86	1.63
6				one cotton yarn + + two yarns type 4	508	44	79	1.69
1				one cotton yarn + + three yarns type 4	525	43	84	1.62
10				one cotton yarn + + one yarn type 5	597	47	81	1.62
11				one cotton yarn + + two yarns type 5	589	47	82	1.66
12				one cotton yarn + + three yarns type 5	634	47	75	1.74
21		wool yarns combination	one wool yarn + one yarn type 2	one wool yarn + + one yarn type 5	521	47	71	1.65
20				one wool yarn + + two yarns type 5	562	47	69	1.65
19				one wool yarn + + three yarns type 5	603	46	69	1.66
2	plaited rib	cotton yarns combination	one cotton yarn + one yarn type 2	one cotton yarn + + two yarns type 3	728	38	60	3.41
4				one cotton yarn + + three yarns type 3	825	37	63	3.34
8				one cotton yarn + + one yarn type 4	705	37	61	3.52
9				one cotton yarn + + two yarn type 4	834	37	65	3.32
15				one cotton yarn + + one yarn type 5	785	37	61	3.39
14				one cotton yarn + + two yarn type 5	828	36	59	3.58
13				one cotton yarn + + three yarn type 5	878	36	59	3.61
16		wool yarns combination	one wool yarn + one yarn type 2	one wool yarn + + one yarn type 5	766	36	57	3.33
17				one wool yarn + + two yarn type 5	802	34	60	3.53
18				one wool yarn + + two yarn type 5	846	33	59	3.57

the sample. In order to characterize the fabric, electrical measurements were performed on yarns. The tested yarn- samples are represented in figure 2. In the magnified image on the right part of the figure, carbon covered fabrics can be observed.

Test setup and electrical measurements

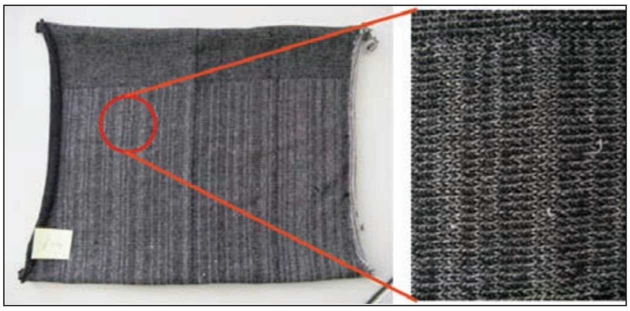
In order to characterize the electrical resistance of fabrics and yarns, two-point and four-point electrical measurements were performed.

Two point electrical measurement

The principle of the two point measurement is represented in figure 3. The voltage is applied through the two clips and the corresponding current through the yarn is measured. Applying Ohm's law the resistance can be calculated. The contact to the probe is done by two clips which add their resistance R_c to the resistance of the yarn R_{yarn}, leading to the total resistance $R_t = R_{yarn} + 2R_c$. The introduced error can be

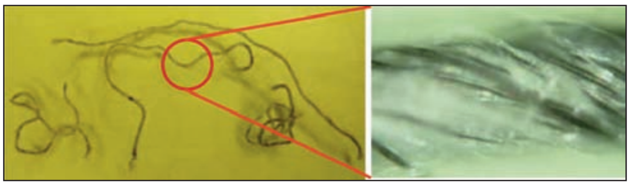
Table 3

CHARACTERISTICS OF KNITTED SAMPLES								
Sample no.	**Air permeability**, l/m²/s	**Water vapor permeability**, %	**Thermal resistance**, m²K/W	**Thermal conductivity**, mW/m·K	**Shielding factor**, *S*	**Discharge time**, $t_{1/2}$ (F_1)	**Discharge time**, $t_{1/2}$ (F_2)	**Conductive yarn percentage**, %
5	593.4	39.1	0.03913	40.25	0.84	0.0227	0.0228	4%
3	646	41.6	0.03901	41.65	0.92	0.0251	0.0228	5%
7	484.6	42.5	0.03354	48.65	0.82	0.0274	0.0246	5%
6	475.8	40.9	0.03447	49.05	0.83	0.025	0.0232	6%
1	539	38.3	0.03869	41.85	0.9	0.0249	0.0268	6%
10	374.4	36.2	0.03269	49.55	0.72	0.0258	0.0273	4.5%
11	395	35.9	0.04444	37.35	0.70	0.0252	0.0258	6%
12	401.4	34	0.03395	51.25	0.81	0.0235	0.0263	7.5%
21	643	34.2	0.04291	37.45	0.80	0.0253	0.0282	4.5%
20	601	34.8	0.04099	40.25	0.73	0.0227	0.024	6%
19	647.6	30.06	0.03567	46.55	0.80	0.0251	0.0255	7.5%
2	667.8	37.6	0.04950	69.05	0.82	0.0245	0.0245	5%
4	581.2	40.8	0.04805	69.45	0.82	0.027	0.0238	6%
8	654.8	38.1	0.05052	69.75	0.78	0.026	0.0268	4.5%
9	495.8	35.8	0.04664	71.15	0.90	0.0234	0.0241	6%
15	454.2	30.1	0.05481	61.85	0.81	0.0238	0.0274	4.5%
14	576.6	28.5	0.05732	62.45	0.83	0.0255	0.0248	6%
13	609.8	32.4	0.05726	63.05	0.7	0.0241	0.0228	7.5%
16	593.6	29.3	0.07881	42.25	0.86	0.0252	0.0244	4.5%
17	800.8	29.5	0.07801	45.25	0.80	0.0236	0.0269	6%
18	872.2	28.6	0.07786	45.85	0.82	0.0227	0.0241	7.5%



a ***b***

Fig. 1: *a* – tested fabric sample; *b* – magnified image



a ***b***

Fig. 2: *a* – tested yarn sample; *b* – magnified image

quite large, if the value of yarn resistance and contact resistance are of similar magnitude.

Four point electrical measurement

To overcome this, the four point measurement technique should be applied. The principle of the four point measurement is represented in figure 4. The voltage is applied through the two clips F_l and F_h and the corresponding current through the yarn is measured. To measure only the resistance of the yarn, R_{yarn}, two additional sense clips, S_l and S_h are connected to the sample and are used to measure the

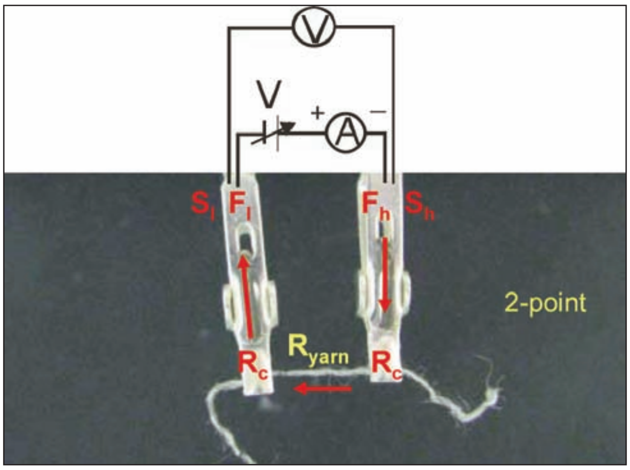


Fig. 3. Two point measurement technique for yarn electrical resistance

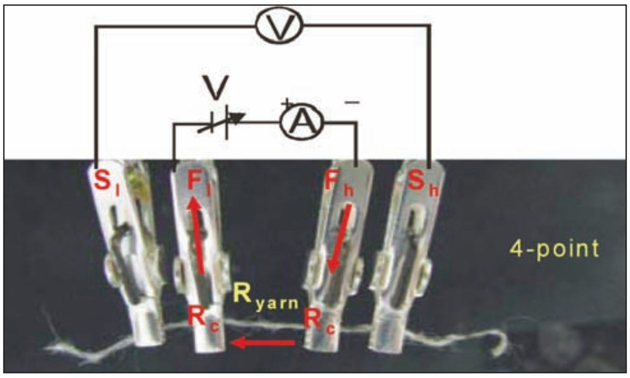


Fig. 4. Four point measurement technique for yarn electrical resistance

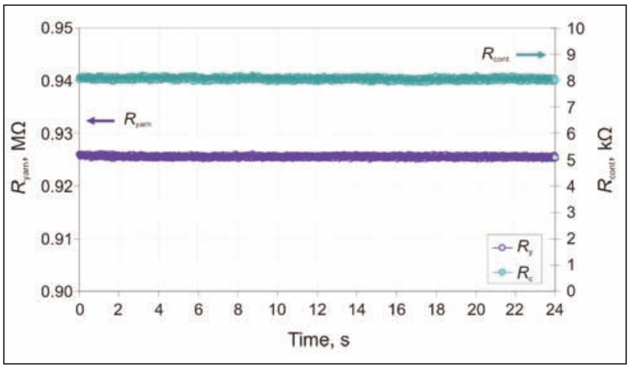


Fig. 5. Electrical resistance measurement of R_{yarn} and R_c, for an applied voltage of V_{appl} = 10 V, yarn length l_y = 5 mm

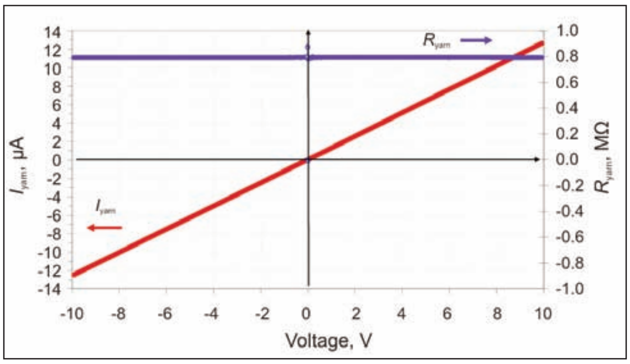


Fig. 6. Linear *I* - *V* characteristic during voltage sweep >> ohmic resistance of yarn: *l* = 5 mm, voltage sweep –10 V – +10 V

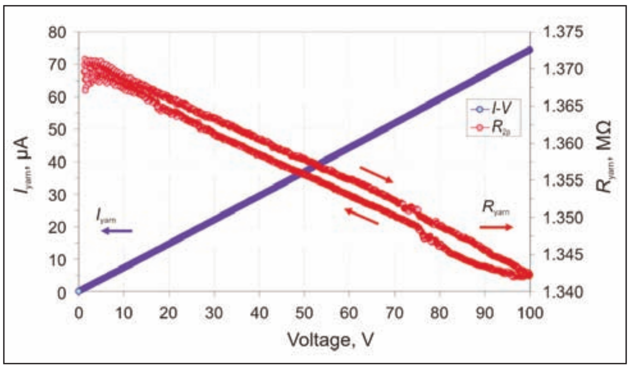


Fig. 7. R_{yarn} during voltage sweep, hysteresis due to self-heating of yarn: *l* = 5 mm, voltage sweep 0 –100 V – 0 V

voltage drop across the yarn. Since there is nearly no current flowing through the sense clips $R_t = R_{yarn}$. Figure 5 indicates the two measured resistances. It can be observed that in this case. In this case the contact resistance can be neglected and a two point measurement is sufficient.

Electrical measurements on yarns

To determine the DC-behaviour of the yarn resistance, a DC-sweep was performed. Figure 6 shows the behaviour of the yarn during a double voltage sweep up from –10 to +10 V. The current-voltage characteristic is linear and the resistance is ohmic and constant over the whole voltage range. But a voltage sweep from 0 V to +100 V leads to self-heating of the yarn. The resistance decreases due to self-heating and the negative temperature coefficient of resistance, TCR of the carbon covered fibres of the yarns. This behaviour is presented in figure 7, underlining the reversibility of the self-heating process for this high voltage value.

The thermal behaviour of the yarn was analysed by applying a constant voltage until steady state was reached. The yarn temperature was recorded by an IR-camera. For an applied voltage of 200 V the yarn temperature reaches approx. 95°C, as represented in figure 8. No visible changes could be observed after cooling down.

Electrical measurements on fabrics

Using the same test-setup as for yarns, only small samples of fabrics could be measured, as shown in figure 9. A voltage sweep from negative to positive values of the applied voltage reveals the linear behaviour of the current-voltage characteristic and the constant resistance values over this voltage range. This behaviour could be observed for both orientations of the fabric, as represented in figure 10 and figure 11. Applying a constant voltage of 200 V self-heating of the fabric was also observed, as depicted in figure 12. The temperature reached only 27.3°C, due to the higher thermal mass of the fabric than that of a single yarn.

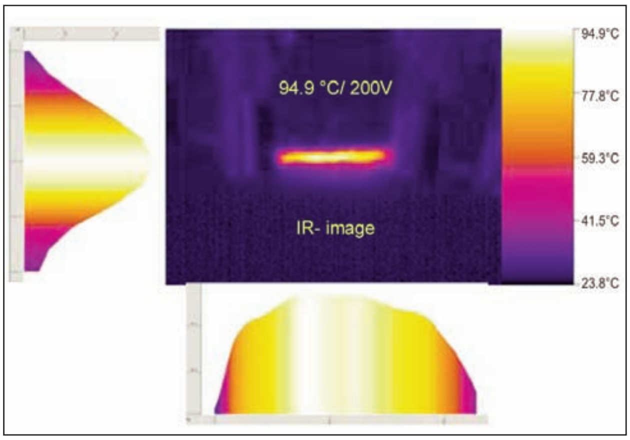


Fig. 8. Temperature distribution during self-heating of yarn

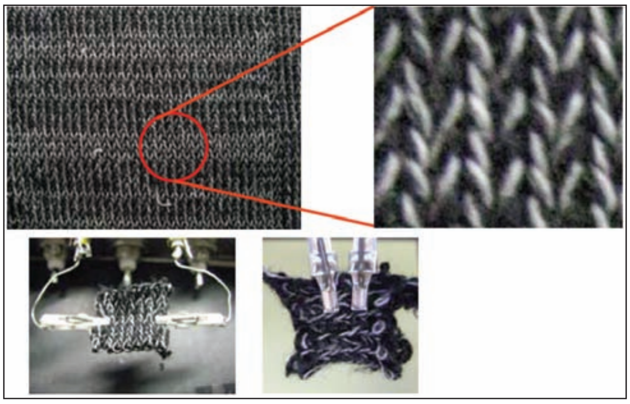


Fig. 9. Surface electrical resistance measurement of fabric with two-point technique, two orientations of fabric

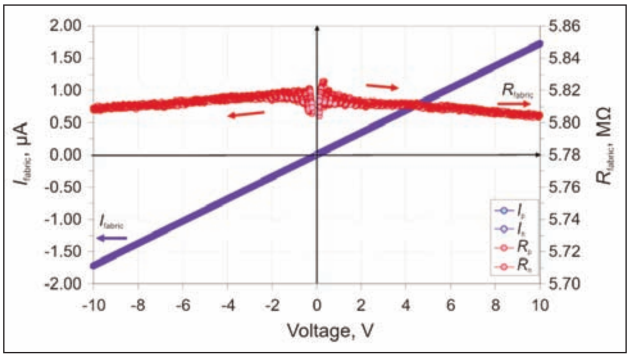


Fig. 10. Linear current - voltage characteristic during voltage sweep >> ohmic resistance of fabric, first orientation

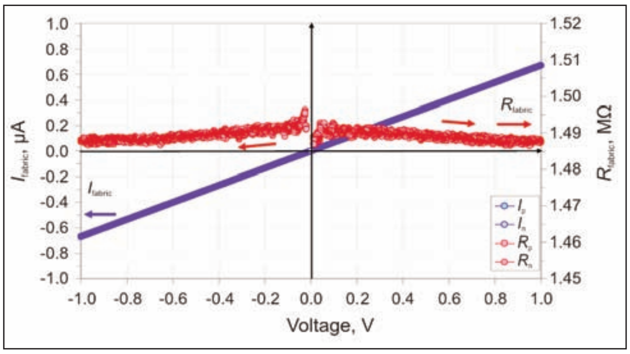


Fig. 11. Linear current - voltage characteristic during voltage sweep >> ohmic resistance of fabric, second orientation

CONCLUSIONS

When using a rib structure, voluminousness of the fabric will be higher due to the spatial arrangement of the stitch elements, due to an increased amount of incorporated air into the knitted structure. These aspects favour on the one hand a very high thermal comfort and on the other hand an effective air flow, respectively perspiration vapours between body and environment.

Electrostatic shielding factor has no significant differences for the analysed samples, sample 9 from rib structure group and sample 3 from jersey structure group show the best possible attenuation of the electrical load.

The presence of type 2 yarn on the front face 1 determines the performance improvement by using the yarns 3, 4 and 5 for all knitting configurations, knitting structure does not significantly influence the ability of electrostatic discharge.

The variation limits for water vapour permeability are between 28.5% and 42.5%, statistical events that agglomerate in the optimal zone indicate the presence of the cotton yarn in rib structure. Air permeability causes sensations of warm and cool of clothing products, the best value being obtained by samples 10, 11, 12 (374–400 l/m^2/s), characterized by the presence of cotton yarn and conductive yarn with nylon filaments superficially saturated with carbon particles.

Simple electrical measurements, like two point and four point DC-measurements reveal the good electrical behaviour of the yarn and fabric of the tested samples, when containing carbon covered fibres.

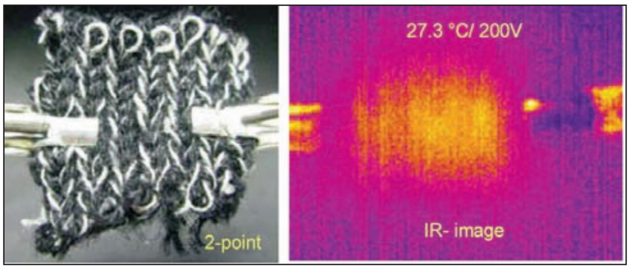


Fig. 12. Visible and IR image of fabric during self-heating under high voltage application

The measured resistances are in the MΩ – range and are sufficient to avoid electrical charge build-up in the fabric.

ACKNOWLEDGEMENT

This work was supported by a grant of the Romanian National Authority for Scientific Research, CNDI – UEFISCDI, project PCCA 179 – 2012 "Haine ESD realizate din fibre cu miez conductor tricotate bistrat" (ESD garments made of two-layer knitted fibres with conductive core).

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DOCUMENTARE

Noi tehnologii

TRATAMENT DE IGNIFUGARE A ȚESĂTURILOR

Compania specializată în peliculizări de înaltă tehnologie ***Alexium International Group Ltd.,*** din Greer, S.U.A., a lansat două produse chimice de ignifugare, fără conținut de halogen, destinate țesăturilor tehnice. O soluție de mare eficiență, *Nycolon FR*, utilizată deja pe piața echipamentelor de lucru și a celor militare din S.U.A., este pusă la dispoziția clienților din întreaga lume, pentru a conferi rezistență la flacără țesăturilor realizate din amestecuri de poliamidă și bumbac.

Nuvalon este un produs conceput special pentru țesăturile din amestecuri de poliester și bumbac, disponibil pe piețele din întreaga lume. Tratamentul aplicat asigură o protecție ignifugă durabilă pentru amestecurile obișnuite de țesături, care au un conținut de până la 65% poliester.

Tratamentul elaborat de compania Alexium a fost aplicat pe țesături obținute din amestecuri de poliester și bumbac. El a fost validat în cadrul unui test de producție la Universitatea de Stat din Raleigh, Carolina de Nord, și s-a constatat o performanță foarte bună la încercarea la flacără verticală, chiar și după 50 de cicluri de spălare/uscare.

Alexium a anunțat încheierea unui contract important de comercializare a produselor chimice FR către *Essex Flameproofing Ltd*. (Euroflam), din Horndean – Marea Britanie, o companie specializată în finisarea textilelor de interior, a stofelor de mobilă și a materialelor pentru tapițerie. Euroflam va folosi aceste produse pentru producția internă și va coordona proiecte cu companiile mari specializate în finisarea textilelor și cu comercianții de țesături din Marea Britanie.

Melliand International, martie 2014, nr. 1, p. 14

Evaluation of impact behaviour of composite materials using Taguchi method

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REZUMAT – ABSTRACT

Evaluarea comportamentului la impact al materialelor compozite, utilizând metoda Taguchi

Studierea comportamentului la impact al materialelor compozite este mare importanță, având în vedere solicitările dinamice la care sunt supuse acestea pentru diverse aplicații. Materialele compozite ranforsate cu structuri tricotate tridimensionale au o largă utilizare în domeniul articolelor tehnice. Principalele lor avantaje sunt reprezentate de formabilitatea foarte bună, anizotropia controlată și un raport optim între masă și rezistență. În lucrare sunt analizate materiale compozite avansate, ranforsate cu structuri tricotate tip sandwich, cu diferite dimensiuni ale celulei, realizate din Kevlar și Twaron, fire de in și o matrice termorigidă. Comportamentul la impact de intensitate redusă al materialelor compozite ranforsate cu structuri tridimensionale, tricotate din bătătură, a fost modelat prin metoda Taguchi, în scopul optimizării caracteristicilor semnificative pentru acest tip de impact. Rezultatele obținute în urma modelării au fost validate prin studii experimentale.

Cuvinte-cheie: materiale compozite, impact de intensitate redusă, metoda Taguchi

Evaluation of impact behaviour of composite materials using Taguchi method

Impact behaviour is a major target in designing advanced composite materials because composites are often used in applications which imply dynamic loads. Composite materials reinforced with 3D knitted fabrics present a wide range of applications in the technical field. Their main advantages refer to excellent formability, controlled anisotropy and good mass/strength ratio. The paper considers advanced composite materials reinforced with sandwich fabrics with various cell sizes, made of Kevlar, Twaron and linen yarns, and thermoset matrix. Low velocity impact behaviour of composite materials reinforced with 3D weft knitted fabrics is modelled using the Taguchi method based on orthogonal arrays, in order to maximize the composite characteristics significant for this type of impact. The results obtained through Taguchi analysis are validated by experimental data.

Key-words: composite materials, low level impact, Taguchi method

Technical textiles are high performance textiles with increased functionality that are produced primarily for their functional characteristics and technical performance rather than for their aesthetic or decorative properties which are less or not important.

The applicability of composite materials reinforced with technical textile structures in engineering has grown exponentially in recent decades. A composite material is an advanced structure made from at least two distinct materials that are combined at macroscopic scale. The applications where textile reinforced composites are suitable cover an extremely wide range of domains – from buildings [1], to industrial applications [3], [7], to protective equipment [6] or even medical products.

Composite materials are primarily a suitable choice for producing light-weight structures due to their excellent weight/stiffness and weight/ ratio properties. The mechanical properties of composite materials are influenced by the mechanical properties of matrix, producing technology, properties of reinforcement preforms, adhesion between matrix and reinforcement preforms and fibre volume fraction.

Composite preforms refer to textile materials that can be obtained by knitting, weaving, braiding and nonwoven technologies. In order to select the optimal technological process for producing composite performs both strengths and weaknesses of each must be taken into account. The potential and the use of 3D knitted fabrics for technical applications are documented throughout literature [4]. Knitted preforms present the advantages of a superior mass/resistance ratio, low implementation time, low amount of resulting wastes and better control of the final shape and the quality of the product.

The sandwich fabrics with connection through knitted layers (single or double) are characterized by a complex geometry, for which the shape and dimensions of the cross section depend on the connecting layer [4]. The shape of the connecting layer can be different, varying from rectangular to elliptic, *V* shaped, trapeze etc.

The principle of producing sandwich fabrics requires knitting the two independent layers, and then the connecting layers while the outer layers production is stopped. The recommended needle selection for knitting the outer and connecting layers is 1:1.

In order to obtain composite materials different techniques can be used:

– embedding of reinforcement material in a matrix, that can be represented by a macromolecularsubstance or a colloidal solution or suspension with coagulation properties;
– reinforcing the matrix with layers of reinforcement materials, resulting a laminate composite.

In the last decades a significant research effort has been made to study the impact behaviour of composite materials with textile reinforcements. Several researchers [2], [8], [9] have studied the problem by examining the material properties before and after the impact. Impact load can be classified [9] in low, medium, high and hyper velocity, according to the impactor speed, as presented in figure 1. The effects of low impact velocity have gained importance in real life situations, such as bird hits, tool drop or contact with other materials that can cause internal invisible damage, irremediably affecting the mechanical properties of composites.

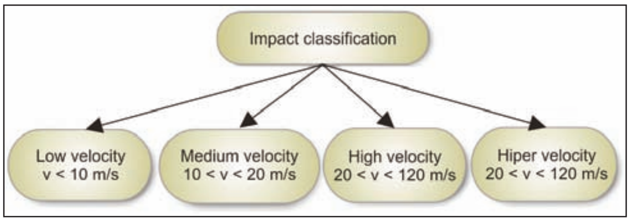


Fig. 1. Impact classification

Up to now, the evaluation of impact behaviour of composite materials reinforced with sandwich weft knitted fabrics has been made through the classical methods that involve varying one parameter at a time and keeps the rest of them constant. These methods require a large number of experimental samples, are time consuming and do not provide information regarding the interactions between parameters. In order to solve these problems, Taguchi method was designed based on the concept of orthogonal arrays. Although Taguchi method was successfully applied in many fields, such as chemical engineering, electronics, genetic algorithm, so far it has not been widely used in textile areas.

The statistically based Taguchi method was used to identify the importance of factor designs and to suggest an optimized design that can produce the optimal impact resistance for composite materials reinforced with 3D sandwich knitted fabrics.

DEVELOPMENT OF 3D COMPOSITE MATERIALS

The experimental work focused on the production of *U* shaped sandwich knitted preforms, presented in figure 2. The 3D knitted fabrics were programmed on a Sirix station and produced on Stoll CMS 320 TC weft flat knitting machine, gauge 10E.

The outer layers are knitted independently on selected needles. When initiating the connecting layer the even needles are inactive, when knitting the outer layer the uneven needles receive a tuck loop that binds the outer and connecting layer together. After finishing the final row in the connecting layer, the stitches are transferred on the uneven needles of the opposite bed, ensuring the sandwich fabric integrity. The knitting of the outer layers restarts. The cell height depends on the number of rows in the outer layers, while the cell width is determined by the number of rows in the connecting layer.

The outer layers were made using Kevlar-Inox and Twaron yarns and for connecting layers linen and Kevlar-Inox yarns were used. The fabric compactness, required to increase the volume fraction of the composites, was improved by inserting Twaron yarns, as illustrated in figure 3.

The 3D composite materials studied in this paper were produced using epoxy EPICURE 04908 resin as matrix and Vacuum Assisted Resin Transfer Moulding Technology (VARTM). The composites were cured at room temperature (23ºC). The mixing ratio of the matrix had 30% EPIKURE Curing Agent 04908 and 5% Dearing agent BYK A535. Table 1 presents the main characteristics of the resin used.

The final aspect of composite materials reinforced with weft knitted sandwich preforms is exemplified in figure 4. All knitted fabrics and composite materials were produced at University of Minho, Portugal.

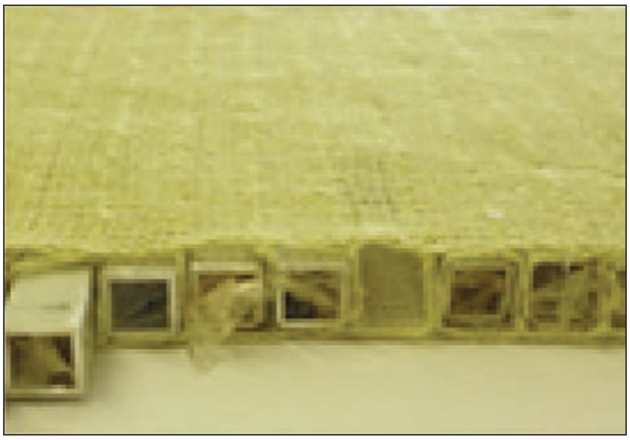


Fig. 2. *U* shaped sandwich knitted fabric

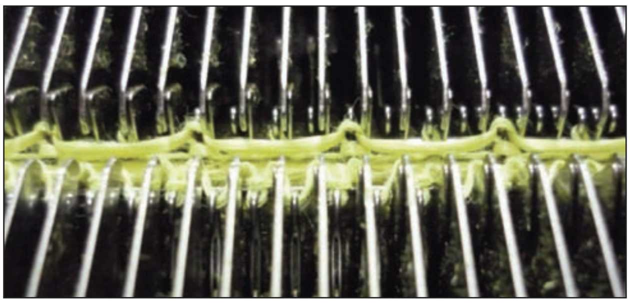
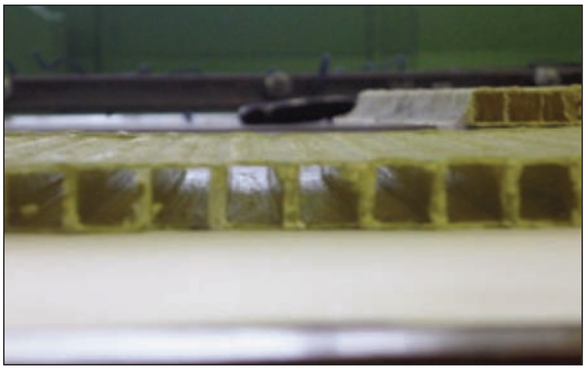
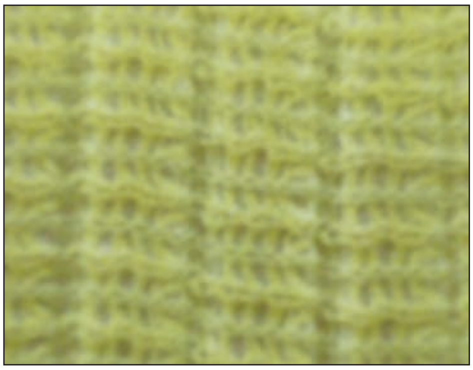


Fig. 3. Production of sandwich fabric



a



b

Fig. 4. Composite material with 3D textile reinforcements – aspect of outer and connecting layers

Table 1

PRINCIPAL CHARACTERISTICS OF EPOXY EPICURE 04908 RESIN		
Properties	**Unit**	**Epoxy Epicure 04908**
Viscosity at 25°C	Mpa.s	10
Density	g/cm^3	1.15
Tensile strength	MPa	74
Tensile strain	%	9.4
Tensile modulus	MPa	2 900
Flexural strength	MPa	112
Modulus in flexure	MPa	3 100
Water absorption after 24 hours, 23°C	pbw	0.18
Water absorption after 168 hours, 23°C	pbw	0.432

Table 2

SIGNAL FACTORS SELECTION		
Levels	**Structure**	**Cell dimension**
1	Kevlar	10 x 10 mm
2	Kevlar/Linen	10 x 15 mm
3	Kevlar/Twaron	-
4	Kevlar/Twaron/Linen	-
Symbol	*A*	*B*

EVALUATION OF IMPACT BEHAVIOUR USING TAGUCHI METHOD

The experimental matrix contains two input variables, one at four levels and the other at two levels, as presented in table 2. The selection of the signal factors was carried out so that the studied process can achieve the expected performance and have a minimum sensitivity to noises. The current study targeted the influence of signal parameters on the impact resistance of advanced composite materials.

The first two columns of table 3, noted *A* and *B* represent the signal factors (structure and cell dimension), while the following two, noted N_1 and N_2 are the noise factors (impact resistance of composite materials). Signal to noise ratio (noted *S/N* ratio) is a measure used in science and engineering that compares the level of desired signal parameters to the level of background noise parameters.

The main purpose in applying the *S/N* ratio is to find the optimum combination of signal parameters that influence the system so that the *S/N* ratio is maximized. This way, these parameters become system control factors [5]. The *S/N* ratio can be determined for the following cases:

Table 3

EXPERIMENTAL DESIGN USING L_8 ARRAY AND EXPERIMENTAL RESULTS							
Experiment	***A***	***B***	N_1	N_2	**Mean**	**Standard deviation**	***S/N* ratio**
1	2	2	1 419.35	1 395.68	1 407.515	16.7372	62.9681
2	3	1	891.22	964.08	927.65	51.5198	59.3276
3	3	2	1 161.28	1 076.5	1 118.89	59.9485	60.957
4	4	2	1 170.29	1 054.69	1 112.49	81.7415	60.8907
5	2	1	2 430.59	2 385.23	2 407.91	32.0744	67.6316
6	1	2	1 181.29	1 225.12	1 203.205	30.9925	61.6025
7	1	1	1 245.3	1 287.35	1 266.325	29.7338	62.0473
8	4	1	2 024.49	1 998.21	2 011.35	18.5828	66.0692

- smaller the better

$$\frac{S}{N} = -10\log(s^2 + \bar{y}^2) \qquad (1)$$

- nominal is the best

$$\frac{S}{N} = 10\log\left[\left(\frac{\bar{y}^2}{s^2}\right) - \frac{1}{n}\right] \qquad (2)$$

- larger the better

$$\frac{S}{N} = -10\log\left[\sqrt{\bar{y}^2}\left(1 + 3s^2 \cdot \sqrt{\bar{y}^2}\right)\right] \qquad (2)$$

where:
s is standard deviation;
y – nominal value;
$\bar{y}$ – average of determined values;
n – number of runs.

After the analysis of orthogonal array models, the signal parameters and their specific levels, L_8 orthogonal array has been considered adequate. After defining the signal and the noise factors, the statistical analysis and determination of the mean, standard deviation and *S/N* ratio can be carried out, as shown in table 3. The *S/N* ratio has been calculated using the formula defined for larger the better case.

The significance of the signal factors reported to *S/N* ratio is given after performing the Taguchi analysis. The classification of influence level, presented in table 4, is: maximum influence – the factor *A* (knitted structure) and minimum influence – the factor *C* (cell dimension).

The results that were obtained after the statistical analysis for *S/N* ratio and mean was performed are graphically represented in figure 5 and figure 6.

Table 4

RESPONSE TABLE FOR *S/N* RATIOS LARGER IS BETTER FORMULA		
Level	***A***	***B***
1	61.82	63.77
2	65.3	61.60
3	60.14	-
4	63.48	-
Delta	5.16	2.16
R_5 ank	1	2

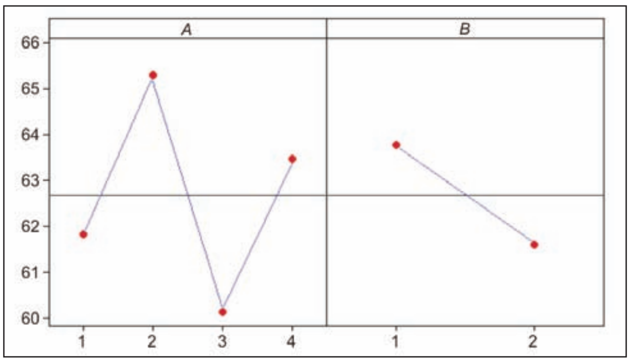


Fig. 5. Main effects plot for *S/N* ratio

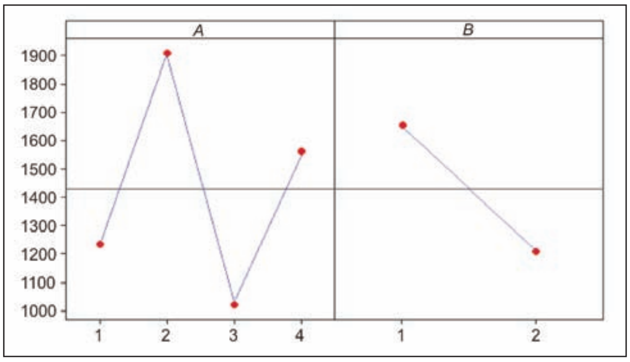


Fig. 6. Main effects plot for means

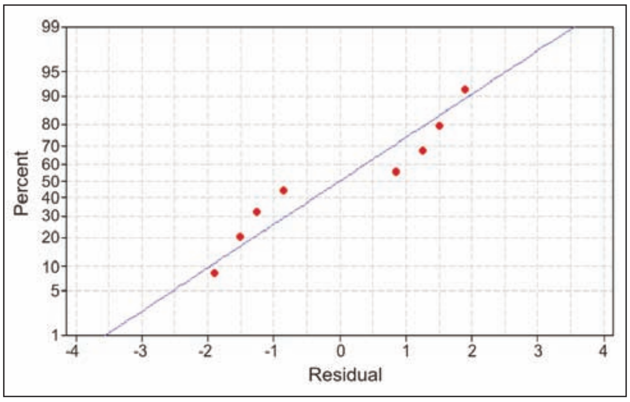


Fig. 7. Normal probability plot

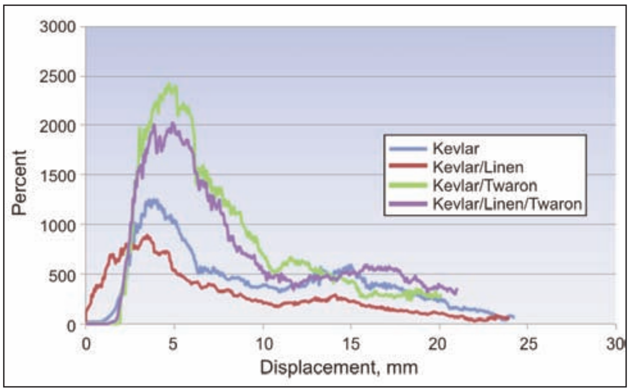


Fig. 8. Force displacement curves

The optimal combination of signal parameter in order to obtain the higher value for *S/N* ratio is A_2B_1, meaning a composite material reinforced with Kevlar-Inox and Twaron yarns and a cell dimension of 10 x 10 mm.

MODEL VALIDATION

In order to determine the accuracy of Taguchi model that was presented normal probability plot was drawn (fig. 7). It can be noted that the residual values distribution reported to the median is normal.

The reliability of presented model was also confirmed by performing a number of experimental tests taking into account the intervals for the defined signal parameters. Samples have been tested at low velocity impact using an Ceast Fractovis Plus 2 000 testing machine, an impactor with hemispherical head with diameter of 20 mm and an impact height point of 750 mm according to ISO 6603 and ISO 7765 standards. The more significant results are graphically exemplified in figure 8.

From the graphical analysis of the experimental samples, Taguchi results correspond to the experimental results. The level of forces in the system determined through testing corresponds to variant A_2B_1, defined in the Taguchi analysis and presents the maximum value – 2 430.59 N.

CONCLUSIONS

Composite materials reinforced with sandwich *U* shaped knitted fabrics with various cell dimensions and epoxy resin were manufactured using VARTM technique. The low velocity impact behaviour has been evaluated.

The orthogonal array was defined based on structure composition and cell dimension as signal parameters an impact resistance of composite materials as noise factors. The best results have been obtained for a composite material reinforced with Kevlar-Inox and Twaron yarns and a cell dimension of 10 x 10 mm.

The model was validated based on experimental results obtained by testing sandwich composites produced with similar parameters in similar technical conditions.

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Comparative analysis of real and virtual garment fit

KRISTINA ANCUTIENE

REZUMAT – ABSTRACT

Studiu comparativ privind probarea reală și virtuală a articolelor de îmbrăcăminte

Articolul prezintă un studiu comparativ asupra probării reale și virtuale a articolelor vestimentare. Folosind programul „Modaris“ (CAD Lectra) a fost creat un articol de îmbrăcăminte, pentru o siluetă simplă și dreaptă. Au fost analizate caracteristicile fizico-mecanice ale unei țesături prespălate, din 100% in. Evaluarea deformării la efort și purtare a articolului de îmbrăcăminte s-a efectuat pe patru persoane, având o conformație similară tipodimensiunii 164 – 88 – 92. Pentru a realiza o analiză comparativă între gradul de potrivire a unui articol de îmbrăcăminte pe un corp real și unul virtual, s-a folosit programul „Modaris 3D Fit“ (CAD Lectra), realizându-se probarea produsului pe manechini 3D virtuali, având dimensiuni similare purtătorilor reali, luați în studiu. Rezultatele privind confortul și deformarea la efort, prezentate de purtătorii reali ai articolului de îmbrăcăminte, au fost comparate cu cele oferite de software.

Cuvinte-cheie: probă virtuală, 3D CAD, formă vestimentară, e-magazin

Comparative analysis of real and virtual garment fit

In this article investigation of real and virtual garment fit is presented. The dress of simple straight silhouette was constructed using the program „Modaris“ (CAD Lectra). The dress fitting was performed using mechanical parameters of the prewashed 100 % linen fabric. Four respondents, whose figures were similar to the typical figure 164 – 88 – 92, were used to evaluate the ease and strain deformations of the dress. For the comparison of real fitting process with the virtual, 3D mannequins of respondents were made using the program „Modaris 3D Fit“ (CAD Lectra) and virtual try-on of the dress was done. The scores that were given by the respondents wearing the real dress were compared to the ease and strain results that were given by the software.

Key-words: virtual try-on, 3D CAD, garment fit, e-shop

Garment CAD is research area which has attracted high attention nowadays [1]. 3D mannequin and 3D garment are the most interesting topics in textile engineering, computer graphics and 3D garment CAD [2]. 3D simulation of the garments allows simulation of all pre-production elements, as stitching, fitting and visualization of textile materials on the garment.

Complex 3D simulations of garments placed on virtual mannequins especially depend on mechanical and physical properties of different types of textile materials and mechanical model of the fabric as well as constructed garment pattern [3]. In our earlier research [4] virtual garment fit was investigated and it was proved that virtual garment would fit different on the human body depending on textile materials properties. For the investigation of virtual garment fit distance ease values in patterns and virtual garment, also strain in three girths – bust, waist and hip were measured. For this reason measurable and calculable parameters were derived.

Abram Zver et. all [5] also concluded that virtual presentation has a great potential as a tool to evaluate the fit of a garment in a relatively simple and quick way. Furthermore, the progress achieved in the field of virtual presentations of garments presents a very important and promising link for e-tailoring chain of the future. By using 3D virtual prototyping product development time and the cost involved in multiple iterations of sample garment production could be reduced.

The popularity of online shopping has risen hugely nowadays in garment retail [6]. One main problem of garment online shopping is that consumers must select a size without real try-on of garment what often results in fit dissatisfaction [7]. Fit is a primary reason why consumers fail to purchase garments. Virtual fitting room could solve this problem because sizes differ, depending on country, brand and so on. Virtual fit could rise confidence of garment purchase via internet and could decrease returns. But still there are differences between real and virtual garment during try-on if seen from wearers sensation side. Therefore investigations are made to compare virtual and real try-on processes [3], [15] – [17], so that in the future virtual try-on could become precise and informative for consumer.

Rudolf A. et. all [3] researched the appearance and shape differences between the virtual and real dress, also the influence of mechanical parameters of a fabric on garment behaviour in different garment areas. Comparison of a real and virtual dress confirms the usability of virtual prototyping of garments. The similarities of both dresses and their draping behavior were obvious. They concluded that virtual dress can be used for estimating the fit and appearance of a dress before it is actually produced.

Garment fit could be evaluated referring to experts' opinions [7] – [9] or wearers' responses [10] – [12]. Rating scales with the critical fit areas are often used to evaluate garment fit, so virtual simulation technology is used for garment fit evaluation from quantitative and interview data [7]. For fit evaluation distance ease, shape of the garment and strain distribution is estimated without producing the garment [7], [13]. 3D technology can reduce or eliminate costly sample making and fitting process. However, there is no systematic investigation of 3D garment simulation for fit analysis, especially taking into the account fabric properties [14].

Usually virtual try-on in e-shop is performed in static posture and only few companies (Optitex, Tukatech) offer dynamic try-on possibility. In the later cases garment fit could be evaluated more precisely, also fabric behavior during movements could be estimated. Garment fit could be evaluated measuring strain, ease also pressure. These technologies are used in many 3D garment CAD systems (Lectra, Tuka3D and others) but just few e-shops offer this possibility because of many troubles related to consumers perception of garment strain or ease values looking to the virtual garment. Therefore research in this area should be done to suggest the ways for garment fit and comfort conception in virtual fitting room.

So, the goal of this research is to perform comparative analysis between the virtual fit of the garment and the fit on a real person using respondents survey and virtual try-on. The paper presents the construction of straight silhouette woman's dress with the use of program “Modaris 3D Fit” (CAD Lectra) and virtual appearance on a virtual parametrical model of a woman's body. The dress was also sawn and tried onto a real person, after which comparative analysis between real garment fit and virtual woman's dress was performed.

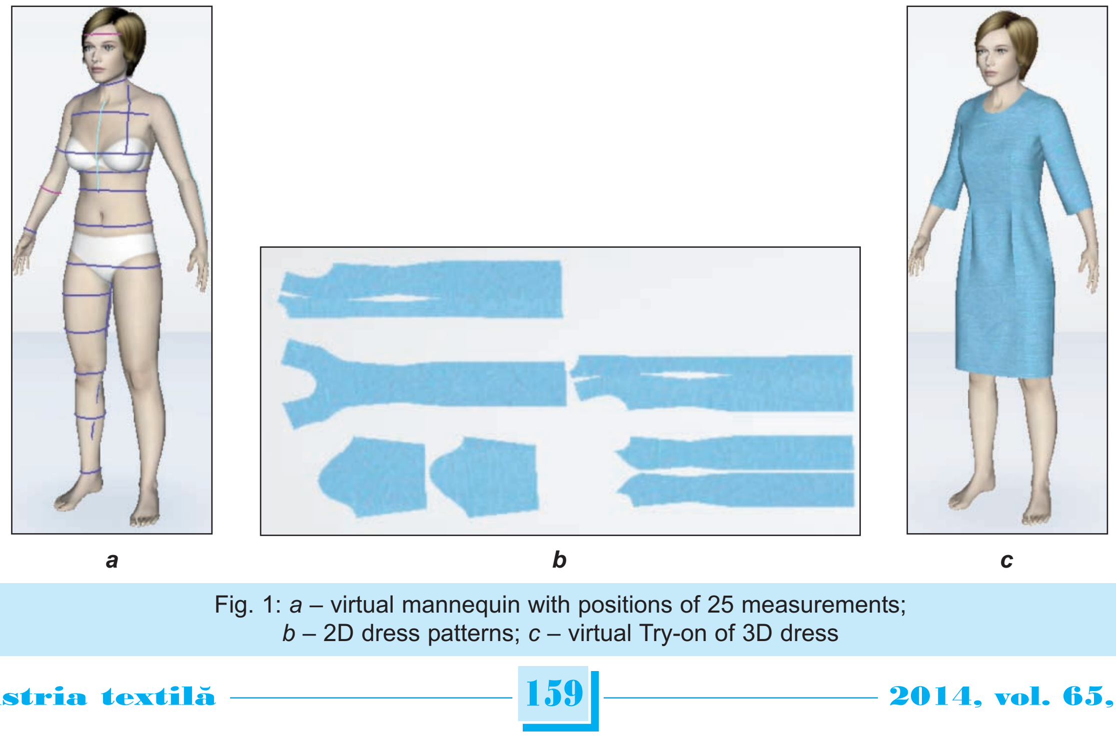
MATERIALS AND METHODS

For the garment fit investigations four female bodies were selected with height, bust and hips measurements different less than a half of meaningless interval limits, e.g. 3 cm for the height and 2 cm for bust and hips. These bodies were assigned to the same 164-88-92 size group and were named as the respondents R_1, R_2, R_3 and R_4.

The close-fitting straight dress with the 2/4 sleeves was constructed in software “Modaris” (CAD Lectra) also was produced for the same 164-88-92 size in order to make comparison between a real and virtual garment fit. In order to apply virtual dress on a human body, a virtual parametric model of a female body using the software “Modaris 3D Fit” was prepared. The adaptation of virtual body measurements in regards to real model is simply and precisely because 25 body measurements could be used for the input (fig. 1 *a*). So, virtual mannequin has the same body measurements as the real body for those measurements, which could be adjusted. For the virtual simulation 2D patterns are imported into 3D software (fig. 1 *b*) and after selecting textile material for garment virtual Try-on could be done (fig. 1 *c*).

According to the characteristics (less stretchable, stable, stiff) which give clarity and precision, the 100% linen fabric was chosen for the virtual and real dress. Fabric was pre-washed to give more softness and then was tested using KES (Kawabata evaluation system) equipment. The main mechanical and structure characteristics, necessary for virtual dress simulation in “Modaris 3D Fit” software, are presented in table 1.

Real and virtual models wearing tested real and virtual dresses made from 100% linen fabric are presented in figure 2. For the investigations respondents were fitted dress and made different dynamic movements that were presented at the survey (static posture at the beginning, then raising arms to the sides, then to the forefront, step forward, sitting and bending to the side) and completed the survey. The respondents marked the spots by red colour if they felt the strain and by the blue colour if they felt the



a *b* *c*

Fig. 1: *a* – virtual mannequin with positions of 25 measurements; *b* – 2D dress patterns; *c* – virtual Try-on of 3D dress

Table 1

CHARACTERISTICS OF TESTED FABRIC								
Area density, g/m^2	**Thickness,** mm	**Composition**	**Bending rigidity,** *B*, 1^{-6} Nm		**Tensile strain,** *EMT*, %		**Shear rigidity,** *G*, Nm^{-1}/°	
			warp	*weft*	*warp*	*weft*	*warp*	*weft*
215	0.45	100 % linen	5.35	5.87	19.37	18.98	0.33	0.36

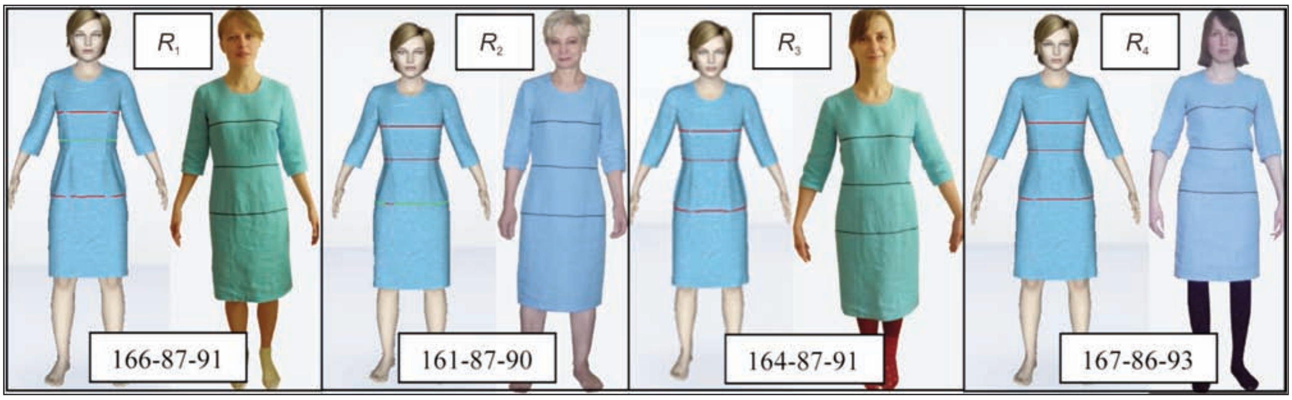
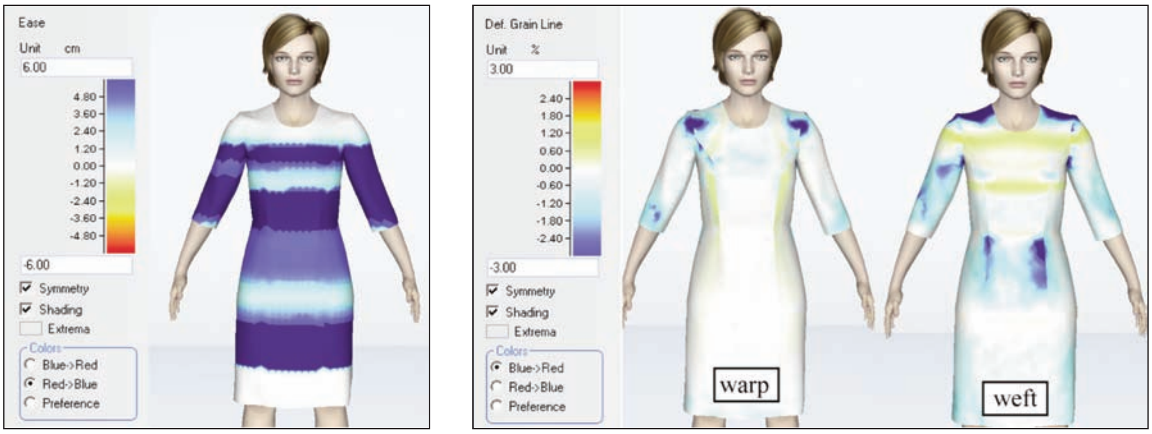


Fig. 2. Real and virtual respondents with the tested real and virtual dress



a *b*

Fig. 3: *a* – distribution of the ease in virtual dress;
b – distribution of tensile strain in warp and weft directions

ease. Also they were asked to score the feeling of the strain and the ease: *1* – weak ("a little"), *2* – medium ("more") and *3* – strong ("very much"). If respondent feel comfortable then didn't score any girth and any garment area. The feeling is subjective because for the one 5 cm ease value in bust area could be big, when for the other – suitable. This was done purposeful because during the gathering of the garments in the shop customers usually have subjective estimation before garment purchasing. The same dynamic movement was repeated several times if it was necessary for the respondents to be confident in garment fit evaluation.

Virtual dress fit was evaluated by measuring garment ease (cm) and tensile strain (%) in three main girths – bust, waist and hip, also by analyzing distribution of ease and strain in the whole virtual garment (fig. 3). For the comparison of different virtual garments the same ease value of 6 cm and the same strain value of 3% were chosen. Strain distribution was also evaluated differently in warp and weft directions. Virtual simulation was done three times for the reliability of results and the coefficient of variation didn't exceed 2.6%.

RESULTS AND DISCUSSIONS

After the real dress fit evaluation virtual dress fit investigation was done and comparative analysis was performed. Summary of the results is presented in table 2 and table 3 according to the tested posture also analyzed girth or garment area. Results of real

Table 2

RESULTS OF EASE INVESTIGATION IN VIRTUAL AND REAL DRESS									
Respondent		R_1		R_2		R_3		R_4	
Posture	**Girth**	**Ease,** cm	**Score**	**Ease,** cm	**Score**	**Ease,** cm	**Score**	**Ease,** cm	**Score**
1 – static	bust	3.19	-	4.96	-	4.18	-	4.66	-
	waist	10.97	-	8.20	**2**	9.10	-	10.12	**2**
	hip	5.23	-	6.23	**2**	5.27	-	3.25	-
2 – hands rising to the sides	bust	3.69	-	3.75	-	3.21	-	4.76	-
	waist	11.14	-	7.80	**2**	8.92	**1**	10.46	**2**
	hip	5.37	-	6.96	**2**	5.40	**1**	3.35	-
3 – hands rising to the forefront	bust	3.84	-	4.64	**1**	3.27	-	4.88	-
	waist	9.43	-	7.24	**2**	7.13	-	8.41	**2**
	hip	5.78	-	6.25	**2**	5.75	-	3.65	-
4 – step forward	bust	3.02	-	4.03	-	4.22	-	4.69	-
	waist	11.31	-	8.47	**2**	9.08	-	10.13	**2**
	hip	6.67	-	7.31	**1**	6.69	-	4.36	-
5 – sitting	bust	3.93	-	5.67	-	4.10	-	5.74	-
	waist	11.44	-	9.21	**1**	9.56	**2**	10.95	-
	hip	21.22	-	9.60	-	11.03	-	9.27	-
6 – bending to the side	bust	4.28	-	4.71	-	4.81	-	5.12	-
	waist	11.79	-	9.89	-	9.76	-	11.42	-
	hip	7.02	-	8.17	**2**	7.29	-	5.16	-

Table 3

RESULTS OF STRAIN EVALUATION IN VIRTUAL AND REAL DRESS													
Posture	**Analyzed area**	R_1			R_2			R_3			R_4		
		Strain, %		**Score**	**Strain, %**		**Score**	**Strain, %**		**Score**	**Strain, %**		**Score**
		warp	*weft*		*warp*	*weft*		*warp*	*weft*		*warp*	*weft*	
1 – static	armpit area in back	0.56	–5.69	1*	0.57	–4.18	-	0.56	–5.96	-	0.39	–6.51	-
2 – hands rising to the sides	shoulder	–14.8	–9.80	3	–5.95	–4.93	2	–14.3	–5.80	3	–6.59	–4.49	-
	armpit	–0.58	0.34	-	–2.03	0.25	-	0.21	0.88	3	–2.99	0.28	-
	shoulder-blade area	–0.69	–5.62	2	–1.65	–4.31	-	–0.26	–0.86	-	–0.12	–1.28	-
3 – hands rising to the forefront	shoulder	–12.13	–4.09	3	–3.42	–3.94	2	–11.2	–7.27	2	–11.2	–6.66	-
	armpit in front	0.50	–3.12	-	0.36	–0.86	-	2.18	–5.19	3	0.58	0.68	-
	armpit area in back	1.23	0.80	3	0.28	0.30	-	1.55	1.25	3	0.64	0.55	-
4 – step forward	armpit area in front	1.06	–0.38	3	0.32	–7.15	-	0.85	–3.74	1	0.45	0.54	-
	armpit area in back	–2.27	0.18	-	0.98	–3.87	-	–0.80	–7.60	-	–2.08	–11.5	-
5 – sitting	thigh	–0.34	0.73	3	–0.26	0.25	1	–0.25	0.32	3	–0.17	0.84	1
6 – bending to the side	left armpit in back	1.03	–10.3	1	0.96	–11.6	-	1.08	–12.9	-	1.24	–13.9	-
	left armpit in front	0.92	–0.42	-	0.64	0.94	-	1.88	–0.59	-	0.81	–0.42	-
	side	-6.05	–2.10	3	–7.04	–1.10	3*	–5.11	–1.60	3	–3.90	–0.91	3

* in red colour is scores of strain and in blue of compression

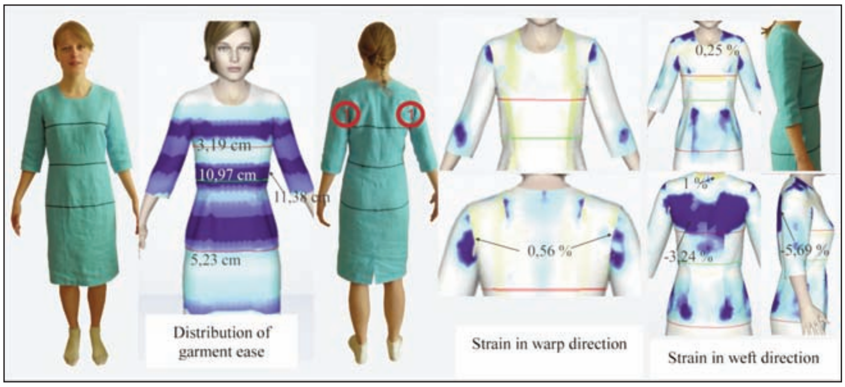


Fig. 4. Comparison between real and virtual dress fit for R_1 respondent in static posture

Fig. 5. Comparison between real and virtual dress fit for R_1 respondent during hands rising to the sides

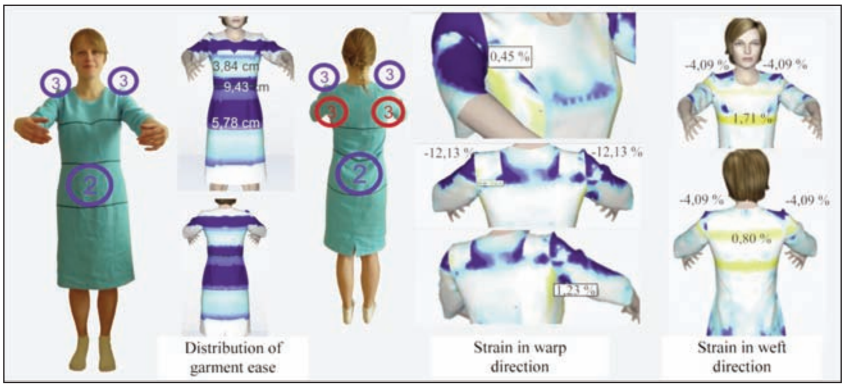


Fig. 6. Comparison between real and virtual dress fit for R_1 respondent during hands rising to the forefront

and virtual dress fit comparative investigation for respondent R_1 are presented in figures 5 – 9.

In static posture during ease evaluation respondent R_1 (fig. 4) noted that hips fit well (ease value here is 5.23 cm), when in the area under bust is too much ease (11.38 cm) but she didn't mark this area with the score. Ease value in bust girth for all respondents are in the range of 3.19 – 4.96 cm and they didn't feel discomfort therefore didn't give any score (table 2). It should be mentioned that respondent R_2 is a dressmaker so she has more understanding about garment fit than other respondents therefore she gave scores for ease in many postures comparing to the other respondents. Ease value in waist girth is too big 9.10 – 10.97 cm) for R_1 and R_3 respondents, but they didn't felt it. As shown, the strain results (table 3) in static posture only respondent R_1 gave score 1 at armpit area in back. In warp direction there is 0.56% tensile strain when in fabric weft direction at shoulder-blade area is compression of –3.24% (fig. 4). There was the concern that this respondent felt discomfort in back armpit. In this area strain of the same gauge was also evaluated for other respondents but they didn't feel it.

Referring to garment ease evaluation in three main girths (table 2) we can think that respondent R_1 felt comfortable in all tested dynamic movements because she didn't mark any score. Or it could be the case that she didn't concentrate on the girths because in many analyzed areas she scored ease or strain (table 3). In that case we can state that ease feeling in main girths was not such important for her because other garment areas presented discomfort.

For respondents R_2 and R_4 ease evaluation in three main girths have tendency remain the same as in static posture, e.g. if respondent feel ease during static posture she feel ease and during many movements at the same girth. This could be stated except sitting and bending to the side movements because in these cases ease value distribute differently and evaluation of the ease differs. Respondent R_2 felt medium ease (score 2) in waist and hip girths during postures *1 – 4*, while sitting she felt less ease (score 1) in waist and comfortable ease (no score) in hip girth. Herewith she marked strain (score 1) at thighs (table 3). In summary it could be stated that static posture of virtual garment in internet shop virtual fitting room don't provide right information about garment ease during movements and it should be supplemented by sitting and bending movements for better understanding about garment fit.

During fit evaluation when hands were rising to the sides respondent R_1 gave score 2 at shoulder-blade area (table 3), where compression of –5.62% arise in fabric weft direction (fig. 5). High value of compression (–4.31%) at this area was evaluated also for respondent R_2 but she didn't marked this area. All respondents marked shoulder area as discomfortable (score 2 or 3) except respondent R_4 though shoulder rising was clearly seen for all. This discomfort feeling is due to the high compression in both fabric directions (table 3).

In armpit area only respondent R_3 marked strong strain feeling (score 3). This could arise because of the smallest ease value (3.21 cm) at bust girth (table 2) what originated tensile strain at bust area in weft direction of 2.5%. Therefore during hands rising to the sides discomfort was felt in armpit area. Strain value in armpit area in weft direction is the highest (0.88%), but value is comparatively small.

During fit evaluation when hands were rising to the forefront (fig. 6) respondent R_1 marked shoulder area as discomfortable (score 3) the same as other two respondents R_2 and R_3 (table 3). In this area we see large compression values in warp and weft directions and can say that discomfort feeling is almost the same as in posture hand rising to the sides.

During hands rising to the forefront R_1 and R_3 marked strong strain feeling (score 3) in armpit area. It is interesting that respondent R_1 during hands rising to the sides didn't feel here discomfort. Strain value in warp direction here is higher (1.23%) and because of different strain distribution during hands rising to the sides and to the forefront, later movement rise higher strain values in fabric warp at back also weft direction at shoulder-blade areas. So, it could be stated that more important movement is hands rising to the forefront than to the sides for right garment fit evaluation.

Fig. 7. Comparison between real and virtual dress fit for R_1 respondent during step forwards

Fig. 8. Comparison between real and virtual dress fit for R_1 respondent during sitting

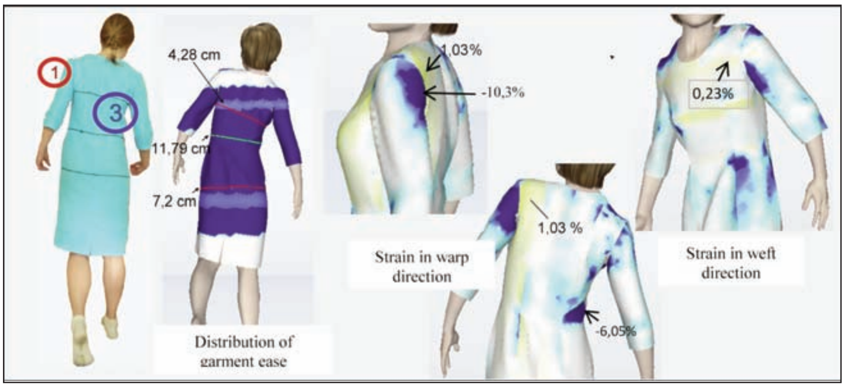


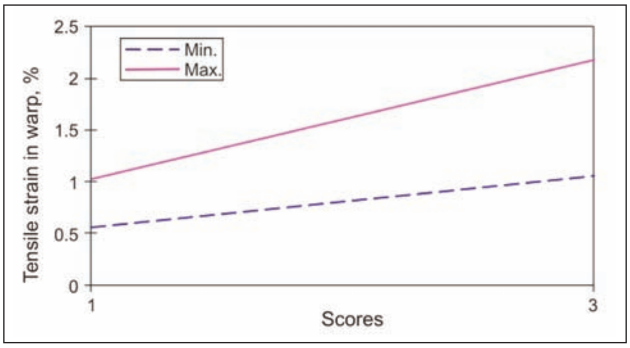
Fig. 9. Comparison between real and virtual dress fit for R_1 respondent during bending to the side

During fit evaluation when stepping forward (fig. 7) respondent R_1 marked only strong strain (score 3) at front armpit area, which was influenced by the tensile strain of 1.06 % in warp direction. Respondent R_3 felt small strain (score 1) comparing to posture when hands rising to the sides or forefront (score 3) because here one hand is rising to the front when other to the back. This redistributes strains in armpit area and discomfort feeling remains only in front armpit where strains act in warp direction.

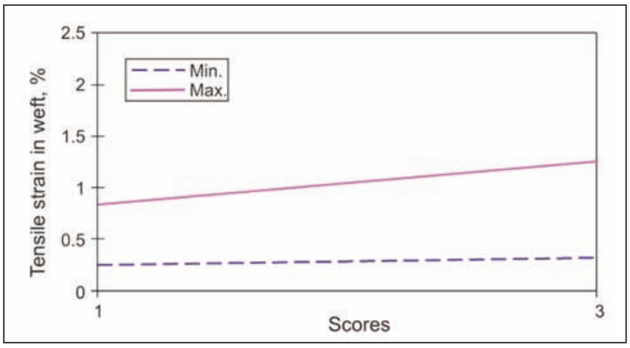
During fit evaluation, while sitting, all four respondents felt discomfort in thigh area (table 3). Respondent R_1 and R_3 marked it as strong discomfort (score 3). As we can see in virtual garment (fig. 8), during sitting ease value which was in static posture becomes negative, e.g. tightness (yellow colour) originate. For respondent R_1 this value is –2.01 cm, when for R_3 –2.59 cm. This is the main reason for discomfort feeling, because strain values in warp and weft directions are relatively small.

During fit evaluation when bending to the side all respondents marked strong ease feeling (score 3) because here fabric folds and reaches compression of –3.9 – 7.04% in warp direction.

Respondent R_1 marked small strain feeling (score 1) at sleeve head in left hand. We can see (fig. 9) that



a



b

Fig. 10. Range of tensile strain values at different scores in:
a – warp direction; *b* – weft direction

tensile strain in warp direction near shoulder is 1.03% and compression in sleeve head area is –10.3%. This could be the reason for discomfort feeling but other respondents didn't mark this area though tensile strain was similar (0.96 – 1.24%).

To summarize real and virtual garment fit results, ranges of minimal and maximal ease values were estimated in respect to respondents' scores. Respondents didn't give any score, e.g. they feel comfortable, when ease value vary from 3.02 up till 5.74 at bust girth, from 7.13 up till 11.79 cm at waist girth, and from 3.25 up till 21.22 cm at hip girth. Last ease value is related to sitting posture, when ease measuring tool in software measures all folds originated in garment. After elimination of ease values at this posture, range for hip is between 3.25 and 8.17 cm. It is obvious that very wide range of ease values is observed in girths when respondents didn't feel any discomfort. This is influenced not only by psychological factor but also by right understanding about garment ease.

Respondents give score 1 for ease which was between 8.92 and 9.21 at waist girth and between 5.4 and 7.31 at hip girth. Score 2 was given for ease between 7.24 and 10.46 at waist and between 6.23 and 6.96 at hip girth. Score 3 was not given at ease evaluation the same as scores at bust girth. It is seen that ease values at hip girth are smaller than waist at the same score. This means that conception of ease values at hip girth is better than at waist.

In strain investigation case for one posture more important is strain in warp direction, for other posture – strain in weft direction. For compression investigation influence of compression direction is also uneven. For example, during hands rising to the forefront comfort at armpit area in front is influenced by strain in warp direction when in back by strain in both directions. During hands rising to the sides more influence to armpit comfort have strain in weft direction while during step forward for the armpit area in front more influence have strain in warp direction. During sitting more important is strain in weft direction.

In summary could be stated that both strain in warp and weft directions are related to corresponding areas and in general case if strain increases scores increase too. In figure 10 we can see ranges between strain values in all postures when scores 1 and 3 were given (score 2 was not given in this research). We can see that strain in weft direction more influent respondents' conception of comfort sense than in warp because smaller values of strains were marked by the same scores.

CONCLUSIONS

The method presented, comparing real and virtual garment fit, showed that respondents' conception of strain in different garment areas are clear, but the sensation of ease are different. Such results may be influenced by psychological factor, different understanding about purpose of the garment (for some it must be comfortable, for some it must look gracefully). It is recommended that the scale of scores offers just two evaluations – "weak" and "strong", what could bring more understanding about comfort evaluation on the ground of strain and ease.

The increase of strain values in garment areas increase respondents evaluation scores. Strain in weft direction influent respondents' comfort sense conception more than in warp direction.

Static posture of garment which is the most common in internet shop virtual fitting room don't provide right information about garment ease and strain during movements and it should be supplemented by sitting and bending movements for better understanding about garment fit.

Further research in this area will be extended by real and virtual garment fit evaluation starting by virtual garment evaluation using the view of strain and ease distribution and after real garment fit estimation and comparison. For this purpose a larger range of mannequin sizes should be considered, to have wider range of strain and ease values.

So, virtual presentation has a great potential as a tool to evaluate the fit of a garment in a relatively simple and quick way. The progress achieved in the field of virtual presentations of garments presents a very important and promising link for e-tailoring chain of the future. By using 3D virtual prototyping product development time and the cost of sample garment production could be reduced. This paper also provides important information for the researchers to identify future research directions in 3D garment fit investigations.

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Mathematical and statistical modelling of project management performance

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REZUMAT – ABSTRACT

Modelarea matematică și statistică a performanței managementului de proiect

Managementul performanței reprezintă o abordare strategică și integrată a asigurării succesului de durată în activitatea organizațiilor, prin îmbunătățirea performanței organizației, echipelor și indiviziilor. Caracterul strategic rezultă din preocuparea pentru problemele mai generale cu care se confruntă organizația, pentru a funcționa eficient în mediul extern în care se află, potrivit direcției generale pe care dorește să o adopte în îndeplinirea obiectivelor pe termen mediu și lung. De altfel, instituirea oricărui program de management strategic are ca punct de pornire misiunea, viziunea, obiectivele și strategiile organizației, definite în fazele planificării strategice. Pornind de la acestea, ulterior sunt dezvoltate procese de evaluare și de măsurare a performanței.

Cuvine-cheie: performanță, management de proiect, modelare, statistică

Mathematical and statistical modelling of project management performance

Performance management is a strategic and integrated approach to long-term success for organizations, by improving the performance of the organization, teams and individuals. Strategic character comes from emphasis on the general problems the organization encounters, in order to function effectively, in its external environment and regarding the general direction it plans to adopt to accomplish its medium and long-term goals. In fact, the mission, vision, goals and strategies of the organization, defined in the strategic planning stages, are the starting point for any strategic management program. At this point, the performance evaluation and measurement processes are developed.

Key-words: performance, project management, modelling, statistics

Scientific research evaluation becomes extremely important as research is one of the main fields where gaps between economies worldwide can be reduced. The competitiveness of a country becomes a general goal, which can be accomplished by following two fundamental goals: generating and exploiting knowledge. Performance is one of the key terms frequently used in project management. However, there is no unanimously accepted interpretation of this term. There are various points of view regarding its meaning and what makes an organization efficient in project management.

The evaluation can contribute the most as far as policies regarding research, development and innovation are concerned, since success is not guaranteed and the implementation does not occur without obstacles in this field. There must be a balance between demand and offer for research evaluation, hence the necessity of efficient management and adequate planning.

When applied adequately, evaluation helps clarify some inherent uncertainties of complex situations. Social-economic development is not an exact science; it is a complex process, with numerous uncertainties. Selecting the goals and measures, designing the programs, implementing and ensuring their dynamic development require analysis, power of anticipation, feedback systems and support from various institutions, agencies and groups through scientists and researchers.

Bryde [1] proposed a model of project management performance evaluation whereby a project can have the best performance possible, with certain variations depending on the organization (PMPA model).

Romania, a developing country, needs new synchronized approaches of the developed world in order to improve its efficiency, reliability and success, which are mandatory for the business world. The success factors of projects evolve in a new dimension in line with the changes and according to the “survival of the fittest” principle. The principle of the competitive business environment is characteristic today. The project can be defined as “a transitional society” created to exclusively generate a result or a service Barad et al. [2]. The transition refers to the fact that the project has an end date. This means that the exclusiveness of the project’s final results is different from the results of the organization’s other functions. Bowen et al. [3] use case studies to suggest that the only way organizations can accomplish important goals is through projects.

Project management is a vital and imperative task to evaluate project management performance. In this case, it is important to make the distinction between project performance and project management performance. Both are interconnected, but they are completely different from each other, so these terms are not to be confused [4]. Although project management (PM) may seem “poor”, the actual project can be “prosperous”, just as a project can be useless despite great project management [5].

EXPERIMENTAL PART

Designing evaluation methodologies based on economic theoretical bases allows a link between measuring the results and the marginal impact of the policies on the economic welfare. In most cases, in order to simplify the dynamic complex of scientific and technological development, the approaches of the evaluation adopt the linear innovation model. For certain purposes and in the context of more complex innovation models, these are used in the evaluation. One of the basic attributes of this conceptual evaluation model for project management performance is the identification and analysis of the relation between a set of short and medium-term performance factors and the impact factor of research and development projects through canonical correlation analysis. Through this model, the contributions of the predictive factors on the predicted variable can be identified. Thanks to canonical correlation analysis, the contributions of the qualitative factors can be extracted quantitatively.

The model is adequate when the external qualitative criterion (dependent variables) can be estimated based on information regarding the qualitative attributes for every subject (independent variables).

In this study, all the qualitative measurement variables of each performance factor are observed on a Likert ordinal scale (the answers to the questions were “yes”/“no”. The binary code was: “yes” = 1; “no” = 0 (http://en.wikipedia.org/wiki/Truth_value 'Classical logic').

The performance factors of this study consist in impact input and output. The input and output factors are the project management performances in the institution, interpreted in the short and medium term, while the impact factor is the performance at international and national scale, interpreted as a long-term performance. In order to identify the relation between the four items of the project’s impact (dependent variables) as a function of the project’s performances, management and results (independent variables), the canonical correlation analysis was used.

In this study, we have used the statistical canonical correlation method to identify the relation between the four items of the project’s impact (dependent variables) as a function of the project’s performances, management and results (independent variables). In other words, we used it to analyze the relation between the impact factor in the long run and the performances obtained in the short and medium run by institutions with national and/or European R & D financing. The proposed evaluation model is original because it will provide a group analysis and a risk analysis for these institutions, crucial feedback on innovative performances in R & D financing. This conceptual evaluation model proposes a set of four factors and various measurable variables (4 binomial qualitative variables for every factor), part of the conceptual evaluation model with the horizontal axis (the goals of the evaluation) and vertical axis (the evaluation methodology). These generate performance in project management and the institutional performance to monitor/manage projects.

In addition, group analysis and risk analysis are applied to the result of the canonical correlation analysis in order to explore similar characteristics of certain groups of institutions according to the type, size and main or secondary research-development activity. In the last section, the results of the study are discussed and the conclusions are drawn. The results of this research are expected to contribute to a better understanding of the impact of R & D financing.

The research purpose and methodology

- The research methodologies used in this study were: investigations, interviews and questionnaires, as well as canonical correlation analysis. In order to identify the relation between the four items of the project’s impact (dependent variables) as a function of the project’s performances, management and results (independent variables), canonical correlation analysis was used.
- This multivariate analysis technique was used in order to investigate two sets of variables (dependent and independent). This statistical method reduces type I errors [6].
- The canonical correlation analysis was performed using the Statistica program (StatSoft Inc., v. 8.0), with a significance threshold of 5%, so probabilities smaller than 0.05 were considered statistically significant.

The investigation methods

For the research, at first, 143 experts with 10–35 years of experience in evaluations of national and European projects were chosen to participate in a survey, to collect data and information, in order to establish a conceptual model of project management performance evaluation in the organizations that benefited from national and/or European financing. Due to the time deficit, we originally used the e-mail survey for anthology data. Through this method, we collected answers to a series of key questions regarding the national and European evaluation system, good and bad practices. However, there were also certain problems that reduced the answer efficiency rates. For instance, certain experts took a lot of time before sending the e-mails, so data elaboration was delayed. In order to deal with this challenge, phone interviews were used as well: representatives of institutions in charge of monitoring and evaluation in Romania, managers of institutions that have and are carrying out nationally and internationally financed projects, project managers, directors of research and development departments from these institutions. As a result, the response rate was better, of a higher quality and quicker. The answers were collected from 69 institutions. This method was relatively more productive, allowing us to collect more answers. The descriptive investigations were made in order to establish an image of the current problems and the relational investigations were developed for an empiric analysis.

The questionnaire method

This research was based on conventional sampling (which was not based on probability), used as a modus operandi to make the research process quicker, in order to obtain more respondents (162) and so that the questionnaire would be filled in quickly and economically both on the online platform and on the e-mail of this questionnaire's author. The institutions selected to fill in the questionnaire were: public institutions and authorities (11), research-development institutes (27), private organizations (15), universities (16) whose main or secondary activity is research and development (table 1).

The purpose of this study is to find the "relation and impact between the financing resources, PM leadership, the PM staff, the PM policy, the PM strategy, the resources used, the life cycle of PM and the project management performance", impact and sustainability of the results of scientific research projects.

The system of indicators was defined based on the structure of four fundamental components in performance evaluation, taking into account the approach of the performance in terms of efficiency/ effectiveness.

These components are:

I. Resources attracted in the project

Resources (*S*) are characterized by 4 binomial qualitative variables (S_1, S_2, S_3, S_4):

- S_1 – identifying the financing sources;
- S_2 – evolution of the absorption of funds;
- S_3 – developing the capacity of human resources;
- S_4 – developing the research infrastructure.

II. Project performance management in the organization

Management (*M*) is characterized by 4 binomial qualitative variables (M_1, M_2, M_3, M_4):

- M_1 – PM policies and strategies;
- M_2 – PM leadership;
- M_3 – the project team staff;
- M_4 – the PM key indicators.

III. The relevance and visibility of the results of scientific research projects

Results (*R*) are characterized by 4 binomial qualitative variables (R_1, R_2, R_3, R_4):

- R_1 – types of results;
- R_2 – result indicators;
- R_3 – identifying other results;
- R_4 – the volume of scientific production.

IV. The impact and sustainability of the results of scientific research projects

Impact (*I*) is characterized by 4 binomial qualitative variables (I_1, I_2, I_3, I_4):

- I_1 – scientific impact;
- I_2 – social impact,
- I_3 – economic impact,
- I_4 – technological impact.

The criteria according to which the performances of the indicators identified in each of these four groups are evaluated, through this system, are:

- the international relevance of the performances;
- the national relevance of the performances;
- management performance.

In the study, the following factors associated with R & D performance were conceived: the financing resources, management, results, as well as their impact factor.

We adopted the four factors and measurement variables based on two aspects. Firstly, we regrouped the input and output indicators into a set of short and medium-term performances, to discover their relation to the impact factor. In various studies we have taken part in, we classified the R & D fund for performance according to three aspects: input, output and the impact factor. Nevertheless, although the input and output indicators directly indicate the level of accomplishment of a R & D project and the impact factor presents the indirect accomplishments and its long-term effects, we will try to combine the input and output indicators into a set of short and medium-term performances. Additionally, in this particular study, the inputs and outputs are the performances of project managers from institutions and the impact represents the national/European performance.

For this purpose, we will analyze the relation between a series of short and medium-term performance factors and the impact factor. The second aspect refers

Table 1

THE EXPERIENCES AND WORKPLACES OF THE RESPONDENTS

Institutions	No. of institutions	No. of respondents from the institutions	Experience in the field, years
Public institutions and authorities	11	29	5-15
Research-development institutes	27	62	10-35
Private organizations	15	15	5-10
Universities	16	56	5-25
Total	69	162	Average: 16

to the fact that we will use quantification methods to identify the non-linear model between the two groups of performance factors, because the contribution of the qualitative factors can be extracted through canonical correlation analysis.

Description of the tools

The feedback questionnaire contains 16 questions (a question for every binomial variable). The answers to the questions were "yes"/"no". The binary code was: "yes" = 1; "no" = 0 (http://en.wikipedia.org/wiki/Truth_value 'Classical logic').

Mathematical model

If $S_{1.1}$, $S_{1.2}$, $S_{1.3}$ and $S_{1.4}$ are Bernoulli (0/1) variables, then $S_1 = S_{1.1} + S_{1.2} + S_{1.3} + S_{1.4}$ is a binomial variable (0, 1, 2, 3, 4). Resources (S) are characterized by 4 binomial qualitative variables (S_1, S_2, S_3, S_4). Management (M) is characterized by 4 binomial qualitative variables (M_1, M_2, M_3, M_4). Results (R) are characterized by 4 binomial qualitative variables (R_1, R_2, R_3, R_4). Impact (I) is characterized by 4 binomial qualitative variables (I_1, I_2, I_3, I_4).

The interaction model can be seen as [7]: Resources (S_1, S_2, S_3, S_4) · Management (M_1, M_2, M_3, M_4) Results (R_1, R_2, R_3, R_4) = Impact (I_1, I_2, I_3, I_4), therefore identifying the contributions of the predictive factors on the predicted variable is possible. Through canonical correlation analysis [8], [9], the contributions of qualitative factors can be quantitatively extracted (fig. 1).

Statistical analysis

The variables were summarized as absolute frequencies and percentages. The associated 95% confidence intervals were calculated for the percentages, under the assumption of the binomial distribution [10]. The association in the 2 x 2 contingency table between pairs of items was analyzed through Cochran test. Null statistical hypothesis: the answer to an item is independent from the answer to the second item. A p value associated to Cochran statistic, smaller than the applied significance threshold (5% in this case, so $p < 0.05$), indicates a statistical significant dependence as far as the answer to the two investigated items is concerned. The canonical correlation analysis was made through the program Statistica, v. 8, with a significance threshold of 5%.

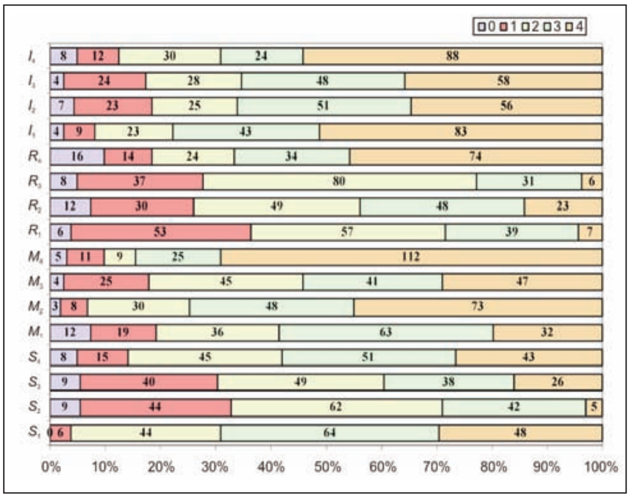

Fig. 1. The values of the response functions for: impact ($I_1 – I_4$), results $R_1 – R_4$, management ($M_1 – M_4$) and resources ($S_1 – S_4$)

RESULTS AND DISCUSSIONS

Evaluation of project performance

Summarization of the positive answers ("yes"): absolute frequency (percentage [a confidence interval of 95%]) is presented in table 2.

Summarization per goal: where S_1, S_2, S_3, S_4, M_1, M_2, M_3, M_4, R_1, R_2, R_3, R_4, I_1, I_2, I_3, I_4 = countif (array, "yes") is presented in table 3.

Canonical factor no. *1* is an ascending factor for all the observed qualitative variables and variables R_1 and M_2 are associated with the main canonical factor (F_1), which suggests that they are the most important for it. Additionally, in relation to canonical factor no. *2*, response variable I is separated in I_1 (a negative projection in relation with F_2 factor) and I_2, I_3 and I_4 (positive projections in relation with F_2 factor). Therefore, if F_1 factor is a measure of the central tendency in practically all the measured variables (both in the impact measurable and predicting variables), factor F_2 is a measure of variability in accomplishing the impact goals (figures 2–4).

F_2 factor also contains the management observables almost all the time (except for M_2 in $F_1 – F_2$ projection), so it expresses the "Management" factor to a great extent.

Table 2

SUMMARIZATION OF THE POSITIVE ANSWERS ("YES"): absolute frequency (percentage [a confidence interval of *95%*])				
Reso-urses	***1* *n* (% [*IC* 95%])**	***2* *n* (% [*IC* 95%])**	***3* *n* (% [*IC* 95%])**	***4* *n* (% [*IC* 95%])**
S_1	160 (98.77 [95.68; 99.99])	124 (76.54 [69.14; 82.71])	96 (59.26 [51.24; 66.66])	98 (60.49 [52.47; 67.90])
S_2	124 (76.54 [69.14; 82.71])	68 (41.98 [33.95; 49.99])	102 (62.96 [54.94; 70.37])	20 (12.35 [7.41; 18.52])
S_3	92 (56.79 [48.77; 64.81])	104 (64.20 [56.18; 71.60])	52 (32.10 [24.70; 40.12])	108 (66.67 [58.65; 74.07])
S_4	134 (82.72 [75.93; 88.27])	118 (72.84 [65.44; 79.63])	106 (65.43 [57.41; 72.84])	72 (44.44 [36.42; 52.47])

Note: *n* is absolute frequency; IC 95% is confidence interval of 95%

Table 3

CSUMMARIZATION PER GOAL:
where S_1, S_2, S_3, S_4, M_1, M_2, M_3, M_4, R_1, R_2, R_3, R_4, I_1, I_2, I_3, I_4 = countif (array, “yes”)

Factor	Variable	Abb.	Frequency of the variable				
			0	***1***	***2***	***3***	***4***
Project performance	Do you think the project tool helped attract financing funds?	S_1	0	6	44	64	48
	Which of the financing funds between 2007 and 2013 generated an increasing absorption through the project tool in your organization?	S_2	9	44	62	42	5
	Your projects contributed to the development of human resources’ capacity through… ?	S_3	9	40	49	38	26
	Did your projects aim to develop the research infrastructure through the following actions?	S_4	8	15	45	51	43
Project management	Name the project management (PM) policies and strategies in your organization.	M_1	12	19	36	63	32
	PM leadership described in your organization.	M_2	3	8	30	48	73
	Was/is the staff in the project team in the following situations: …?	M_3	4	25	45	41	47
	Did the following key indicators ($K_1 – K_4$) of project management exist?	M_4	5	11	9	25	112
Results of the projects	The project mainly focused on developing… ?	R_1	6	53	57	39	7
	Were the following result indicators planned/achieved in your projects?	R_2	12	30	49	48	23
	Do you agree with the following statements?	R_3	8	37	80	31	6
	Evaluate whether the volume of scientific production expressed through the number of articles/books you published had a significant increase.	R_4	16	14	24	34	74
Impact of the projects	To what extent do you agree with the following statements?	I_1	4	9	23	43	83
	Which of these statements do you agree with?	I_2	7	23	25	51	56
	To what extent do you agree with the following statements?	I_3	4	24	28	48	58
	Do you think the projects from your organization generated impact factors in the field?	I_4	8	12	30	24	88

Canonical factor no. *1* is an ascending factor for all the observed qualitative variables and variables R_1 and M_2 are associated with the main canonical factor (F_1), which suggests that they are the most important for it.

Additionally, in relation to canonical factor no. *2*, response variable *I* is separated in I_1 (a negative projection in relation with F_2 factor) and I_2, I_3 and I_4 (positive projections in relation with F_2 factor).

Therefore, if factor F_1 is a measure of the central tendency in practically all the measured variables (both in the impact measurable and predicting variables), F_2 factor is a measure of variability in accomplishing the impact goals.

F_2 factor also contains the management observables almost all the time (except for M_2 in $F_1 – F_2$ projection), so it expresses the “Management” factor to a great extent.

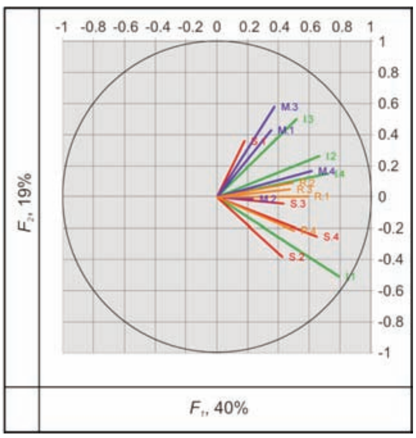

Fig. 2. Projection after factors F_1 and F_2

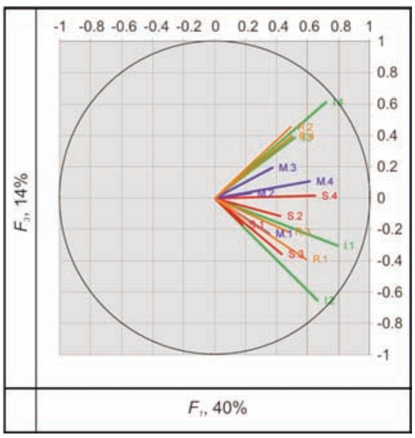

Fig. 3. Projection after factors F_1 and F_3

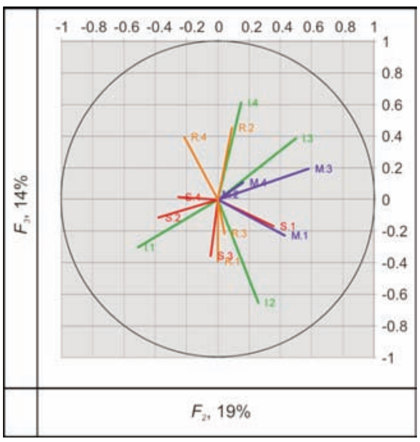

Fig. 4. Projection after factors F_2 and F_3

This study was carried out on a basic level, since there was no data available prior to this research.

CONCLUSIONS

Evaluation is an essential tool for good management of practices. It is a tool that not only helps measure the success of a program/project, but also contributes to its success. Research evaluation currently requires trans-disciplinary expertise and a new set of tools. In this thesis, the term “research evaluation” is approached as a wide concept, connected to the research financing process and the evaluation of the research process, as well as the result. Evaluation practices also vary according to the actors involved in the process and the method of evaluating RDI and the types of problems of research evaluation practiced in every country:

- The results show that using an evaluation model concept (identifying the goals/choosing the methodology of optimizing it in the implementation; identifying the factors/measurables and following the feedback of the evaluation) has a significantly positive impact on project management. The results obtained through the analysis show that every criterion has a significant association with PM, which proves the main hypothesis.
- Secondly, the key performance indicators (*KPI*) have the biggest impact on PM, followed by PM leadership in accomplishing the results planned in the project. All these results support the main hypothesis.
- This study also stressed that in relation to canonical factor no. 2, response variable *I* is separated in I_1 (a negative projection in relation with F_2 factor) and I_2, I_3 and I_4 (positive projections in relation with F_2 factor). Therefore, if F_1 factor is a measure of the central tendency in practically all the measured variables (both in the impact measurable and predicting variables), F_2 factor is a measure of variability in accomplishing the impact goals.
- F_2 factor also contains the management observables almost all the time (except for M_2 in $F_1 – F_2$ projection), so it expresses the “Management” factor to a great extent.
- The study also reveals that making performance evaluation efficient is also necessary. Through this study, the performance of the R & D financing programs was analyzed, using statistical methods, such as the canonical correlation method, which was used to analyze the relation between a series of short and medium-term performance factors and the impact on R & D projects (table 4 and table 5).
- Unlike previous studies, this study dealt with the categorical Likert-scale answers using the canonical correlation method to identify the relation between the four items of the project’s impact (dependent variables) as a function of the project’s performances, management and results (independent variables).
- This multivariate analysis technique was used in order to investigate two sets of variables (dependent and independent). This statistical method reduces type I errors [6].
- By grouping the types of institutions that received the questionnaire with the performance results of the four other factors, we discovered which group (type of organization) had poor or strong results. Out of the four categories, three groups (public institutions and authorities, research-development institutes, universities), except for the private organizations group have good performances for all the four factors. Throughout the analysis of the groups, we suggested that the companies within the private organizations group, which generally report poor performances for the four performance factors, should make efforts to achieve progress as far as every performance-related aspect is concerned. Moreover, this group should

Table 4

THE RESULTS OF THE CANONICAL CORRELATION ANALYSIS:
“left set” (resources, management, results) versus “right set” (impact)

	Variables, no.	Variance extracted, %	Total redundancy, %
Left set	12	43.2950	11.5744
Right set	4	100	25.9167

Canonical *R*: 0.63075; *Chi²* (48) = 144.53; $p = 1.42 \cdot 10^{-11}$
Conclusion: the association is distinctly significantly statistical.

Table 5

THE RESULTS OF THE CANONICAL CORRELATION ANALYSIS:
decomposition into canonical factors

F	R_{CC}	R^2	Eigen value	Variability, %	Wilks' Lambda	Approx. *F*	*df*	*p* - value
1	0.6307	0.3978	0.3978	49.51	0.3876	3.2802	48	$1.42 \cdot 10^{-11}$
2	0.4366	0.1906	0.1906	23.73	0.6437	2.1201	33	$4.07 \cdot 10^{-4}$
3	0.3771	0.1422	0.1422	17.70	0.7953	1.7956	20	$2.06 \cdot 10^{-2}$
4	0.2698	0.0728	0.0728	9.06	0.9272	1.3002	9	0.2413

R_{CC} is the canonical correlation coefficient; R^2 – the square canonical correlation coefficient be offered adequate guidance or performance monitoring in project management.

- According to the risk analysis applied to this type of institution, institutions that own a research department have been proven to be more efficient. Based on the results of the risk analysis, we suggest that the institutions that participate in financing competitions through projects must pay more attention to the dimension of project management in order to increase the performance of the institutions in obtaining national and/or European funds.

However, this study does have certain limits. The small dimension of the set of data, formed of 162 respondents, along with the number of institutions that filled out the questionnaire (69), caused fewer values and made the task of carrying out an applicable conceptual evaluation model with national performances more difficult. Nevertheless, it can be a longitudinal evaluation model; researching the model is extremely useful for project managers, program managers, strategies and policies when elaborating the RDI policies because it identifies and consolidates good practices for efficiency in project management.

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The restructuring of peasant households under the impact of urbanization by intensifying the knowledge transfer process and the collaboration with the textile industry

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REZUMAT – ABSTRACT

Restructurarea gospodăriilor țărănești, sub impactul urbanizării, prin intensificarea transferului de cunoștințe și colaborarea cu industria textilă

În condițiile intensificării procesului de urbanizare, gospodăria țărănească – ca formă de organizare predominantă a spațiului rural românesc – este amenințată cu falimentul economic și social, ca urmare a lipsei de competitivitate și a incapacității de a pătrunde, cu producția obținută, pe o piață din ce în ce mai dezvoltată și mai competitivă. Tehnicile și tehnologiile uzate atât din punct de vedere fizic, cât și moral, reticența față de nou, managementul defectuos al exploatațiilor sunt doar câteva dintre aspectele care ar putea fi soluționate prin intensificarea transferului de cunoștințe în mediul rural. În cadrul acestui studiu, prin analizarea situației actuale din spațiul rural românesc, s-a încercat să se demonstreze faptul că informațiile reprezintă un factor de producție de importanță crucială pentru dezvoltarea activității fermelor și, în final, pentru obținerea unei producții superioare din punct de vedere cantitativ și calitativ, care să reziste pe piață și să asigure fermierului român un nivel de trai comparativ cu cel din statele dezvoltate ale Uniunii Europene. Una din modalitățile de restructurare a gospodăriei țărănești o reprezintă orientarea activității către cultura plantelor tehnice destinate industriei textile, având în vedere compatibilitatea dintre caracteristicile acestui tip de exploatații și tehnologiile de cultură specifice acestor plante.

Cuvinte-cheie: urbanizare, transfer de cunoștințe, gospodărie țărănească

The restructuring of peasant households under the impact of urbanization by intensifying the knowledge transfer process and the collaboration with the textile industry

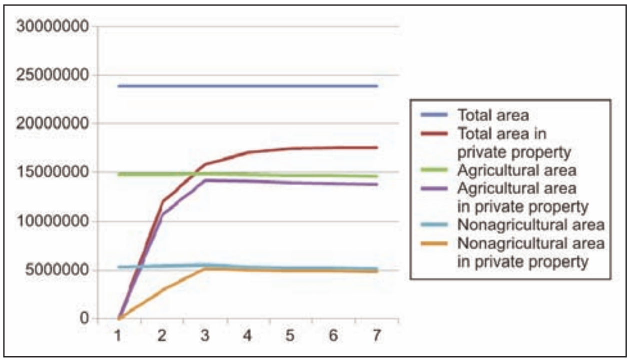
Under the conditions of the intensification of the urbanization process, the peasant household, as the dominant organizing structure from the Romanian rural area, is threatened with economic and social "bankruptcy", due to its lack of competitiveness and inability to penetrate with its production a market that is becoming more developed and competitive. Old and outdated techniques and technologies, poor management of holdings, are just some of the issues that can be solved by intensifying the knowledge transfer process in the rural areas. In this study, by performing an analysis of the current situation in the Romanian countryside, we tried to demonstrate that information is a crucial factor of production for the farm development, and for obtaining, in the end, a higher production in terms of quantity and quality that will withstand the market and will provide the Romanian farmer with living standards similar to those from the developed states of the European Union. We also suggest, as a means of restructuring the farms, to direct their activity towards the cultivation of technical plants for the textile industry, our suggestion being based on the compatibility between the characteristics of this type of farm and the technology for growing textile plants.

Key-words: urbanization, knowledge transfer, peasant household

Since the beginning of 1990s countries of Central and Eastern European are going through a continuous transformation process characterized by a series of administrative, political and economic reforms, the transition period causing a decline of economic performances (the decrease of real income, consumption and the increase of unemployment). Due to these considerations, the difference between the level of development of the Eastern and Western European countries became higher, the global crisis of the transformation process covering, more or less, all activity sectors of the national economies [1].

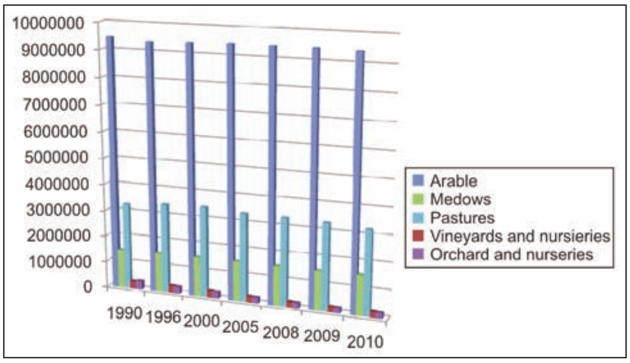
In agriculture, as a basic branch, there were significant lags and the gaps between Western states got bigger. In Romania, the main issues raised are related to land, the existing farmland, used or not, have an excessive fragmentation and they have a steady decline, “losing ground“ to urban areas as a result of an increased urbanization process.

Romanian rural areas are facing the urbanization problem, which has lately taken out of use a considerable part of the agricultural area, the impact of this process on the land bringing significant changes to it. That is why the relationship between land, urbanization and constructions became a topic of maximum interest in nowadays Romania, especially the aspects connected to the way in which the development was made and the multiple implications they produce [2]. The impact of the urbanization process was felt especially in the relationship between rural and urban areas resulting in strong differences between the two.



Source: www.insse.ro, consulted March 2012

Fig. 1. The dynamic of the Romanian land, ha



Source: www.insse.ro, consulted May 2012

Fig. 2. The dynamic of agricultural land during 1990–2010

The unbalance produced is reflected in the process of obtaining the daily necessary supplies for the consumption of the population that becomes even more difficult to be achieved. Looking at things from the perspective of system inputs, particular attention should be paid to the natural factor in which the land is a key element, which cannot be replaced and thus it implies continuous growth of its productive potential by practicing modern and sustainable agriculture. Together with the other elements of the production factors system, such as labor and capital, it provides existence and economic and social progress of the human society. To achieve this progress, most of the times, activities are performed in a completely wrong way, resulting in a chaotic development, imposed and induced at the same time by the new needs of today's society, characterized by an ever-expanding industrial system and forced degree of urbanization.

In Romania, the proportions of this phenomenon become alarming because of the industrial establishment that was done without taking into account the protection of agricultural land and thus without giving due consideration to the limited land for this purpose. Actually, the main factor of production, namely agricultural land becomes a clear target of the urban activity, either by transformation into urban land or by degradation by mechanical and forced chemicalized activity designed to provide the necessary food needed by the growing urban population [3].

The fact that contributes to the transformation described above is related to the evolution of the real estate market, which has seen a rapid development path in recent years, especially in the case of construction, in which old maize, wheat crops etc. were located.

In fact, balancing relations within the coalition building-land-urbanization should not be seen as an end of the development process, but as a rationalization, by establishing an optimal framework for the evolution of the three components so that each of them to better meet the objectives.

The main cause of the chaotic development of the urbanization is represented by the existing legislative framework in Romania, which has the role to regulate territorial organization – seen as a way of action applicable in all situations, regardless of the social or government system and formed on the basis of the development plans [10]. At the same time, standardization is taken into account and it involves finding a common way of action and clear implementation plans on which each state should guide. Standardization in this field means:

– compulsoriness of applying the Law no. 350/2001 [4] regarding land development and planning;
– common and unitary framework for the elaboration of land development documentation by defining its characteristic elements;
– compliance with the principles and directions set out in the European Union.

However, Romania has difficulties in having a land record and management caused by a number of key issues, such as: lack of ownership documents, lack of clear statistics of legal circulation of land or no real organization and planning systems of land, which led to new land problems quantified clearly by the data in table 1.

Study on the dynamics of land can be achieved through progressive analysis of its components, starting from agricultural areas compared to non-agricultural areas to individual analysis of the evolution of each of the component elements [5], [6].

For this purpose we transpose the data in table 1 to graphs (fig. 1) in order to show clearly the way in which the land recorded values in Romania have evolved.

Translated into numbers, the figures above show that in the total area of Romania, starting from 1990 until now there have been significant increases in private ownership of land, from 49.94% to 73.45%, mainly because of the legislation enacted after 1990. This phenomenon is also observed in the agricultural and non-agricultural land, indicating that in the case of agricultural land, a percentage of 94% is privately owned.

As regards land development in terms of destination, the agricultural area decreased slightly compared to 1990. The main cause of this phenomenon is due to land destination change through the process of urbanization [7].

Figure 2 illustrates the distribution of the agricultural area in the five categories of use: arable land, pastures, meadows, vineyards and vine nurseries, orchards and tree nurseries. According to the chart

Table 1

THE DYNAMIC OF LAND IN ROMANIA DURING 1990 – 2010								
The use of land	**Owner-ship**	**Years**						
		1990	**1996**	**2000**	**2005**	**2008**	**2009**	**2010**
Total	Total	23 839 071	23 839 071	23 839 071	23 839 071	23 839 071	23 839 071	23 839 071
	Private property	-	11 906 357	15 873 954	17 040 004	17 428 580	17 557 628	17 509 533
Agricultural	Total	14 769 028	14 788 730	14 856 845	14 741 214	14 702 279	14 684 963	14 635 520
	Private property	-	10 693 577	14 218 221	14 087 125	13 979 624	13 897 772	13 808 087
Arable	Total	9 450 395	9 338 951	9 381 109	9 420 205	9 415 135	9 422 529	9 405 024
	Private property	-	7 808 936	9 050 741	9 053 000	9 021 110	9 016 945	8 997 941
Pastures	Total	3 262 509	3 391 672	3 441 667	3 364 041	3 333 028	3 313 785	3 228 881
	Private property	-	1 077 300	3 197 705	3 132 780	3 054 618	2 981 023	2 919 988
Medows	Total	1 465 364	1 498 561	1 507 190	1 514 645	1 532 342	1 528 046	1 529 671
	Private property	-	1 404 758	1 469 061	1 482 326	1 500 887	1 498 263	1 498 164
Vineyards and nurseries	Total	277 371	288 971	272 252	224 082	214 463	215 382	213 431
	Private property	-	218 318	261 506	215 295	206 889	207 729	205 308
Orchards and nurseries	Total	313 389	270 575	254 627	218 241	207 311	205 221	198 583
	Private property	-	184 265	239 208	203 724	196 020	193 812	186 686

Source: www.insse.ro, consulted April 2012

below we can see the oscillations recorded over the period. By percentage, during the 1990–2010 the surface of vineyards and vine nurseries, orchards and fruit tree nurseries decreased by 24% and 37%, while arable land losses are of 0.5%. If you want to compare the values in this interval, we note that the percentage of 37% means a loss of 114 806 ha, among orchards and tree nurseries, 24% are 63 940 ha of vineyards and vine nurseries, while 0.5% means 45 371 ha of arable land. Analyzing the pastures and meadows, we see that both categories had an increasing trend of 0.8% and 4.5% in value, meaning an increase of 26 302 hectares of pastures and 64 307 hectares of meadows.

The restructuring of agriculture has led to new changes in the ownership and exploitation. The privatization of agricultural land in Romania led to the creation of two structural disadvantages, namely: large land area and small households, and too many farmers who are approaching or beyond the retirement age have large areas of land in small farms. Many of the emerging agricultural units are too small and the land is excessively parceled [15].

The fact that there are too many small holdings (farms) in parallel with very large ones provides a structural imbalance, which affects agriculture and its competitiveness. The situation is even worse if we take into account that the high number of small farms produce mainly for self-consumption, with low market sales of production and that they are not economically viable. Excessive land fragmentation is a serious impediment to the development of a modern and competitive agriculture [14].

A specific element of our agriculture is the peasant economy, which is the main form of organization in rural areas in Romania and the most common form. Peasant households are actually small farms which produce for their own consumption and occasionally sell a small part of their production. They seem a weak link agriculture, but in essence they maintain stability and transfer rural traditions and customs from one generation to another.

The great economist V. Madgearu (1940) claimed that the small peasant farm with an area of 3–4 hectares of agricultural land does not provide full employment of peasant family or completely necessary agricultural products for its own consumption by being in a subsistence economy, compared to large farms that produce both for their own consumption, pay in kind a part of the peasants' work, but first of all they produce mostly for the market. Although his works were initially focused on the industry, V. Madgearu [8] became one of the biggest supporters of the predominance of agriculture in the Romanian economy, based on the perpetuation of small farms.

The same approach is presented by Gabriel Popescu [12], who considers that due to the lack of means of production and monetary resources, peasant households become vulnerable on the food market as a direct and immediate effect of price scissors, leading to added value transfers with no equivalent from those entities to economic structures upstream or downstream of it.

Within these structures, self-consumption is dominant, and is intended for marketing only the surplus, and most of it is insignificant. Because of these particularities, small farms produce little income from their agricultural activity, which prevents them from purchasing industrial inputs delivering technical and technological progress. The existence of these farms shouldn't be approached negatively, they must be supported to enter the market trade flows, because these rural households create stability and provide environmental protection and their disappearance and strengthening being the result of a natural process and operation of the free market [13].

Most peasant households have only a social role, namely to provide a livelihood for the rural population and partly to an urban population and most of the production is oriented towards self-consumption [9].

The peasant household represents the primary dominant form of production and consumption in the developing countries, which provides the family, integrally or partially with the necessary food and has an important role in the economy [3].

Most farmers have chosen to work the land in their own households and because of this they have a very important social role as they provide the subsistence in the countryside and in a small part of the urban regions. Subsistence households rely mostly on traditional crops that require a small amount of inputs.

Programs of the Food and Agriculture Organization of the United Nations (FAO) and the international community have sought to improve the production system based on the participation of family farmers. Although small households have a stabilizing role on the environment and develop cooperative links upstream and downstream, their viability is limited and without guidance policies and integrated development support, farms will not be able to use efficiently natural and labour resources.

The program "African agriculture over the next 25 years" developed by FAO included numerous measures to develop agricultural production and environmental protection, and its objective is to support farms in four directions: stimulation of marketable surpluses, granting of loans to purchase inputs, organization of institutions for the promotion and development of agriculture, development of production infrastructure, distribution and marketing [16]. Similar programs were also applied in India, Indonesia, Thailand, Nepal and had a crucial role in the positive results of "the green revolution" [3]. "The Program for Development of Farming Systems" applied by FAO to support peasant households had the following main goals: obtaining sufficient quantities and reasonable prices for inputs required by the official services, the development of agricultural systems with the participation of family farmers, the support from rural organizations and groups of farmers to introduce new technologies etc.

In Central and Eastern European countries more than half of the agricultural areas are operated in small peasant households that produce for their own consumption. In Eastern European countries, peasant farms are an important source of survival for rural families. Poor technical equipment and the lack of operating capital emphasize the subsistence character of agriculture and limited market development. In these areas the development of alternative economic activities and practicing organic production systems are important ways of approaching the requirements of integration in the European Union structures. The setting up of complex economic farms that generate high incomes from small areas and the development of alternative economic activities are ways supported by EU programs, member countries of the Organization for Economic Cooperation and Development (OECD) and by some international financial institutions, International Monetary Fund (IMF) and World Bank [16].

The re-launch of the agricultural sector is not possible without restructuring the existing majority of holdings, respectively without the transition from peasant farms which produce almost entirely for self-consumption to commercial farms, which produce for the market. We suggest in this work, that the objective of these farms should be to grow crops for the textile industry because the specific characteristics of Romanian farms are adequate to the technological requirements of this type of cultures. Such a scenario would also contribute on one hand to the increase of revenue and hence to the living standards of farmers, with impact on the sustainable development of rural areas. On the other hand, the yields obtained would cover part of the demand for raw materials in the light industry [11].

Textiles, clothing and leather-footwear represented for Romania some of the most productive industries, with a major contribution to the country's gross domestic product. By its output, they cover most of the internal demand and contribute significantly to Romanian exports. In this regard, in 2007, the following values of the main macroeconomic indicators of the branch were recorded: 2.72% of GDP, 5.2% of industrial output, 18.42% of Romania's exports, 9.2% of Romania's imports and 23.35% of the total number of employees in the industry.

With the liberalization of world trade in textiles and as a result of developments in the euro exchange rate, the activity in the textile industry began to decline steadily. Thus, in 2009 the total number of companies that operated in Romania was approximately 8 100, of which 2 100 were producing footwear and 6 000 garments and textiles. The decrease was significant if we take into account that in 2006 the number was 9 500. A decrease was also recorded in terms of staff. Only during 2006–2009 the number went from 4 500 000 to 3 000 000, as a percentage, representing a decrease of over 30%. The export value had the same downward trend, dropping by about 335 million in 2007–2008.

The main problems of the sector are the lack of competitiveness due to the use of old and outdated products and technologies and the high cost of input supply. The sector recovery is not only recommended but necessary if we consider its current socio-economic role, that of employing a significant part of the workforce all over the country, especially women (due to dispersion of production units in all counties) and has still a high share of GDP and total exports of the country.

The existence of the local and regional infrastructure and the specialized personnel should facilitate the policies for the sector restructuration and recovery. There are however two main problems currently faced by entrepreneurs, such as the lack of competitiveness due to the use of old and outdated products and technologies and high cost of the input supply. To obtain a higher production in terms of quantity and quality, upgrading the production departments for most units in the field is mandatory. This process is mainly based on knowledge transfer, allowing not only access to the latest and modern techniques and technologies on the market, but also their proper use through the training of the staff involved. Also, many of the information strategies applied by the management personnel of the institutions are ineffective. The role of information in the development activities performed is not yet fully understood by most of the decision makers.

But even if the information and knowledge are intangible assets and often have a strong public character, in the case of industries, the high consumption of products and services makes their procurement involve high costs that exceed the available budget of the units.

A reduction of other costs, such as those with inputs could lead to the reallocation of certain amounts to the departments in charge with information and availability of new techniques and technologies. This is where the relationship between agriculture and the textile industry appears. Agriculture is one of the main suppliers of raw materials for the industrial sector, especially the textile, clothing and leather, both in crop production (flax, hemp, cotton) and animal production (leather, wool). Unfortunately, if in terms of livestock we could identify in the Romanian agricultural plan few industrial farms whose production can be considered raw material, technical plant crops for the textile industry are insignificant and cover only partly the necessary of the industry.

Given the particularities of technical plant production technologies a chance to solve the problem of supply of textile raw material required would be conversion of subsistence and semi-subsistence peasant households by targeting their activities to crops of hemp, cotton etc. But such an approach is only possible if the initiative comes from the industrial sector, the only one that currently has the means of information and knowledge necessary to guide peasant farms to obtain yields that reflect the desired quality and quantity standards.

A form of partnership by which the farmer has to be ensured that he has where to place the obtained production (under certain conditions) and the industrialist has to be sure he has the necessary raw materials is one of the solutions that we consider appropriate in this context and had good results in countries such as India or Pakistan.

It is undeniable that, by means of inward strategies, strengthening the cooperation between the two basic sectors of economy, industry and agriculture, could have an important contribution to economic recovery and sustainable development by using more national resources and fewer external resources.

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DOCUMENTARE

Noi tehnologii

TEHNOLOGIE DE AUTOCURĂȚARE A MATERIALELOR TEXTILE

Compania americană ***Luna Innovations Inc.***, din Roanoke, a încheiat un acord cu *UltraTech International Inc.*, din Jacksonville, S.U.A., prin care UltraTech primește licență exclusivă pentru comercializarea noii tehnologii *Luna*, privind folosirea agenților de îndepărtare a murdăriei.

Noii agenți de îndepărtare a murdăriei de pe materialele textile asigură protecție împotriva agenților patogeni, pentru personalul de prim-ajutor, și împotriva agenților contaminanți, pentru lucrătorii din domeniul industrial, menținând în același timp țesătura curată.

Procedeul permite aplicarea sub formă de produs independent pe materialele textile sau folosirea de pelicule tratate prin noua tehnologie integrată.

Pentru a asigura atât respingerea lichidelor pe bază de apă, cât și a celor pe bază de ulei, astfel încât acestea să nu poată pătrunde în materialul textil, noua tehnologie chimică, brevetată, folosește pelicule textile nanostructurate. Aceste pelicule, rezistente la fluide, conțin particule așezate ierarhic, formate din diverse materiale disponibile pe piață.

Peliculele cu autocurățare pot fi aplicate, pe scară largă, în procesele de finisare clasică a diferitelor materiale textile, sintetice și naturale.

Aplicarea noii tehnologii de tratament duce la optimizarea proprietăților de respirabilitate, autocurățare și durabilitate ale materialelor textile, precum și la reducerea greutății acestora.

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