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Determining the optimum spinning conditions to produce the rotor yarns from cotton wastes

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REZUMAT – ABSTRACT – INHALTSANGABE

Determinarea condiţiilor optime de filare pentru obţinerea firelor filate cu capăt liber din deşeuri de bumbac

În lucrare este abordată problema optimizării unor parametri ai sistemului de filare cu capăt liber, în vederea obţinerii de fire din bumbac de cea mai bună calitate, din deşeurile rezultate în urma procesului de egrenare. S-a studiat influenţa parametrilor rotorului – diametrul şi turaţia, a tipului duzei de debitare, a vitezei desfibratorului, a tipului de separator şi a densităţii liniare asupra calităţii firului, utilizând metoda Taguchi. De asemenea, au fost determinate condiţiile optime de obţinere a firelor filate cu capăt liber, din diferite cantităţi de deşeuri (65%, 50% şi 35%), rezultate din procesul de egrenare.

Cuvinte-cheie: deşeuri din bumbac, filare, calitatea firului, metoda Taguchi

Determining the optimum spinning conditions to produce the rotor yarns from cotton wastes

The paper addresses the optimization of various machine parameters in rotor spinning system to obtain the highest quality index of cotton yarn produced from the ginning process waste. The effect of rotor parameters – diameter and speed, navel type, opener speed, separator type and yarn linear density on the yarn quality were investigated, using the Taguchi method. Also, the optimum spinning conditions for the rotor yarns produced from different ginning waste proportions (65%, 50% and 35%) were determined.

Key-words: cotton wastes, rotor spinning, yarn quality, Taguchi method

Die Bestimmung der optimalen Spinnbedingungen bei der Fertigung der Rotor-Garne aus Baumwollabfällen

In der Arbeit wird das Problem der Optimierung einiger Parameter des Rotorspinnsystems angesprochen, für die Fertigung von Baumwollfaser bester Qualität, aus Abfällen resultiert aus dem Entkörnungsprozess. Es wurde die Wirkung auf der Garnqualität untersucht, was Rotorparameter – Durchmesser und Drehgeschwindigkeit, Typ der Abzugsdüse, Geschwindigkeit des Fadenöffners, Typ des Fadenklaubers und lineare Dichte anbelangt, indem die Taguchi-Methode angewendet wurde. Es wurden gleichsfalls die optimalen Bedingungen für die Fertigung der Rotorgarne bestimmt, die aus verschiedenen Abfallmengen (65%, 50%, 35%) des Entkörnungsprozess stammen.

Schlüsselwörter: Baumwollabfällen, Spinnen, Garnqualität, Taguchi-Methode

Environment protection and wastes recycling are the most important challenge for the future. For minimizing the production costs, the spinner has to improve exploitation of the raw material. The first solution is to provide a high-cleaning efficiency during the blowing and carding processes. The second solution lies in recovery of fibers from wastes [1].

In the 1970s, the emerging rotor technology was marketed on the premise of being able to spin lower-grade cotton into acceptable yarn quality at considerable savings. But in the 1980s, it was recognized that good-quality rotor yarns require good raw material allowing the technology to conquer upscale end uses in ever finer yarn counts [2]. Nowadays, rotor yarn can be spun from cotton waste by using high twist values. This is such a good way of recycling waste from the mill that no other spinning system exists enabling this economical application. As a rule, raw material should be selected in such a way as to build a balance between raw material cost and desired yarn characteristics. Many researchers [1, 2, 4, 5] have discussed the reuse of recovered fibers in spinning process. It is reported that waste fibers can be blended with primary raw material, with a percentage up to 20%, without noticeable changes in quality [1].

All those studies have discussed the effect of the fibers recovered from blowing and carding processes on the yarn quality. In this investigation, we study the effect of some rotor spinning parameters on the quality of the yarns produced from three different ratios of the fibers, which were gained from the ginning process. In order to estimate the optimum process conditions and to examine the individual effects of each of the controllable factors on a particular response, Taguchi's experimental design was used.

EXPERIMENTAL PART

The raw materials were cotton fibers and cotton waste collected from ginning machines. Cotton fibers were chosen for all mixtures with secondary raw material for three different proportions (50–50%, 35–65% and 65–35%). The cotton and waste fiber properties were measured by the Spinlab 900 according to ASTM (Standard test method for measurement of cotton fibers) by HVI (High volume instruments), ASTM D4605, with a total of four samples taken for evaluation. A statistical summary of raw fiber property measurements is included in table 1.

The blending was carried out after carding at the first passage in the drawing frame. Second passage was used to improve the homogeneity of the blending.

Table 1

FIBER PROPERTIES SUMMARY

Fiber properties	Waste cotton	Cotton
Fiber tenacity, cN/tex	23.2	27.1
Fiber elongation, %	6.6	6.7
Mean length by weight, mm	21.77	23.90
CV length by weight, %	1.94	1.91
Short fiber content by weight, %	11.1	7
Maturity index	0.81	0.82
Micronair	4.29	4.38
UQL (upper quartile length by weight), mm	27.19	28.89

Table 2

SLIVER PROPERTIES SUMMARY								
	Fiber content, %		1st drawing passage			2nd drawing passage		
Yarn code	Cotton	Waste	*U*, %	*CVm*, %	Linear density, ktex	*U*, %	*CVm*, %	Linear density, ktex
CW 63	65	35	7.91	9.92	2.90	8.61	10.82	3.35
CW 55	50	50	6.92	8.69	2.90	7.15	9.11	3.35
CW 36	35	65	7.09	8.88	2.90	7.41	9.36	3.35

Table 3

SETTING OF ROTOR SPINNING MACHINE	
Machine parameters	Description
Rotor diameter, mm	48, 54, 66
Rotor speed, rpm	41 000, 47 000, 53 000
Twist (T.P.M.)	775, 911, 1008
Opening roller type	OK40 (for cotton fibers)
Opening roller speed, rpm	9 800, 8 400, 7 350
Navel type	8 flutes, 4 flutes and without flutes (steel)
Separator angle	15°, 45°, 60°
Yarn linear density, Ne	12, 16, 20
Delivery speed, m/min.	52.4 m/min.
Yarn linear density, Ne	12, 16, 20

A statistical summary of sliver property measurements is included in table 2. Slivers were used to produce yarns with three different linear densities Ne 12, Ne 16 and Ne 20 on Elitex rotor-spinning machine. The setting of the machine is shown in table 3.

In order to estimate the optimum process conditions and to examine the individual effects of each of the controllable factors on a particular response, Taguchi's experimental design was used. This experimental design involves using orthogonal arrays to organize the parameters affecting the process and the levels at which they should be varied. The controllable factors, which were considered in this research, are rotor diameter and speed, opening roller speed, navel type, yarn linear density and separator angle. We chose the orthogonal array L_{27} shown in table 4, because it required only twenty-seven runs for combinations of six controllable factors varied at three levels.

In the rotor spinning machine, after the opening roller, the separated fibers running through the channel enter the separator and then the rotor collecting surface. The separator tooth is adjusted in three different angles (15°, 45°, 60°) as shown in figure 1. The shape of different navel types used in this investigation is shown in figure 2.

Table 4

EXPERIMENTAL DESIGN, L_{27}						
Run	Yarn count, P_1, Ne	Rotor speed, P_2, rpm	Opener speed, P_3, rpm	Rotor diameter, P_4, mm	Separator angle, P_5, degrees	Navel type, P_6
1	12	41 000	9 700	5	15	*A*
2	12	41 000	9 700	54	45	*B*
3	12	41 000	9 700	54	66	*C*
4	12	48 000	8 400	66	15	IA
5	12	48 000	8 400	66	45	*B*
6	12	48 000	8 400	66	66	*C*
7	12	53 000	7 350	48	15	*A*
8	12	53 000	7 350	48	45	*B*
9	12	53 000	7 350	48	66	*C*
10	16	41 000	8 400	48	15	*B*
11	16	41 000	8 400	48	45	*C*
12	16	41 00	8 400	48	66	*A*
13	16	48 000	7 350	54	15	*B*
14	16	48 000	7 350	54	45	*C*
15	16	48 000	7 350	54	66	*A*
16	16	53 000	9 700	66	15	*B*
17	16	53 000	9 700	66	45	*C*
18	16	53 000	9 700	66	66	*A*
19	20	41 000	7 350	66	15	*C*
20	20	41 000	7 350	66	45	*A*
21	20	41 000	7 350	66	66	*B*
22	20	48 000	9 700	48	15	*C*
23	20	48 000	9 700	48	45	*A*
24	20	48 000	9 700	48	66	*B*
25	20	53 000	8 400	54	15	*C*
26	20	53 000	8 400	54	45	*A*
27	20	53 000	8 400	54	66	*B*

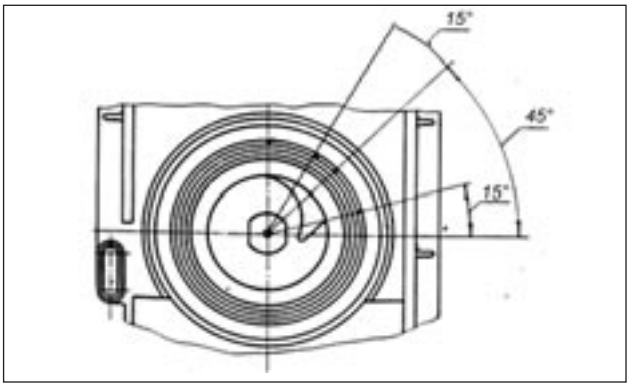


Fig. 1. Three setting angles for the separator

The yarns produced with the three rotor diameters, three different rotor speeds, opening roller speeds, navel types and separator angles were tested for tenacity, elongation and evenness. Samples were kept in standard testing condition for 24h prior to testing. Variations were to be expected within individual yarn bobbins, so the first few meters were discarded. In addition, samples were taken at various locations in the yarn bobbins. We used ASTM (Standard test method for tensile properties of yarns) by the Single-strand method, ASTM D2256, to determine single yam strength (cN/tex) and elongation at break (%) by the Instron. The testing speed was 60 mm/minute, pre-tension forces were 15 cN, and the gauge length was 500 mm. For each yarns sample, we took ten specimens to obtain an average value. We measured yarn unevenness with reference to ASTMD 1425, by the Uster Tester 3 at 100 m/minute, for 5 minutes.

The total yarn quality index (TQI) was the response in our study. This index was calculated using the following formula [6].

$$TQI = \frac{Tenacity\left(\frac{cN}{Tex}\right) \cdot Elongation\,(\%)}{CV\%} \qquad (1)$$

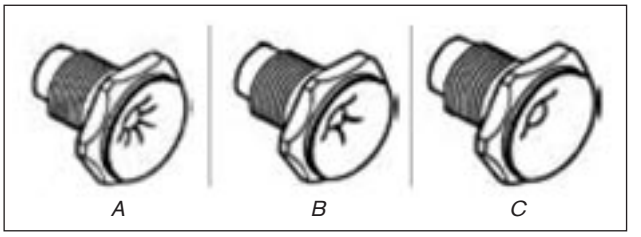


Fig. 2. Different navel types:
A – 8 flutes; *B* – 4 flutes; *C* – without flute

RESULTS AND DISCUSSIONS

A level average analysis was adopted to interpret the results. This analysis is based on combining the data associated with each level for each factor. The difference in the average results for the highest and lowest average response is the measure of the effect of that factor. The greatest value of this difference is related to the largest effects of that particular factor. The results of the different spun yarns (CW36, CW55 and CW63) produced on the rotor spinning system are given in tables 5, 6 and 7.

To facilitate the yarn quality analysis, we used the statistic delta defined as the difference between the high and the low effect of each factor. A classification can be done to determine the most influent factor. The study of yarn quality is a larger-the-better problem and optimum spinning conditions are determined by selecting the levels that show the highest average responses in tables 5, 6 and 7.

According to the level average analysis (table 5), for cotton-waste (35/65) spun yarns, rotor diameter has the largest effect on the yarn quality. Yarn linear density factor is second and it is followed by separator angle factor, opener speed factor, navel type factor, and rotor speed factor.

For cotton-waste (50/50) spun yarns, yarn linear density factor, shows the strongest effect. Rotor diameter factor is second and it is followed by rotor speed

Table 5

RESPONSE TABLE OF ROTOR SPUN YARNS FOR COTTON/WASTE (35/65)						
Factors	Average response value of each level					Optimum level
	I	II	III	Delta	Rank	
Linear density, P_1	9.662	10.069	8.214	1.854	2	II
Rotor speed, P_2	9.506	9.742	9.445	0.297	6	II
Opener speed, P_3	9.138	9.903	8.904	0.998	4	II
Rotor diameter, P_4	9.519	8.087	10.339	2.251	1	III
Separator angle, P_5	9.908	9.229	8.808	1.100	3	I
Navel type, P_6	9.658	9.213	9.074	0.584	5	I

Table 6

RESPONSE TABLE OF ROTOR SPUN YARNS FOR COTTON/WASTE (50/50)						
Factors	Average response value of each level, mean total quality index of yarn					Optimum level
	I	II	III	Delta	Rank	
Linear density, P_1	11.402	11.543	8.297835	3.245963	1	II
Rotor speed, P_2	10.681	11.069	10.29842	0.770922	3	II
Opener speed, P_3	10.685	10.619	9.938031	0.681912	4	I
Rotor diameter, P_4	10.740	9.1527	11.35061	2.197815	2	III
Separator angle, P_5	10.842	10.505	9.895983	0.609594	6	I
Navel type, P_6	10.743	9.9125	10.58807	0.675485	5	I

Table 7

RESPONSE TABLE OF ROTOR SPUN YARNS FOR COTTON/WASTE (65/35)						
Factors	Average response value of each level, mean total quality index of yarn					Optimum level
	I	II	III	Delta	Rank	
Linear density, P_1	11.667	12.093	9.179	2.913	1	II
Rotor speed, P_2	11.251	11.318	10.57	0.740	5	II
Opener speed, P_3	11.297	11.075	10.56	0.729	6	I
Rotor diameter, P_4	10.648	10.244	12.04	1.802	2	III
Separator angle, P_5	11.093	11.361	10.48	0.876	3	II
Navel type, P_6	11.453	10.882	10.60	0.849	4	I

Table 8

OPTIMUM LEVEL FOR CONTROL FACTORS OF ROTOR SPUN YARNS						
Spun yarns	Optimum level for each factor					
	Linear density	Rotor speed	Opener speed	Rotor type	Separator angle	Navel type
Cotton/waste (35/65)	11.402	11.543	8.297835	3.245963	1	II
Cotton/waste (50/50)	10.681	11.069	10.29842	0.770922	3	II
Cotton/waste (65/35)	10.685	10.619	9.938031	0.681912	4	I

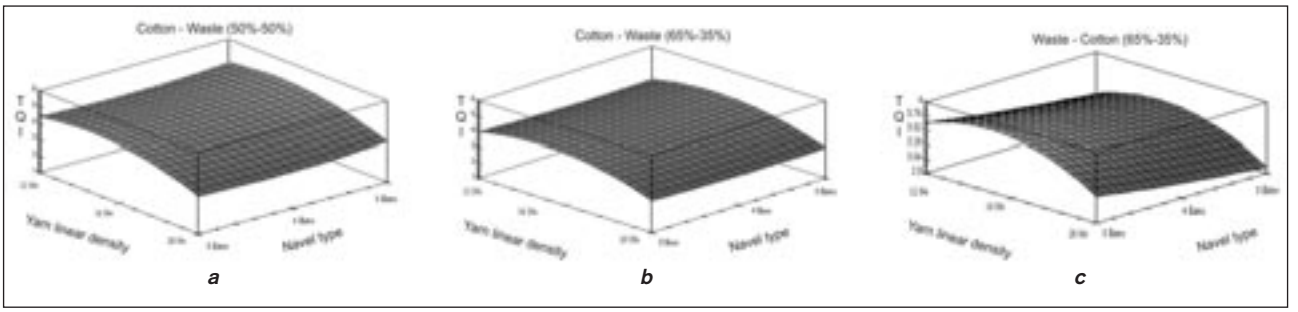
factor, opener speed factor, navel type factor and separator angle factor. For cotton-waste (65/35) spun yarns, the dominant parameters that influence the quality index are the yarn linear density, rotor diameter, navel type, separator angle, rotor speed and opener speed. As mentioned earlier, the selected study is a larger-the-better problem and optimum conditions are determined by choosing the levels that show the highest average responses in tables 5, 6 and 7.

Considering this principle, the recommended levels are summarized in table 8. Tables 5, 6 and 7 show that the increase in waste proportion degrades quality index. The empirical relationships between yarn quality and the six controllable factors were analyzed using DOE wisdom software.

The findings reveal that the yarns spun with linear density Ne 16 present the highest total quality index, compared with other yarn linear densities.

Figure 3 shows the effects of the navel type and yarn linear density on the total quality index of the yarns produced from different waste proportions. The findings show that, for all spun yarns produced from different waste proportions, application of the navels with 8 flutes gives the best yarn quality. To obtain a good spinning stability, the yarn must have sufficient twist at the peeling point, where it leaves the rotor groove. The rotation of the yarn around the inner wall of the navel creates an additional false twist on the yarn, between the rotor groove and the yarn draw-off tube. This action ensures a high twist moment, enabling the propagation of twist in the rotor groove and an increase in yarn tenacity. Increasing the number of the navel flutes causes to increase the friction between the yarn and the navel and, consequently, the false twist insertion. McCreight et al. [7] found that additional twist created inside the navel and increased in the yarn draw-off tube caused higher yarn evenness values. Therefore, the navels with 8 flutes result in higher tenacity and evenness, compared with other navels used in this investigation.

Figure 4 shows the effect of the rotor diameter and yarn linear density on the total quality index of the yarns produced from different waste proportions. The results reveal that the best yarn quality is achieved when the smallest rotor diameter is used. Generally, a smaller rotor diameter gives higher yarn strength [4, 8]. Similar observations were made by other researchers [9, 10]. The larger rotor diameter gives a smaller number of wrapper fibers, as expected. The yarn strength, in general, increased with the number of wrapper fibers. It was found that the number of belts per unit length increased with relatively smaller rotors. In addition, increasing the number of tight belts increases the elongation at break. The fiber individualization is also lower for the smaller rotor; thus, it is quite possible that the ring of fibers deposited in the groove of the smaller



a *b* *c*

Fig. 3. Effect of the navel type on the TQI index:
a – 50–50% cotton-waste; ***b*** – 65–35% cotton-waste; ***c*** – 35–65% cotton-waste

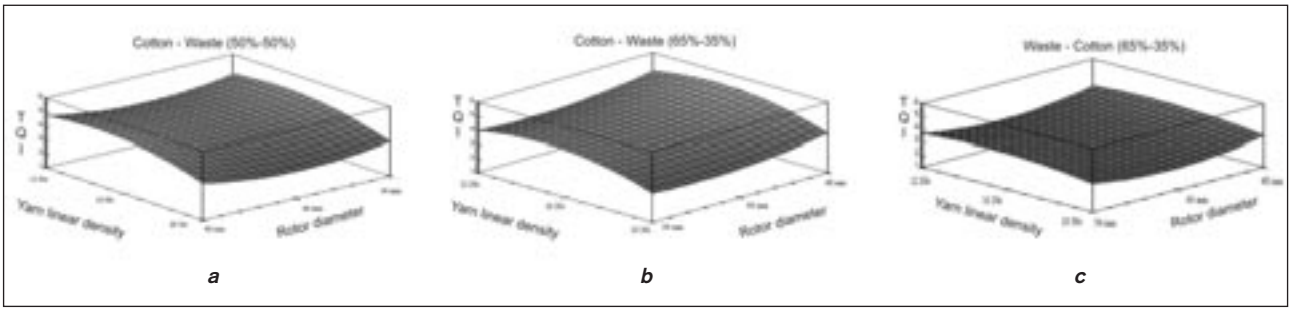


Fig. 4. Effect of the rotor diameter on the TQI index:
a – 50–50% cotton-waste; ***b*** – 65–35% cotton-waste; ***c*** – 35–65% cotton-waste

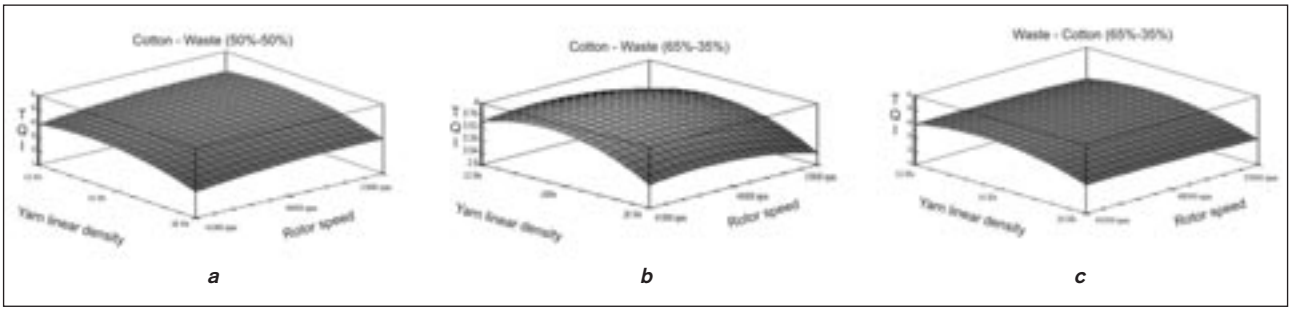


Fig. 5. Effect of the rotor speed on the TQI index:
a – 50–50% cotton-waste; ***b*** – 35–65% cotton-waste; ***c*** – 65–35% cotton-waste

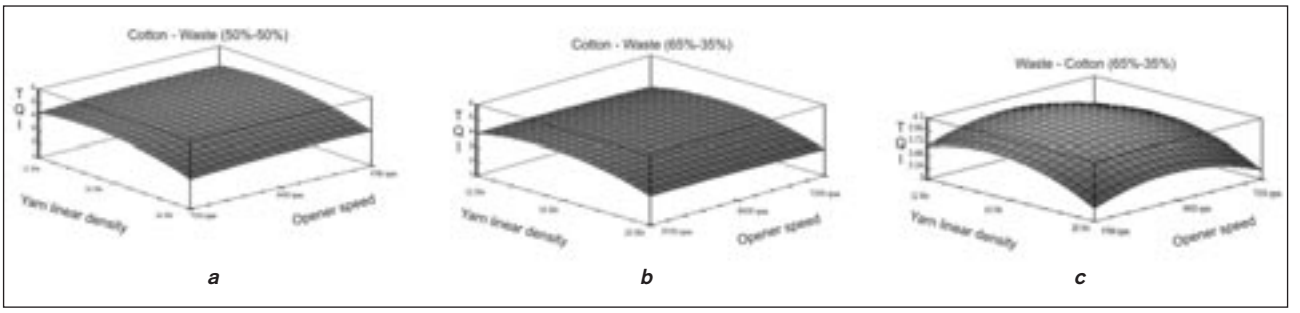


Fig. 6. Effect of the opener speed on the TQI index:
a – 50–50% cotton-waste; ***b*** – 65–35% cotton-waste; ***c*** – 35–65% cotton-waste

rotor experiences a greater false twist instead of real twist, thereby producing an irregular yarn. As a result, the total quality index of the yarns will increase.

Figure 5 shows the effect of rotor speed on the total quality index produced from different waste percentages.

For all spun yarns, the optimum rotor speed to produce the best yarn quality is achieved at 48 000 rpm. Kampen et al. [8] proposed that the deterioration of yarn tenacity at higher rotor speeds could be attributed to the increased false twist. Rotor speed has also a deteriorating effect on yarn tensile properties [11, 12]. Lawrence [13] reported that there is a slight decrease in strength with increasing speed, but a considerable decrease in the elongation at break. Yarn elongation at break, in general, decreases as the rotor speed is increased. It may be due to increase in the spinning tension, which causes a permanent strain in the yarn [13]. The irregularity of rotor yarns is dependent on the rotor speed [13]. Increased rotor speed can lead to increased yarn irregularity. Lower rotor speed gives relatively larger elongation for a certain rotor diameter. The yarn quality (yarn irregularity and imperfections) tends to deteriorate as the rotor speed increases. The deterioration of yarn regularity can be attributed to the formation of more wrapper fibers, as the rotor speed increases.

Figure 6 shows the effect of the opener speed on the total quality index produced from different waste percentages. The optimum opener speed must be determined according to raw material used in the rotor spinning. An increase in opener speed can affect negatively the yarn tenacity and elongations [14]. As the opening roller speed increases, the carrying factor (i.e. the effective number of wire points per unit time) increases, which in turn increases the opening efficiency of the opening roller. Due to the better opening of fibers, it can be expected that the fiber tufts of smaller size and uniform dimensions are fed into the transport tube and thus into the rotor groove. But at the same time, too high an opening roller speed results in higher rotor deposition, and fiber orientation inside the transport tube also deteriorates drastically, which causes higher end breakage [15]. The results show that the optimum opener speed for a yarn produced from higher waste percentage is 8 400 rpm and, for the yarns with lower waste percentage, the opener speed can increase to 9 700 rpm.

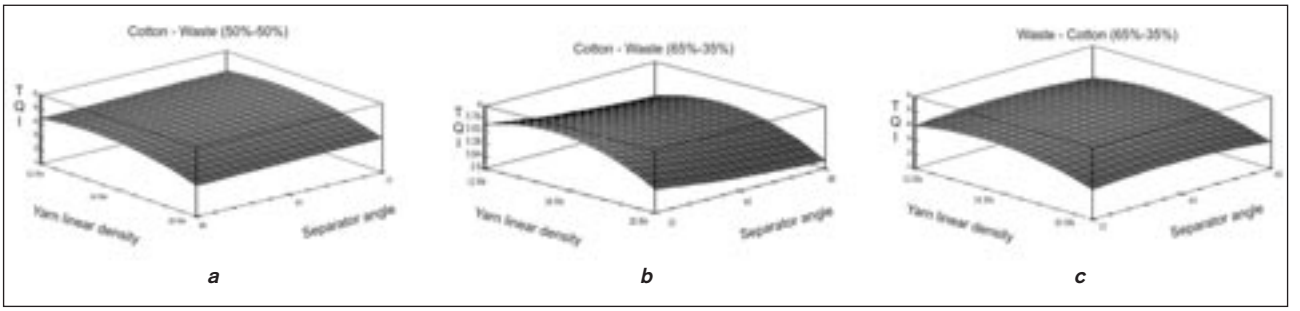


Fig. 7. Effect of the separator angle on the TQI index:
a – 50–50% cotton-waste; ***b*** – 65–35% cotton-waste; ***c*** – 35–65% cotton-waste

Figure 7 shows the effect of the separator angle on the total quality index produced from different waste percentages. The results show that the highest total quality index will be obtained, when the lowest separator angle (15^o) is used. For obtaining the highest quality of yarn produced from cotton/waste (65/35), the separator angle 45^o should be used. It is due to higher fiber length of this yarn. The angle of separator (fig. 1) has a great effect on the fiber tension. At the angle of 15^o, the tension of fiber during feeding process is the least. Therefore the wrapping fibers are formed more tightly and the yarn tenacity will increase. For the yarns which are produced from higher waste proportions, the higher quality will be obtained at lower separator angle. In the lower waste proportions, the higher angle can be used.

CONCLUSIONS

Various parameters of the rotor spinning system were optimized to produce the cotton yarn from the ginning waste. Using the Taguchi method, the effect of rotor parameters (diameter and speed), navel type, opener speed, separator type and yarn linear density on the yarn quality was investigated. According to the level average analysis, for cotton-waste (35/65) spun yarns, rotor diameter has the largest effect on the yarn quality. Yarn linear density factor is second and is followed by separator angle factor, opener speed factor, navel type factor and rotor speed factor.

For cotton-waste (50/50) spun yarns, yarn linear density factor shows the strongest effect. Rotor diameter factor is second and is followed by rotor speed factor, opener speed factor, navel type and separator angle factor. For cotton-waste (65/35) spun yarns, the dominant parameters that influence the quality index are the yarn linear density, rotor diameter, navel type, separator angle, rotor speed and opener speed. For the rotor yarns produced from different ginning waste proportions, the optimum spinning conditions were determined.

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Effects of pre-treatment processes on reactive dyeing of viscose fabrics

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REZUMAT – ABSTRACT – INHALTSANGABE

Influenţa proceselor de pretratare asupra vopsirii cu coloranţi reactivi a ţesăturilor din viscoză

Asemenea altor fibre regenerate, fibrele din viscoză sunt, în mod natural, fibre curate. Cu toate că procesele de pretratare a acestor fibre nu au un grad ridicat de complexitate, nu există un procedeu standard de vopsire a ţesăturilor din viscoză. În lucrare este analizată influenţa diferitelor procese de pretratare asupra modificărilor survenite atât la nivelul structurii macromoleculare şi al suprafeţei fibrei, cât şi asupra eficienţei culorii. După fiecare proces de pretratare, ţesăturile, având diverse structuri, au fost vopsite cu coloranţi reactivi de diferite nuanţe. În scopul analizării acestor efecte, s-au folosit metoda absorbţiei de iod şi, respectiv, metoda SEM. În plus, au fost utilizate mai multe teste post-hoc Duncan, pentru compararea influenţei proceselor de pretratare, a tipurilor de coloranţi şi de nuanţe şi a diverselor structuri ale materialului asupra morfologiei şi suprafeţei fibrei, dar şi asupra eficienţei culorii.

Cuvinte-cheie: viscoză, vopsire, coloranţi reactivi, caustificare, pretratare, metoda absorbtiei iodului, testare post-hoc Duncan

Effects of pre-treatment processes on reactive dyeing of viscose fabrics

Like other regenerated fibers, viscose fibers are naturally clean fibers. Although their pre-treatment processes are not complicated, there is no standard process of viscose fabrics. This paper examines the effects of different pre-treatment processes in terms of the changes in macromolecular structure, fiber surface and color efficiencies. Subsequent to each pre-treatment process, fabrics having different structures were dyed with different reactive dyestuffs in different shades. For analyzing these effects, the iodine sorption method and, respectively, SEM photographs, were used. Moreover, multiple Duncan post hoc comparison tests were used for comparing the influences of pre-treatment process, dyestuff and color shades and different fabric structures on the morphology and fiber surface, as well as on the color efficiency.

Key-words: viscose, dyeing, reactive dyestuff, caustic zing, pre-treatment, iodine sorption method, Duncan post hoc test

Die Einwirkung der Vorveredlungsprozesse auf das Reaktivfärben der Viskosegewebe

Ähnlich wie bei anderen regenerierten Faser sind die Viskosefaser, auf natürlicher Weise, reine Faser. Obwohl die Vorveredlungprozesse dieser Faser keinen hohen Komplexitätsgrad aufweisen, gibt es keinen Standardverfahren für die Färbung der Viskosegewebe. In der Arbeit werden die Einwirkungen der verschiedenen Vorbehandlungprozesse auf die Modifizierungen analysiert, welche sowohl auf Ebene der Makromolekularstruktur und auf Faseroberfläche, als auch auf der Farbeffizienz verursacht wurden. Nach jedem Vorbehandlungsprozess wurde das Gewebe von verschiedenen Strukturen mit Reaktivfarbstoffen verschiedener Nuancen gefärbt. Im Sinne der Analyse dieser Defekte, wurde die Iodabsoprtionsmethode und entsprechend die SEM-Methode angewendet. Es wurden mehrere post-hoc Duncan Tests für den Vergleich der Vorbehandlungsprozesse, der Farbmittelt- und Nuancentypen und der verschiedenen Materialstrukturen auf die Morphologie und Oberfläche der Faser, aber auch auf der Farbeffizienz, durchgeführt.

Schlüsselwörter: Viskose, Färbung, Reaktivfarbstoffe, Kaustifizierung, Vorbehandlung, Iodabsorptionsmethode, post-hoc Duncan Test

Viscose fibers are regenerated fibers, a sub group of man-made fibers in which, to guarantee the desired product properties, each stage of processing and spinning requires close attention. The viscose process is a demanding process that requires continuous, year-long operation to prevent gelling of the system and to yield high quality products [1]. The fibril structure of viscose fibers does not develop very well. Fibers from regenerated cellulose (cellulose II) have a semi-crystalline structure and, therefore, are composed of crystallites together with more or less disordered ("amorphous") regions. The fibers are of low crystallinity, but highly accessible to different media, due to their mainly amorphous molecular arrangement and an extensive inner surface. These morphological characteristics enable a stronger swelling effect when compared to the other regenerated cellulose fibers [2–4]. The spinning process involved in viscose type fibers produces a transitory structure caused by the collapse of the high-molecular chains. As a consequence, the treatment induces a relaxed structure in these collapsed cellulose chains at low alkali concentration, which facilitates re-orientation towards the crystalline structure of cellulose II [5].

The swelling of regenerated cellulose in alkali solutions has been applied industrially for many years. This process causes changes in the crystallinity, accessibility, unit cell structure, and orientation of fibrils in cellulosic fibers. The extent to which sodium hydroxide solutions change these properties depends on factors, such as the concentration of the sodium hydroxide solution, the temperature, the degree of polymerization, the source of the cellulose, the physical state of the cellulose (fiber, yarn or fabric), and the degree of tension employed to restrict or promote fiber shrinkage and swelling.

Besides the increase in the swelling tendency, with the aid of causticizing process, colour efficiency of the fabric significantly increases in the further printing and dyeing processes. Since dye intake of the viscose fabric increases and dyeing becomes better even after the treatment with 4–6% caustic soda, it is recommended to apply 6–8 Bé causticizing [6, 7]. Meanwhile, it was reported that causticizing process before reactive printings increased the colour efficiencies too [8].

On the other hand, today, it is more common to use bleaching or reductive washing instead of causticizing after desizing process for the achievement of a high and uniform absorptive of textile fibers for water, dyestuffs and finishing agents. Bleaching additionally provides a sufficiently high and uniform degree of whiteness, in order to ensure the purity of the bright dye shades. Hence, here, it was tried to analyze the different pre-

Table 1

THE FABRICS USED IN THE EXPERIMENTS			
Characteristics	***Fabric A***	***Fabric B***	***Fabric C***
Woven type	Plain	Plain	Fustian
Whiteness degree, brightness	69.07	61.86	67.99
Weight, g/m^2	98	131	191.5
Ends per cm	39	36	65
Picks per cm	24	26	26
Yarn count, Ne Warp Weft	 47 26	 48 45	 57 29

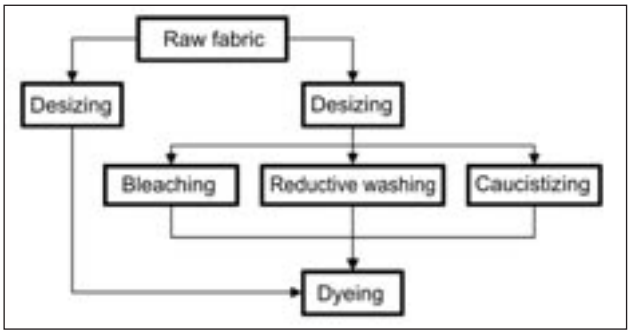

Fig. 1

Table 2

PROCESS CONDITIONS								
Desizing	**Causticizing**	**Bleaching**	**Reductive washing**	**Dyeing**				
				Cibacron H			**Cibacron F**	
2 g/l Na_2CO3 1 g/l Gemsol NS 60	100 Be NaOH	0.5 g/l Cottoclorin NF 1.0 g/l Stabilol – T 0.5 g/l Securan – FE	2.0 g/l Na_2CO_3 1.0 g/l Hydrosulphite	Dyestuff, %	Glauber's salt, g/l	Soda ash, g/l	Glauber's salt, g/l	Soda ash, g/l
		3.0 g/l Na_2CO_3		0.1	10	10	10	6
		2.0 g/l H_2O_2		1	20	15	20	8
70°C, 30 min. LR 1/20	35°C, 7 min. LR 1/20	90°C, 60 min LR 1/20 0.3 g/l CH_3COOH	70°C, 10 min LR 1/20	3	60	20	40	14
		0.3 g/l Biolase APT 55°C, 15 min						

treatment processes of viscose fabrics in terms of color efficiencies and fiber morphology.

EXPERIMENTAL PART

This study was realized in two steps. In the first step of the study, the effect of pre-treatment processes on the colour efficiencies (*K/S*) of fabrics were investigated in different fabric structures, different reactive dyestuffs and colour shades. The evaluation was made statistically by using the values, obtained from *K/S* change of desized fabrics after different pre-treatment processes *(K/S_{change}):*

$K/S_{change} = [(K/S_p) – (K/S_d)]*100/(K/S_d)$;

(K/S_d) = K/S of desized fabric;

(K/S_p) = K/S of pre-treated fabric after desizing (reductive washed, bleached or causticized).

Afterwards, the most effective pre-treatment process was examined in terms of the change in the macromolecular structure of viscose fabrics with the help of iodine adsorption method. Iodine adsorption was determined by calculating the iodine adsorption value ISV (mg absorbed iodine/g cellulose) according to Schwertassek [9, 10].

Iodine adsorption method

0.3 g viscose fibers were added to 1.2 ml KI_3 solution (5 g I_2, 40 g KI, 50 ml H_2O) · 100 ml Na_2SO_4 (200 g/l) was added after 3 minute of stirring and then stirred for one hour and filtered. The iodine concentration of the sample and the reference was determined by titration using Na thiosulfate ($Na_2S_2O_3$, 0.02 N).

The ISV is the amount of iodine adsorbed by one gram cellulose substrate. It is calculated in the following way:

$$ISV = \frac{(a - b \cdot 1.33) \cdot F \cdot 2.538}{M_a}$$

where:

- *a* is volume of $Na_2S_2O_3$ solution (c = 0.01 mol/l) for a liquor of blank KI_3 solution, ml;
- *b* – volume of $Na_2S_2O_3$ solution (c = 0.01 mol/l) for aliquot of sample solution, ml;
- *c* – constant (for cellulose: 1.33);
- *F* – aliquot factor of $Na_2S_2O_3$ solution, determined by $KMnO_4$ (0.02 mol/l);
- m_a – weight of absolutely dry fiber sample, g.

For these studies, three different 100% viscose woven fabrics (table 1) and four different dyestuffs were used in accordance with the test plan (fig. 1). All pretreatment processes were realized on an overflow machine with a capacity of 5 kg (Dogus trade mark) and a tensionless dryer (Santex).

Afterwards, dyestuffs were applied to the viscose fabrics with Cibacron Red FB (CI: Rea. Red184), Cibacron Red HF (CI: Rea. Red 273), Cibacron B. Blue FN G (CI: Rea. Blue 204) and Cibacron Blue H GN (CI: Rea. Blue 266). Fabrics were dyed in Termal trade mark laboratory type exhausting machine, according to the dyeing method, as mentioned in figure 2. Washing-off was performed with the procedure suggested for Cibacron H and F dyestuffs, using a home-type washing machine. During the treatment processes, "soft mill water" (permutit-water) was used (table 2).

K/S values of dyed fabrics were determined with a Minolta CM 3600d Model spectral photometer. Surface of fibers was evaluated by taking fiber photographs with Gemini trade mark scanning electron microscope.

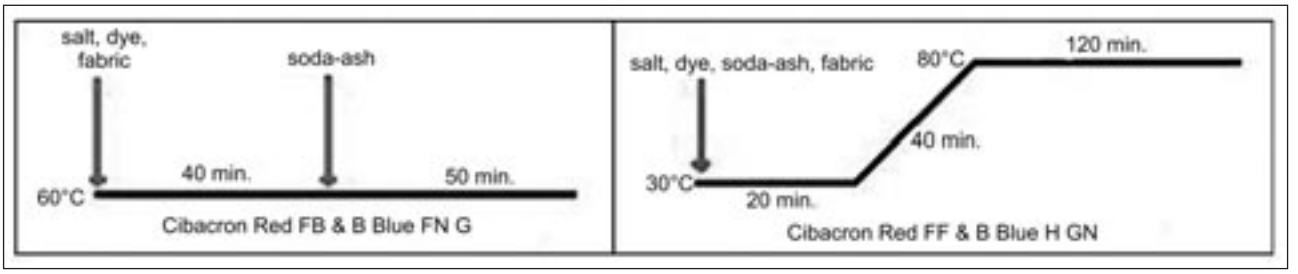

Fig. 2

Table 3

ANOVA TESTS OF BETWEEN-SUBJECTS EFFECTS					
Source	**Type III sum of squares**	**Idf**	**Mean square**	***F***	**Sig.**
Corrected model	37 903.383	37	1 024.416	14.872	0.000
Intercept	10 334.442	1	10 334.442	150.027	0.000
Pre-treatment	28 032.486	2	14 016.243	203.477	0.000
Type of dyestuffs	1 077.081	3	359.027	5.212	0.003
Color shade	736.430	2	368.215	5.345	0.007
Fabric structure	1 269.740	2	634.870	9.217	0.000
Pre-treatment* type of dyestuffs	2 555.197	6	425.866	6.182	0.000
Pre-treatment* color shade	2 941.073	4	735.268	10.674	0.000
Pre-treatment* type of dyestuffs* color shade	600.424	12	50.035	0.726	0.721
Type of dyestuffs* color shade	690.951	6	115.158	1.672	0.141
Error	4 821.866	70	68.884	–	–
Total	53 059.691	108	–	–	–
Corrected total	42 725.249	107	–	–	–

* Dependent variable: MEASURE, *K/S*, %
IR *squared = 0.887 (Adjusted* R *squared = 0.827)*

The results (expressed as mean values/standard deviation) of all assays were compared using ANOVA, followed by a post hoc test (Duncan's test).

RESULTS AND DISCUSSIONS

In order to investigate the effect of such parameters as fabric structure, type of reactive dyestuffs, color shade and pre-treatment processes on colour efficiencies, after reactive dyeing, a statistical research was carried out. ANOVA for K/S_{change} (*K/S* change of desized fabrics after different pre-treatment processes) among 108 samples indicated that there was a significant impact of pre-treatment processes, type of dyestuffs, dye concentration and the fabric type on the colour efficiency change. Moreover, the interaction between pre-treatment – type of dyestuffs, pre-treatment – colour shade, pre-treatment – type of dyestuffs – colour shade and type of dyestuffs – colour shade has also been examined, but only the first and second interaction was significant (table 3).

Table 4

DUNCAN POST HOC TEST OF THE DYESTUFF TYPE			
Dyestuff type	***N***	**Subset 1**	**Subset 2**
Cibacron Blue H GN	27	4.9564	–
Cibacron Red H F	27	8.9858	8.9858
Cibacron Blue FN G	27	–	12.3037
Cibacron Red F B	27	–	12.8825
Sig.	–	0.079	0.107

Obs. Means for groups in homogeneous subsets are displayed.
Based on type III sum of squares the error term is mean square (error) = 68.884

When the parameters were examined one by one, it was seen that the type of dyestuff, fabric structure and the colour shade indicated significant changes in K/S_{change} values. On the other hand, among these parameters, the pre-treatment processes have the most significant effect on the K/S_{change} values, because the *F* value obtained from the ANOVA for pre-treatment processes is the highest value, compared with the other parameters. For the evaluation of above mentioned parameters' impact, the Duncan's post hoc test was carried out.

Type of dyestuff

As seen from table 2, the dyestuffs can be assembled into two groups; while Cibacron Blue H GN and Cibacron Red H F were a group, Cibacron Blue FN G and Cibacron Red F B formed the other group. That is to say, the dyestuffs with low reactivity "Cibacron Blue H GN and Cibacron Red H F" and dyestuffs with high reactivity "Cibacron Blue FN G and Cibacron Red F B" act on K/S_{change} values differently (table 4). On the other hand, it was seen that the dyestuffs which have high reactivity, changed the K/S_{change} values more efficiently than the other dyestuff group. During the dyeing with low reactive dyestuffs, the medium temperature must be high (as recommended in CIBA catalog). As a result of this, the heat increasing decreases the effect of the pretreatment processes onto the colour efficiency changes, because of high temperatures swelling of the fabrics, finally the dye take up increases. It is also obvious that the Cibacron Blue H GN dyestuff is the

Table 5

DUNCAN POST HOC TEST OF THE COLOR SHADE			
%	N	Subset 1	Subset 2
3	36	6.1402	–
1	36	–	11.0732
0.1	36	–	12.1320
Sig.	–	1.00	0.590

Obs. Means for groups in homogeneous subsets are displayed. Based on type III sum of squares the error term is mean square (error) = 68.884

Table 6

DUNCAN POST HOC TEST OF THE FABRICS TYPE			
Fabrics type	N	Subset 1	Subset 2
Fabric *B*	36	5.1388	–
Fabric *A*	36	–	10.8932
Fabric *C*	36	–	13.3142
Sig.	–	1.000	0.220

Obs. Means for groups in homogeneous subsets are displayed. Based on type III sum of squares the error term is mean square (error) = 68.884

Table 7

DUNCAN POST HOC TEST OF THE PRE-TREATMENT PROCESSES			
Pre-treatment processes	N	Subset 1	Subset 2
Bleaching	36	–2.2844	–
Reductive washing	36	–0.9221	–
Causticizing	36	–	32.5527
Sig.	–	0.448	1.000

Obs. Means for groups in homogeneous subsets are displayed. Based on type III sum of squares the error term is mean square (error) = 68.884

least effective dyestuff among the dyes examined in terms of the impacts on the K/S values.

Colour shade

The colour shade is also the other important parameter that affects the change in colour efficiencies as from the ANOVA. Moreover, in table 5 the Duncan post hoc test was carried out for the colour shade parameter in order to evaluate the effect of different impacts of different shades onto the colour efficiency changes. It was determined that the changes occurred in colour efficiencies because of the other factors (pretreatment processes, dyestuffs and fabrics) are minimum when the colour shade is 3%. In this colour shade, there is enough dyestuff in the bath and enough time for the desized fabric. So, the effect of the pre-treatment process onto the colour efficiency changes cannot be seen clearly as the other colour shades. The *K/S* increases with the decrease in the shade. In conclusion, in light shades, the change in K/S is getting higher.

Type of fabric

The other interesting result obtained from the ANOVA shown in table 6 is the effect of fabric structure on the changes in *K/S* values, after carrying out different pre-treatment processes. It was seen that the *K/S* changes were nearly similar in the fabric *A* and *C*. Yet, this change is significantly lower when the fabric *B* is used. Although they are all 100% viscose based fabric, their different effect onto the changes in *K/S* values can be explained with their structure. Fabric *B* has a special feature that its warp yarns have a different twist direction; because of this, its surface appearance gets rough and this affects the colour measurement and reduces the differences caused by the use of different dyestuff and shade and application of different pretreatment processes. So, their impact on the change of color efficiencies were not the same, although fabric *A* and *B* are both plain woven fabrics.

Pre-treatment processes

As already mentioned, among the parameters, the most important parameter which affects the colour efficiencies after reactive dyeing was the pre-treatment process [11]. The impact of the pre-treatment was not changed in the different fabrics, dyestuffs and colour shades. In all cases, the pre-treatment process done before dyeing caused the important reason of change in colour efficiency of desized fabric. But the real issue is which one of these pre-treatment processes has the greatest importance in terms of the change occurred in *K/S* values. Finally, table 7 showed that, among these pretreatment processes, causticizing caused the highest change in the *K/S* of desized fabrics. While causticizing carried after desizing was improving the *K/S* nearly 32%, while the mean of the K/S_{change} of samples dyed after causticizing with four different colour, 3 different shades and 3 different fabric, bleaching and reductive washing decreased the *K/S* slightly.

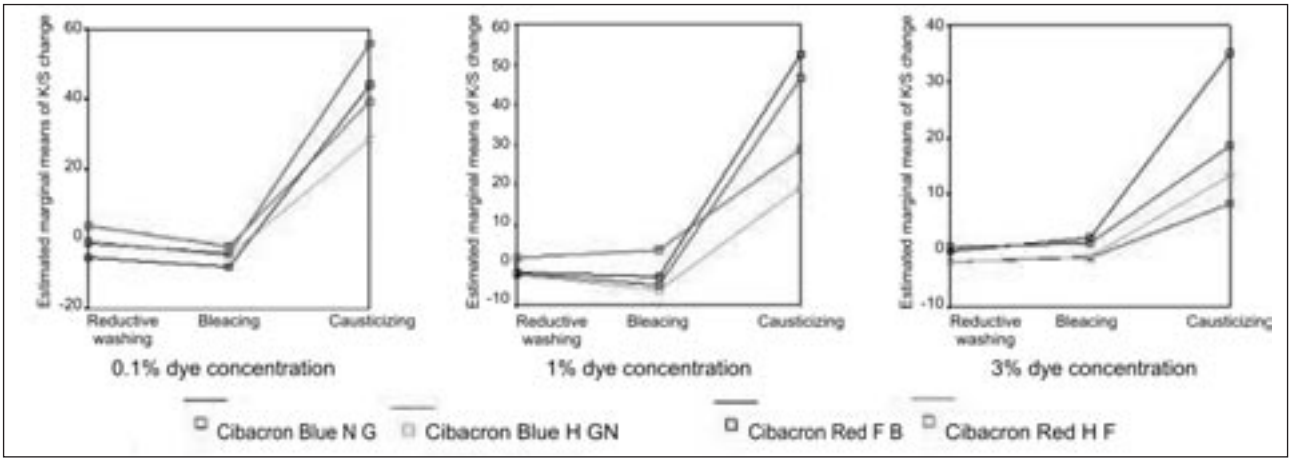

Fig. 3

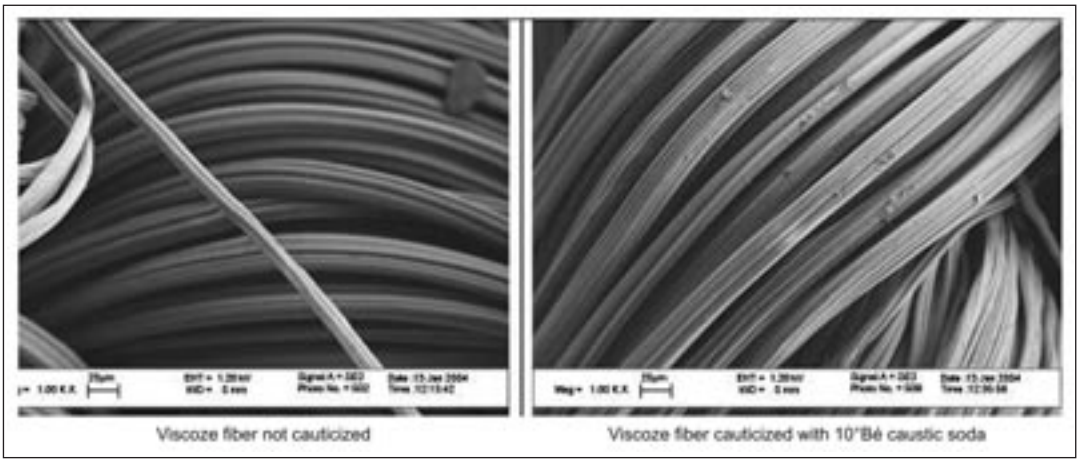

Fig. 4

Table 8

FIBRE ACCESSIBILITY (ISV – IODINE SORPTION VALUE)			
ISV, mg $I^2 \cdot g^{-1}$ sample			
Desized	**Reductive washed**	**Bleached**	**Causticized**
122	122	122	96.63

The results obtained from the Duncan post hoc test of pre-treatment processes confirmed once more the values exhibited for all colour shades in figure 3. In this plots, three different fabrics, accepted as three repeats of experiments. Hence, were figure 3 shows the mean of the K/S_{change} values of three different fabrics. But causticizing gave the highest change, even in the case of using different fabrics, in which fabric B gave the least change in *K/S* values. As a summary it can be readily said that causticizing is the most effective process among the pre-treatment processes, even in the use of different dyestuffs and in different colour shades. In other words, managing a causticizing process before dyeing fairly increased the K/S values of samples, unlike the other pretreatment processes. Because of this high increase in *K/S* values with causticizing, there were some advantages ensured with carrying out causticizing in pretreatment processes. Because it is possible to obtain higher colour efficiencies with causticizing, some ecologically and economically important benefits [11] can be achieved; one is decreasing the dyestuff consumption – it was seen that the use of 30% less Cibacron Blue FNG dyestuff or without the use of salt for causticized viscose is enough to obtain the same colour shade of not causticized fabric dyed with 2% dyestuff.

Iodine adsorption of pre-treated fabrics

A good correlation between iodine adsorption and structural changes is observed, i.e. the increase in crystallinity decreases the accessibility of fibers; thereby, the sorption of iodine becomes lower [3]. So, to identify the morphology of viscose fibers after different pre-treatment processes, iodine adsorption method was used. The results of iodine adsorption test are shown in table 8.

Results show that the desized, reductive washed and bleached viscose fibers have the same ISV and all of these have a significantly higher ISV and hence a lower crystallinity than the causticized viscose fibers. After causticizing, the ISV of the desized viscose fibers increases, while, in the case of reductive washed and bleached, ISV does not change. The causticized viscose fiber has thus a less open and more crystalline structure with less interfibrillar surfaces. As a summary, the values obtained from the iodine adsorption method clearly show that, among the pre-treatment processes only causticizing can affect the macromolecular structure of viscose fibers, that is to say, during causticizing the crystalline structure of the viscose fibers has increased significantly.

SEM photographs of causticized and not causticized fabrics

In order to investigate the change in the fiber surface and fiber cross section, SEM photographs of fibers were taken. It was observed that after causticizing, the surface of viscose fibers become smoother and swell, as shown in figure 4. This is why the causticized fabrics gave higher color efficiencies and dye uptake after dyeing than the one not causticized, treated with other pretreatment processes.

CONCLUSIONS

In this study, we examined statistically the parameters effect on colour efficiencies of viscose fabrics after reactive dyeing with ANOVA, followed by a post hoc test (Duncan's test). As a result of statistical analysis, it was observed that type of dyestuff, colour shade, type of fabric and the pre-treatment have significance over the colour efficiencies. But, among them, especially the pretreatment was the most important factor as seen from the ANOVA. Moreover, post hoc test (Duncan) were applied for all these parameters and, actually, it was found from the post hoc test that, unlike the other pre-treatment processes, only causticizing changed the colour efficiencies considerably. With causticizing, the colour efficiencies after reactive dyeing increases too much, so that it can be possible to dye the viscose fabrics with less dyestuff or salt [10]. Further investigations should be done on this issue.

In addition, because fibers swell after causticizing, they get smoother [10]. Meanwhile, some changes occurred during the causticizing process. When the iodine adsorption method was carried out for pre-treated

viscose fabrics, the values showed that the change of crystallinity index strongly depends on the type of pre-treatment process. While a great increase of crystallinity index is observed by causticizing of viscose fibres, reductive washing and bleaching did not change the crystallinity index.

Regenerated cellulose fibres are more susceptible to the treatment processes than most other fibers, this being particularly so with viscose fibers, owing to the lower crystallinity index. Because causticizing increases the crystallinity index, this disadvantage of viscose fibers can be reduced.

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DOCUMENTARE

Materii prime

FIBRE CU PROPRIETĂȚI ANTIMURDĂRIRE

În cadrul unui proiect elaborat de ***Institutul Hohenstein***, din Bönnigheim/Germania, în colaborare cu ***ITCF Denkendorf*** și finanțat de Ministerul Federal al Economiei și Tehnologiei, au fost dezvoltate textile cu proprietăți de antimurdărire. Datorită structurii de suprafață modificate a fibrelor se poate obține o optimizare a așa-numitului „efect lotus".

Potrivit afirmațiilor cercetătorilor de la Hohenstein, particulele care au fost încorporate pe parcursul prelucrării au avut efecte neglijabile asupra comportamentului coloranților și asupra absorbției acestora în polimer.

Până în prezent, finisarea convențională era utilizată în vederea aplicării pe suprafața fibrelor a unor micro și nanostructuri hidrofobe. Stratul funcțional realizat în acest fel conferea efecte bune de respingere a murdăriei, însă aceste efecte nu erau întotdeauna durabile, în condițiile unei utilizări intense.

În prezent, noua abordare privind producerea de structuri de suprafață a redus semnificativ acest deficit și a optimizat efectul de antimurdărire. Cu ajutorul na-noparticulelor superparamagnetice, încorporate direct în procesul de filare din topitură a fibrelor artificiale, s-a generat o suprafață nanostructurată suplimentară. Această așa-numită structurare feromagnetică a fibrelor, într-o bobină cu câmp magnetic cu energie mare, are loc direct după duza de filare, atunci când topitura de filare se află încă în stare termoplastică, ceea ce permite, ulterior, etirarea normală a filamentului. Eșantioanele de fir și tricot au fost realizate din monofilamente, la scară de laborator, în scopul caracterizării proprietăților de suprafață nou dezvoltate.

Aceste eșantioane au fost apoi evaluate atât din perspectiva caracteristicilor lor hidrofobe și de antimurdărire, cât și pentru durabilitatea pe parcursul utilizării. Pentru producerea la nivel industrial a acestor fibre structurate feromagnetic, reprezentanții Hohenstein au afirmat că este necesară continuarea cercetărilor în parteneriat cu un producător de fibre, pentru optimizarea procesului de filare.

Sursa: www.hohenstein.de

General aspects concerning the development of a female dimensional typology using 3D body scanning measurements

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REZUMAT – ABSTRACT – INHALTSANGABE

Aspecte generale privind elaborarea tipologiei dimensionale pentru femei, pe baza măsurătorior 3D

Fabricarea confecţiilor în sistem industrial nu se poate realiza fără o tipologie dimensională, care să reflecte toate variantele morfologice ale populaţiei. În Uniunea Europeană s-au desfăşurat, recent, studii antropometrice în scopul revizuirii sistemelor de mărimi pentru confecţii. Ţara noastră s-a aliniat acestei cerinţe. În perioada 2007–2009, s-a desfăşurat o amplă cercetare antropometrică asupra populaţiei – femei şi bărbaţi, folosind metoda de scanare 3D. Lucrarea prezintă rezultatele parţiale, după prelucrarea prin metode statistico-matematice a datelor primare de la scanerul 3D, ale anchetei antropometrice desfăşurate asupra populaţiei de sex feminin, din România. Au fost analizate dimensiunile principale ale corpurilor femeilor – înălţimea corpului, perimetrul bustului şi perimetrul şoldurilor, care sunt utilizate în mod curent la caracterizarea formei corpului şi la structurarea standardelor antropometrice pentru adulţi.

Cuvinte-cheie: scanare 3D, date preliminare, tipologie dimensională, prelucrare statistică

General aspects concerning the development of a female dimensional typology using 3D body scanning measurements

Making clothing in the industrial system is not possible without a dimensional typology that reflects all morphological features of the populations. In the European Union, several countries have conducted anthropometric studies aimed to update sizes tables. Our country is aligning to these requirements. During 2007–2009, an extensive anthropometric research was carried out on adult population, women and men, by the 3D scanning method. This paper presents the partial results of the aforementioned survey conducted on Romanian women population, after processing all raw data from the 3D body scanner, using statistical-mathematic methods. Under analysis were the main dimensions of the female body – body height, bust perimeter and hip perimeter – which are currently used to characterize the body shape and to structure the anthropometric standards of the adult wearer.

Key-words: 3D scanning, raw data, dimensional indicators, statistical processing

Generelle Aspekte der Erarbeitung dimensioneller Typologien für Frauen, aufgrund 3D-Messungen

Die Bekleidungsherstellung im Industriesystem kann ohne eine dimensionelle Typologie, weche alle morphologischen Varianten der Bevölkerung in Betracht zieht, nicht durchgeführt werden. In der Europäischen Union wurden vor kurzem anthropometrische Untersuchungen für die Aktualisierung der Masssysteme für Bekleidung unternommen. Unser Land hat sich dieser Richtlinie auch angepasst. In der Zeitspanne 2007–2009 wurde eine umfangreiche anthropometrische Untersuchung der Bevölkerung durchgeführt – Frauen und Männer, indem die 3D Ultraschallaufnahme angewendet wurde. Die Arbeit stellt vor Teilergebnisse der anthropometrischen Untersuchung für die Bevölkerung weiblichen Geschlechts aus Rumänien, nach der Bearbeitung durch statistisch-mathematische Methoden der Primärdaten des 3D Scanners. Es wurden die Hauptdimensionen des Körpers einer Frau analysiert – Höhe des Körpers, Büsten- und Hüftenumfang, welche generell bei der Charakterisierung der Körperform und der anthropometrischen Standardstrukturierung für Erwachsene angewendet werden.

Schlüsselwörter: 3D Scanner, Primärdaten, dimensionelle Indikatoren, statistische Bearbeitung

In the industrial manufacturing system, it is not possible to make clothing items in unlimited sizes, according to each potential wearer, but only in a limited size range.

The number of variants (standardized sizes) must be optimal, in order to ensure a maximum dimensional correspondence between the industrially manufactured clothes and the morphological features of prospective wearers.

Dimensional correspondence human body vs. ready-made clothes is solved by a rational and scientifically based anthropometric standard (SA).

From an objective point of view, the anthropometric standard specifies a set of body types (standard sizes, dimensional variants), which must reflect the variety of morphological types found in a population.

An anthropometric standard is reasonable, when a limited number of body types provides the population the highest degree of satisfaction related to the clothes manufactured in the industrial system.

Recently, in different countries throughout the world, an extensive review of the anthropometric systems for clothing sizes has been carried out.

This research area includes a comprehensive research topic managed by The National Research and Development Institute for Textiles and Leather – Bucharest (I.N.C.D.T.P.) and carried out in partnership with "Gheorghe Asachi" Technical University – Iaşi, the academic staff from the knitwear-clothing department, the Romanian Standardization Association (A.S.R.O.) – Bucharest, and "Lucian Blaga" University – Sibiu.

The research theme *"Developing a national clothing market by 3D scanning method to identify specific anthropometric characteristics of Romanian population"* was addressed along the period lapsed from 2007 to 2009 and aimed to develop core systems of sizes for adult men and women from Romania.

This paper presents some partial results of the research theme addressed, related to the statistical processing of female raw data from the anthropometric survey conducted in Romania. At this stage, it is important to analyze the tendency of key dimensions values: body height – I_c, the bust perimeter – P_b and hips perimeter – $P_ş$, necessary to establish female typology, by statistical methods.

THEORETICAL ASPECTS CONCERNING THE MAIN STAGES OF THE ANTHROPOMETRIC SURVEY AND KEY DIMENSIONS ASSIGNATION

The development of a dimensional typology takes up several steps, as follows:

- preparation of the anthropometric survey, aimed at establishing the volume of the selection, which must be uniform and representative;

- the anthropometric investigation;
- statistical processing of raw data obtained from the anthropometric survey;
- preparing the standard anthropometric draft;
- the anthropometric standard draft validation.

In our country, based on this research, all data about body measurement presented in this paper were gathered by performing a 3D scanning, method that required the acquisition of a powerful scanner by the project coordinator – I.N.C.D.T.P.

The survey was conducted in Romanian geographical areas, in order to have a representative selection. The sample of the female individuals was scientifically established, $n = 684$ and age between 20 to 65 years.

The 3D scanning method allows the measurement of many body dimensions; for each subject, 94 anthropometric dimensions were measured, such as: height, lengths, diameters, widths, arcs, angles and distances. This study will present aspects concerning "key" or main dimensions used to elaborate the female dimensional typology.

Main dimensions shall be those anthropometric dimensions used to underline the differences between the body types and those ones will serve to assign dimensional variants, which will turn out into clothing products. All other dimensions that would characterize in detail every of the body types will be secondary dimensions.

The key dimensions were chosen according to certain criteria:

- Main dimensions must describe all morphological features of the wearers from the population, for whom that body system is made;
- These dimensions are morphological indicators of the body and have the highest size level;
- These dimensions are being measured in different anthropometric plans and they are in correlation with different others having the same body orientation;
- Between these dimensions, there should be a relatively weak correlation, thus justifying their retention as main dimensions;
- Allow a wide interval of indifference.

The body height – I_c and the bust perimeter – P_b are located in different anthropometric planes, with a weak correlation, reasons for which they are morphological indicators used to describe the human body. At the same time, there are strong correlations between different anthropometric dimensions and these two main dimensions [2]. However, introducing only I_c and P_b as main dimensions is not enough to characterize the adult body types. Studies in this area have shown that, for the same value of the bust perimeter, the values of the waist perimeter (men body types) and hips perimeter (women body type) can vary greatly. Therefore, it is known that a single value of the hips perimeter for a specific measure of the bust perimeter is unsatisfactory for describing the human body (see products dimensional correspondence with the wearer's body). Values of hips perimeters best reflect the conformational changes occurring in woman adult population, under the influence of the age factor.

Thus, when developing woman dimensional typology, both worldwide, and in our country [3, 4], body height – I_c, the bust perimeter – P_b and hips perimeter – $P_ş$ are used as key dimensions.

RESULTS AND DISCUSSIONS

A first step in interpreting the anthropometric data obtained from the survey is to process the individual values by the mathematical-statistical method; each dimension is a random anthropometric variable and the values recorded one by one for the individuals are replaced by synthetic values, characteristic for the entire selection.

These values represent what is typical and representative for the studied variable, giving information about the scattering degree of individual values from the typical ones (average) and allowing us to broaden the conclusions over the entire community.

Each "key" dimension was subjected to statistical one-dimensional processing, according to the methodology known in mathematical statistics, using Microsoft Excel programme.

Thus, for a human body shape characterization, the following statistical parameters were computed:

- average value, $\bar{X}$;
- mode value, M_o;
- median value, M_e;
- amplitude, A;
- sample dispersion, S_x^2;
- sample deviation, S_x;
- coefficient of variation, C_v;
- significance test of average value, t_χ;
- sample deviation of average value, S_x;
- limit values of average sample, $\Delta\bar{X}$;
- limit values of the confidence interval for the average value determined for the entire collectivity ($\chi_{inf} = \bar{\chi} - \Delta\bar{\chi}$; $\chi_{sup} = \bar{\chi} + \Delta\bar{\chi}$).

Values standardized for the main dimensions – I_c, P_b and $P_ş$ – are core values for the classes established based on the average value and dimensional interval of each dimension.

In the first stage of the statistical one-dimensional processing, it is necessary to examine if the extreme values of main dimensions are outliers of the variation string.

In this sense, in order to specify the string values for key dimensions, it was necessary to examine whether the extreme values – X_{min}, X_{max} – can be maintained or they are outliers, so it will be necessary to remove them from the primary data. With this view, it was necessary to apply the $r_{max\ (min)}$ criteria [1], given by the large amount of the studied selection ($n = 673$ subjects) [1].

If $r_{max\ (min)} > r_{\alpha,\upsilon}$, in this case X_{max}, X_{min} are considered outliers in the variation string and should be deleted ($\alpha = 0.05$, $v = n - 2$).

Applying this method in successive stages, for each main dimension, there were established both the limits of the variation strings, and the main statistics parameters, comparatively shown in table 1.

Table 1

MAIN STATISTICAL PARAMETERS FOR THE KEY DIMENSIONS						
Main dimensions	$\bar{X}$, cm	X_{min}, cm	X_{max}, cm	S_x, cm	C_v, %	n
Body height, I_c	162.47	143.3	183.3	6.69	4.12	684
I_c reviewed	162.42	147.2	178.6	6.57	4.04	675
Bust girth, P_b	95.78	75.4	136	11.33	11.83	684
P_b reviewed	95.63	75.4	128.2	11.01	11.52	675
Hip girth, $P_ş$	104.2	80	134.4	9.74	9.34	684
$P_ş$ reviewed	104.06	84	133.8	9.38	9.01	675

Table 1 shows that recalculated average value and standard deviation do not differ significantly from the original data, so the central figure of selection (average body type, representative) does not change.

From table 1, we can also notice that, in the studied sample, the representative body type, the central figure, is defined by the following specific values for the key dimensions: 162.42 – 95.63 – 104.06, which determine a correspondent body type 160–96–104, the same resulted from the research carried out [3].

The values for the main dimensions – I_c, P_b, $P_ş$ – were mathematically processed with the Excel programmer, for the whole selection and for the small ones formed by age criteria. Table 2 shows the values for the main statistical parameters.

Analyzing the values from table 2, we inferred the following:

- For the first three age groups, the average values of I_c are very close to one another and slightly lower for the elderly one; it is noticed that the average value for each group has the same standardized value as for the whole sample (I_c = 160 cm);
- For each sample, the values of the variation coefficient are low (C_v < 10%). This value indicates a high homogeneity degree for the selection; body height has a lower variability, because the stature growing process is finished for the adult subjects (growing process is a major influence factor, which determines the body height variation);
- The values of the asymmetry coefficient show that the empirical distribution of body height is shifted to the left ($\gamma < 3$), which means a negative asymmetry;
- The vaulting coefficient values (v) show that I_c is an anthropometric dimension, which has a flat shape for the empirical distribution curve ($\beta < 0$), as compared to the normal curve distribution, because $v < 3$, for all cases;
- The excess coefficient values show that the empirical distribution curve shape is more flattened ($\beta < 0$), namely the particular values place further from the average, for both the total sample, and the small subsamples;
- I_c is an anthropometric dimension, which presents a high amplitude (about 30 cm), even inside the same age group. This result is confirmed by previous anthropometric surveys carried out [3] and by reality, too. It is something natural for an age group to comprise subjects with very different values of the body height;
- The significance test of the average value t_χ, for the whole sample and for the small ones, is higher than for the Student Test, which confirms that the

Table 2

THE MAIN STATISTICAL PARAMETERS TO CHARACTERIZE BODY HEIGHT, I_c					
Statistical parameters	**20–65 years old**	**20–29 years old**	**30–39 years old**	**40–49 years old**	**50–65 years old**
Average value	162.42	164.62	161.88	162.37	159.48
Standard deviation	0.25	0.40	0.64	0.51	0.46
Median value	162.40	164.20	161.90	162.45	159.50
Mode value	156.90	164.90	164.10	156.90	159.50
Sample deviation, S_x	6.57	6.30	6.82	6.15	5.94
Sample dispersion, S_x^2	43.12	39.72	46.45	37.79	35.23
Vaulting coefficient, v	–0.45	–0.60	–0.58	–0.32	–0.51
Asymmetry coefficient, γ	0.08	0.10	–0.03	0.09	0.11
Redundance coefficient, β	–3.45	–3.60	–3.58	–3.32	–3.51
Amplitude, A	31.40	28.80	28.10	31.40	26.70
X_{min}	147.20	149.00	148.20	147.20	147.30
X_{max}	178.60	177.80	176.30	178.60	174.00
C_v	4.04	3.83	4.21	3.79	3.72
t_χ	24.7	26.1	23.7	26.4	26.4
Sample volume	675	252	113	146	164

Table 3

THE MAIN STATISTICAL PARAMETERS TO CHARACTERIZE BUST GIRTH, P_b					
Statistical parameters	**20–65 years old**	**20–29 years old**	**30–39 years old**	**40–49 years old**	**50–65 years old**
Average value	95.63	87.62	95.94	100.65	103.26
Standard deviation	0.42	0.41	0.90	0.81	0.81
Median value	93.60	86.65	94.40	99.70	103.40
Mode value	84.30	84.30	91.00	99.70	93.60
Sample deviation, S_x	11.01	6.55	9.53	9.84	10.32
Sample dispersion, S_x^2	121.30	42.95	90.80	96.81	106.56
Vaulting coefficient, v	–0.26	3.84	0.06	–0.33	–0.42
Asymmetry coefficient, γ	0.59	1.20	0.60	0.34	0.11
Redundance coefficient, β	–3.26	0.84	–2.94	–3.33	–3.42
Amplitude, A	52.80	48.80	45.50	49.30	49.70
X_{min}	75.40	75.40	76.60	78.90	78.40
X_{max}	128.20	124.20	122.10	128.20	128.10
C_v	11.52	7.48	9.93	9.78	10.00
t_χ	8.68	13.3	10.1	10.2	10
Sample volume	675	252	113	148	164

selection is significant from the statistical point of view and all the conclusions drawn from its analysis can be extended to the community from which it was extracted.

By applying the same one-dimensional statistical analysis over the values of the bust perimeter, too, as it was done for I_c, for the whole sample and for the subsamples, we achieved the values presented in table 3.

Analyzing the results from table 3, the following inferences can be drawn:

- Women aged between 20 and 39 years present average values of the P_b that would correspond to the central value, the standardized value within which all the sample matches (P_b = 96 cm);
- For women aged between 40 and 49 years and the ones over 50 years, the P_b average value is larger, progressively, with a ratio equal to the inter-dimensional interval set for this anthropometric size (ΔPB = = 4 cm), result explained by the fact that, when a person becomes older and older, during ageing process, the width of the subcutaneous adipose tissue increases in the thorax region, having a non-uniform distribution;
- The bust perimeter P_b is a curvilinear dimension, with a very large amplitude, also reflected by the variability degree;
- The values of the variation coefficient (C_v = 10–20%) are in-between, because the physical and medical transformations determine a non-uniform distribution of the fat tissue;
- The values for the test of average value t_χ are bigger than the Student Test values, for each sample and for the whole one, and this confirms the idea that the selection is significant from the statistical point of view; so, the conclusions drawn from this analysis can be extended to the entire community.

Concerning the shape of the empirical distribution curve, evaluated by the vaulting, asymmetry and excess coefficients, it is noticed that there are appreciable differences between small samples and the whole one. This fact is explained by the wider variability of the bust perimeter values, already determined by the influence of the fat tissue and the muscular development degree. It is reported that the values of the excess coefficient are positive ($\beta > 0$) for young group; this result reflects the fact that private values are grouped around the average, and P_b has a period of stabilization (i.e. small variability degree).

The same statistical analysis is applied to the values of the hips perimeter $P_ş$, as it was done for P_b. The study was made for different samples; all results are given in table 4.

Data from table 4 show that:

- The average values for $P_ş$ are higher for older adults, as compared to the one obtained for the whole sample. The values of this perimeter are prioritarily determined by the fat tissue distribution, which grows with age;
- The variation coefficient values are small ($C_v < 10\%$), which proves a higher homogeneity degree for all samples studied;
- The values of the vaulting coefficient show a flat shape for the empirical distribution curve ($\gamma < 3$); the experimental values are far away from the average, in correlation with high amplitude. This conclusion is sustained by the value of the excess coefficient ($\beta < 0$), too.

In Romania, the last anthropometric survey was performed in 1997–1998, on a sample of 1,700 adult women subjects. The survey was carried out according to the classical method, by the academic staff from the *Knitting and Clothing Department*, within a research contract [3].

If we compare the average values of the key dimensions for the current sample and the one from 1998 (fig. 1), we notice the need for an ongoing review of the anthropometric standards (size systems for clothing).

From the graphic in figure 1, we see that the levels of the average values related to the main dimensions are

Table 4

THE MAIN STATISTICAL PARAMETERS TO CHARACTERIZE HIP GIRTH, $P_ş$

Statistical parameters	20–65 years old	20–29 years old	30–39 years old	40–49 years old	50–65 years old
Average value	104.06	98.03	105.02	107.59	109.51
Standard deviation	0.36	0.43	0.72	0.73	0.72
Median value	103.30	97.10	104.00	107.30	108.90
Mode value	98.30	96.30	104.40	105.20	106.10
Sample deviation, S_x	9.38	6.77	7.62	8.83	9.27
Sample dispersion, S_x^2	87.96	45.83	58.04	77.88	85.96
Vaulting coefficient, v	0.18	2.26	0.27	0.61	–0.10
Asymmetry coefficient, γ	0.61	1.12	0.61	0.34	0.41
Redundance coefficient, β	–2.82	0.74	–2.73	–2.39	–3.1
Amplitude, A	49.80	43.50	36.60	47.90	45.10
X_{min}	84.00	84.00	89.00	85.10	88.70
X_{max}	133.80	127.50	125.60	133.00	133.80
C_v	9.01	6.91	7.25	8.20	8.47
t_χ	11.1	14.5	13.8	12.2	11.8
Sample volume	675	252	113	146	164

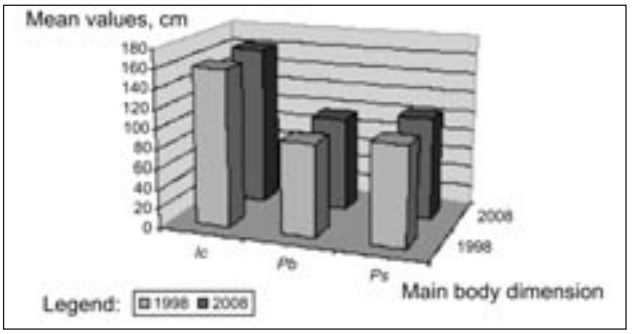

Fig. 1

higher in 2008, than in 1998. The influence of an acceleration phenomenon is reflected in the population size and shape and, thus, it calls for a periodical anthropometric survey, able to mirror the population dimensional evolution and to contribute to a proper dimensional typology up-dating according to the constant change determined.

CONCLUSIONS

Main dimensions are those that underline the differentiation between body types and represent dimensional variants for clothing products. These dimensions are basic information necessary for the sizing patterns of clothing products.

This paper defends the necessity for keeping the body height – I_c, bust perimeter P_b and hips perimeter $P_ş$ as main dimensions employed to describe the woman adult population. Mathematical and statistical methods were used to process all raw data obtained from a 3D body scanner. Data analysis led to the main conclusions below:

- the characterization of the main dimensions variability;
- a representative body type assignation for the sample studied: I_c = 160 cm, P_b = 96 cm, $P_ş$ = 104 cm;
- for the three main dimensions, standard values and limits of the indifference interval are set, the basic structure of an anthropometric standard for the Romanian women adult population.

The number of main dimensions, their variation area and limits values of their dimensional interval are conditioned by the number of body types from an anthropometric standard.

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Study on the measured error of thermal conductivity of fibrous porous materials. Part I. Error analysis

PENG CUI

FUMEI WANG
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REZUMAT – ABSTRACT – INHALTSANGABE

Analiza erorii de măsurare a conductibilității termice a materialelor fibroase poroase. Part. I. Analiza erorii

Având în vedere faptul că formula de calcul al conductivității termice $q = \lambda \frac{\Delta T}{H}$, derivată din ecuația diferențială de transfer al căldurii pentru materialele unicomponente continue, nu se aplică materialelor fibroase poroase, în lucrare este prezentat un model numeric, bazat pe metoda volumului finit, elaborat pe baza transferului de căldură integral, covectiv și conductiv, prin materiale fibroase poroase. A fost investigată eroarea de măsurare a conductibilității termice a materialelor fibroase poroase. Prin analiza rezultatelor simulării, s-a constatat faptul că eroarea măsurată a conductivității termice variază în funcție de conductivitatea termică, grosimea și coeficientul de permeabilitate, la materialele din vatelină, în special, eroarea fiind de cca 50%. În cazul materialelor fibroase poroase, utilizate în mod obișnuit, conductivitatea termică măsurată poate fi considerată drept conductivitate totală, în timp ce, în cazul altor materiale, conductivitatea termică măsurată variază în funcție de coeficientul de permeabilitate.

Cuvinte-cheie: eroare, conductivitate termică, materiale fibroase poroase, model numeric, metoda volumului finit

Study on the measured error of thermal conductivity of fibrous porous materials. Part. I. Error analysis

Given the fact that the formula for calculating the thermal conductivity $q = \lambda \frac{\Delta T}{H}$, which is derived from the heat transfer differential equation of single component continuum materials does not apply to fibrous porous materials, in this paper, a numerical model is presented, based on the integral convective and conductive heat transfer through the body of fibrous porous materials and numerically solved with the finite volume method. The measured error of thermal conductivity of fibrous porous materials was investigated. Through the analysis of the simulation results, it was found that the measured error of thermal conductivity varies with the thermal conductivity, thickness and permeability coefficient, especially for wadding materials, the error can be as high as 50%. For the commonly used fibrous porous materials, the measured thermal conductivity can be considered as completely conductive, whereas in other cases, the measured thermal conductivity varies according to the permeability coefficient.

Key-words: error, thermal conductivity, fibrous porous materials, numerical model, finite volume method

Die Messfehleranalyse der thermischen Leitfähikeit bei porösen Fasermaterialien. I. Teil. Fehleranalyse

Weil die Berechnungsformel der thermischen Leitfähigkeit $q = \lambda \frac{\Delta T}{H}$, abgeleitet aus der differentiellen Wärmetransfergleichung für ein-komponenten kontinuirlichen Materialien keine Anwendung bei porösen Fasermaterialien findet, wird in der Arbeit ein numerisches Modell vorgestellt aufgrund der Methode des finiten Volumens, erarbeitet auf Basis des integralen Wärmetransfers, konvektiv und konduktiv, für poröse Fasermaterialien. Nach der Analyse der Simulationsergebnissen wurde ermittelt, dass das gemessene Fehler der thermischen Leitfähigkeit in Abhängigkeit der thermischen Leitfähigkeit, der Dicke und des Permeabilitätskoeffizientes variiert, indem bei Watte-Materialien der Fehler ungefähr 50% annimmt. Im Falle der generell angewendeten porösen Fasermaterialien, kann die gemessene thermische Leitfähigkeit als Gesamtleitfähigkeit betrachtet werden, während, im Falle anderer Materialien, die gemessene thermische Leitfähigkeit im Abhängigkeit des Permeabilitätskoeffizienten variiert.

Schlüsselwörter: Fehler, thermische Leitfähigkeit, poröse Fasermaterialien, numerisches Modell, Methode des finiten Volumens

With the development of society, people demand more thermal comfortable materials, of which, the fibrous porous materials have a good thermal resistance, light weight and compress elasticity provided by the big amount of air between fibers, such as the wadding fabricated by wool or down, which provides a good shield to the cold environment [1–4].

To enhance the heat preservation performance of material, the heat conduction properties of materials should be considered, such as thermal conductivity, specific heat etc, among which the thermal conductivity is the most obvious indicator to reflect the heat attribute of a material. With more and more methods of measuring thermal conductivity emerging, from the consolidated to the transient ones [5–16], the boundary effect of the tested specimen is commonly ignored, the heat transfer occurring only in the thickness direction. But due to the practical thermal field of fibrous porous materials, held between the hot and cold plate, the assumption brings error to the measure of thermal conductivity, especially for fibrous porous materials with a high degree of penetration.

The study on the measured error of thermal conductivity is very important to enhancing the measuring accuracy of thermal conductivity. Many scholars have employed mathematical models or functions to represent the measured error of thermal conductivity [6, 10–13]. But until now, all the studies have been focused only on the single component continuum materials and none on fibrous porous materials, as theory or model. Only a few reported about the testing method of the heat conductivity property of fibrous porous materials [14–16], but none of the researches were dedicated to testing the accuracy of thermal conductivity of fibrous porous materials.

In the case of single component continuum materials, the interior heat is transformed by the single material, and the relevant factors of total heat flux through the body depend on the thermal physical properties of the component as well as the size of sample, such as the thermal conductivity, specific heat, thickness and so on.

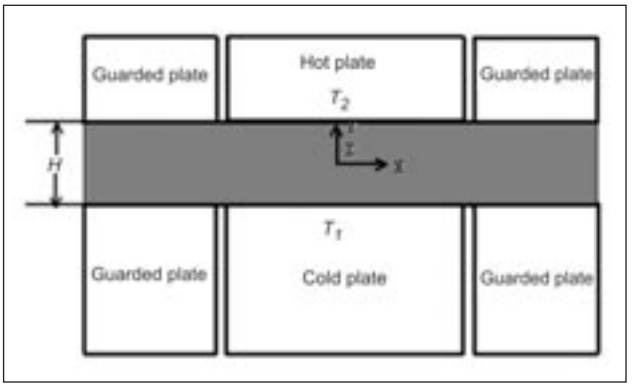

Fig. 1. Side view of the guarded hot plate

But regarding the fibrous porous materials, the interior heat transfer is integrated by the air and fiber together, which is of big difference to the principle of single component continuum materials. Not only the thermal physical properties of fiber and air as well as the size of sample, which are the characteristics of the single component continuum materials, but also the interior heat convection caused by the penetrating porosity, which were not considered by the former scholars, have an effect on the interior heat transfer.

In this paper, the integral conductive, convective and radioactive heat transfer equations are established in order to simulate the total heat flux through the body of fibrous porous materials. The tests were performed following the guarded hot plate's instructions. Through analysis based on simulation, the effect factors of the measured error of thermal conductivity were investigated, in order to form the basis for enhancing the testing accuracy.

DETECTION PRINCIPLE AND BASIC PHYSICAL EQUATIONS

Heat transfer equations of single component continuum materials

The construction of the guarded hot plate instruction is illustrated in figure 1. The sample is held between the hot pate and cold plate, which are rectangular with equal length and width, at temperatures between 30°C and 20°C, according to the measuring standard [17]. After a while from initiating the instruction, the temperatures of hot and cold plates are such as to ensure no heat loss along the edges of the two plates.

As shown in figure 1, when the heat reaches the ideal level, being transferred only in the thickness direction, as in the case of single component continuum materials, in the Cartesian coordinate system, the total heat flux q through the body is expressed as follows:

$$q = \lambda \frac{\partial T}{\partial x} \tag{1}$$

where:

λ is the thermal conductivity;

T – the temperature;

q – constant, the derivative of which is zero, through transformation the equation (1) can be rewritten as:

$$q = \frac{\lambda \cdot \Delta T}{H} \tag{2}$$

Table 1

PARAMETERS IN NUMERICAL SIMULATION, $T_m = 25^oC$		
Parameters	**Symbols**	**Scope**
Thickness, cm	H	0 ~ 5
Thermal conductivity, W/m · K	λ	0.03 ~ 0.3
Permeability coefficient, m^2	κ	$10^{-15} \sim 10^{-2}$
Porosity	ϕ	0.1 ~ 0.99
Air density, kg/m^3	ρ	1.185
Air specific heat, kJ/kg · K	c	1.005
Air dynamic viscosity, kg/(m · s)	μ	$18.35 \cdot 10^{-6}$

where:

ΔT is the temperature difference between the hot plate and cold plate;

H – the thickness of the tested specimen.

Commonly, formula (2) is used for counting the thermal conductivity of fibrous porous materials. We therefore used the evaluation of thermal conductivity based on the assumptions of one dimensional heat transfer of single component continuum materials to substitute the actual thermal conductivity of fibrous porous materials.

HEAT TRANSFER EQUATIONS OF FIBROUS POROUS MATERIALS

In the case of fibrous porous materials, the mechanism of heat transfer cannot be described by the equation (1), but it can be substituted with following energy equation [18]:

$$div\,(\rho u T) = div\,(a_e grad T) + q_R \tag{3}$$

where:

u is the volume average velocity;

q_R – the radiative heat flux, which is minor enough to be ignored for the small temperature difference between the hot plate, cold plate and guarded plate [19];

a_e – the thermal diffusion coefficient;

$a_e = \frac{\lambda}{\rho c}$ – were ρ and c are the material parameters, listed in table 1.

The equation (3) contains two variables, respectively temperature and velocity, both making the equation difficult to solve. But in the premise of small temperature differences between the hot plate, cold plate and guarded plate, the air can be regarded as incompressible and air flow can be treated as laminar within the body of fibrous porous materials. So according to the modified Brinkman-Forchheimer-Darcy model, the general governing equations are introduced as follows [18]:

Continuity equation:

$$div\,(\rho u) = 0 \tag{4}$$

Momentum equations:

$$div\left(\frac{\rho u u}{\phi}\right) = \frac{\partial(\phi p)}{\phi x} + div\left(\mu grad(u)\right) + F \tag{5}$$

$$div\left(\frac{\rho v u}{\phi}\right) = \frac{\partial(\phi p)}{\partial y} + div\left(\mu grad(v)\right) + F \tag{6}$$

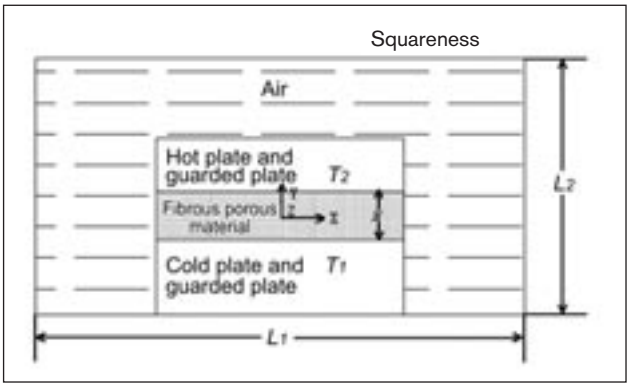

Fig. 2. Side view of the squareness

$$div\left(\frac{\rho wu}{\phi}\right)=\frac{\partial(\phi p)}{\partial z}+div\left(\mu grad(w)\right)+F \qquad (7)$$

where:

ρ and μ – are the static pressure and dynamic viscosity of the air;

F – the joined force exerted on the air;

$F=\frac{\phi\mu}{k}u+\phi G$, k and ϕ – the permeability coefficient and porosity;

$G=(\rho_{ref}-\rho)gk$, g – the gravity acceleration, k is unit vector in vertical direction,

$$\rho_{ref}=\rho(1-\beta\Delta T),$$

$$\beta=\frac{1}{T_m};$$

T_m – the reference temperature,

$$T_m=\frac{T_1+T_2}{2}.$$

As it can be seen in figure 1, apart from the boundary temperature of hot plate, cold plate and guarded plate, the left and right sides of the sample also have the heat convection with the ambient air. The heat transfer equations are written as:

$$-\lambda\frac{\partial T}{\partial x}=h\left(T_s-T_\infty\right) \qquad (8)$$

$$-\lambda\frac{\partial T}{\partial z}=h\left(T_s-T_\infty\right) \qquad (9)$$

where:

h is the heat transfer coefficient between both sides of the sample and ambient air;

T_s – the temperature on both sides of the sample;

T_∞ – the laboratory temperature, $T_\infty = T_1$.

In practical environment, the heat transfer coefficient h is hard to obtain, so a squareness is assumed, the length · width · height all being of 100 cm, regarding the dimensions of the guarded hot plate, as illustrated in figure 2. In air, the heat transfer equations are the same as those of fibrous porous material with the exception of equations (5) ~ (7), which should be rewritten as:

$$div\left(\rho uu\right)=-\frac{\partial(p)}{\partial x}+div\left(\mu grad(u)\right)+F \qquad (10)$$

$$div\left(\rho vu\right)=-\frac{\partial(p)}{\partial y}+div\left(\mu grad(v)\right)+F \qquad (11)$$

$$div\left(\rho wu\right)=-\frac{\partial(p)}{\partial z}+div\left(\mu grad(w)\right)+F \qquad (12)$$

where:

$$F=G=(\rho_{ref}-\rho)gk.$$

The temperatures and pressures on the boundaries of squareness are supposed to be equal, i. e.:

$$T\left(y=\frac{L2}{2}\right)=T\left(x=\frac{L1}{2}\right)=T\left(x=-\frac{L1}{2}\right)=$$
$$=T\left(z=\frac{L1}{2}\right)=T\left(z=-\frac{L1}{2}\right)=T_1 \qquad (13)$$

$$p\left(y=\frac{L2}{2}\right)=p\left(x=\frac{L1}{2}\right)=p\left(x=\frac{L1}{2}\right)=$$
$$=p\left(z=\frac{L1}{2}\right)=p\left(z=\frac{L1}{2}\right)=101355pa \qquad (14)$$

Through numerical computation, it is found under the supposition of boundary (13) ~ (14), the simulated heat flux q is invariable as the length and width are further extended, the squareness being higher than 20 cm as well as. This indicates that, as the squareness dimensions are set to 100 cm · 100 cm · 20 cm, the suppositions of boundary (13) ~ (14) are correct, thus computation space can be saved. Apart from the boundary conditions above, the other boundary conditions are as:

$$\frac{\partial T}{\partial n}=0 \qquad (15)$$

where:

n is the direction vector.

Through the procedures above, the heat convection between both sides of fibrous porous material and ambient air is converted to the heat transfer between different ingredients within the squareness, the numerical simulation of which can conveniently be solved.

Combining equations (3) ~ (7), and equations (10) ~ ~ (12), as the other given parameters of the fibrous porous materials, the total heat flux through the body of fibrous porous material can be numerically simulated according to thermal conductivity, and given the measured heat flux, thermal conductivity, inversely, could also be obtained.

Numerical method (FVM)

Within the squareness, the properties of the material dramatically varied near the interfaces between the fibrous porous material and air, so finite volume method (FVM) was applied to numerically solve the heat transfer equations [20].

A simple finite volume formulation was used for the discretization and the first-order Euler implicit formula [21] was used for temporal differences, because of its simplicity for implementation and unconditional stability in numerical computations.

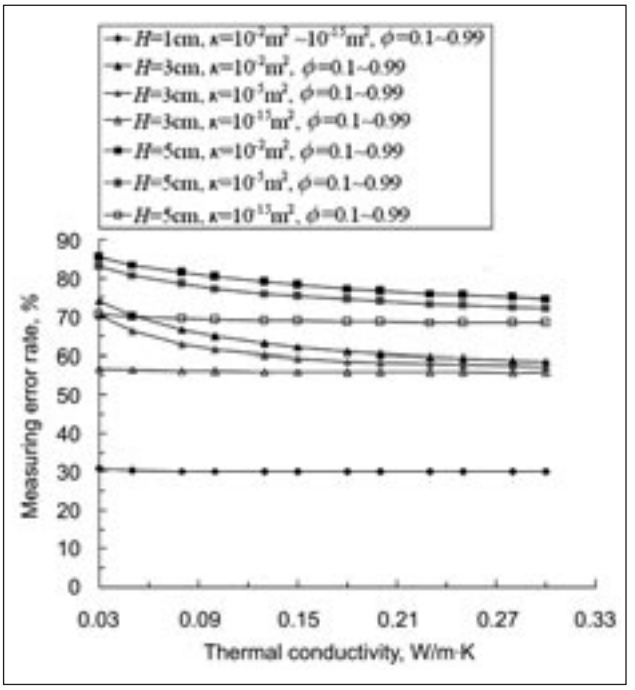

Fig. 3. Variation of the measured error rate with the increase of thermal conductivity

In non-linear problems the discretized equations are solved with iterative methods using an initial guess for the primitive variables and by giving an approximate solution. The iterative methods have the advantage of needing less computer memory as they take advantage of zero elements in the coefficient matrix. In this work, the simple [21] procedure was used for solving the discretized equations.

RESULTS AND DISCUSSIONS

According to geological and thermal physical properties of usual heat preserving materials, the scopes of geological and physical parameters used in numerical simulation are listed in table 1.

Measured error rate

Under given boundary conditions, the interior heat transfer behavior of fibrous porous materials is related with H, λ, k, ϕ, p, ρ as well as μ, and because p, μ and ρ are the determined constant in the experimental environment, therefore the total heat flux q through the body of fibrous porous materials varies with λ, ϕ k and H, i.e.:+26+

$$q = f(\lambda, \phi, k, H) \tag{16}$$

Changing the respective variable in formula (16), the corresponding total heat flux through the body of fibrous porous materials could be simulated and supposed to be measured with the guarded hot plate, while this time the thermal conductivity was assumed to be corresponding to the concrete fiber type and stack structure of fibrous porous materials, viz. actual thermal conductivity. Meanwhile, the thermal conductivity calculated with formula (2), based on the assumptions of single component continuum materials and one dimensional heat transfer, was regarded as λ'. For the convenience of comparative difference between λ and λ', the measuring error rate $\delta\lambda$ was defined as follows:

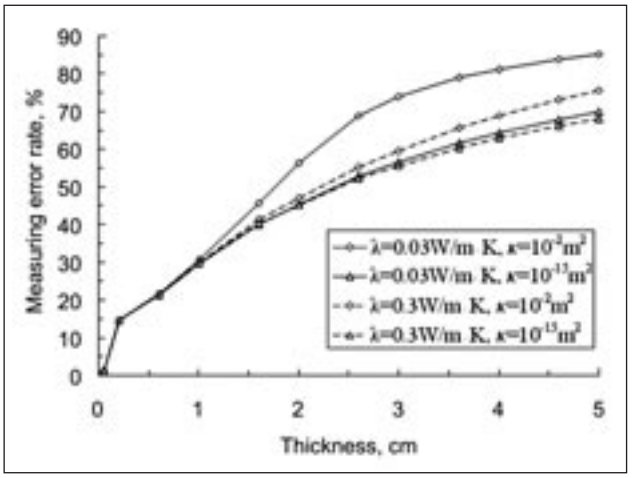

Fig. 4. Variation of the measured error rate with the increase of thichness

$$\delta\lambda = \frac{\lambda' - \lambda}{\lambda'} \cdot 100\% \tag{17}$$

Combing formula (16), it can be observed that the measuring error rate varies with the thermal conductivity λ, porosity ϕ, permeability coefficient k and thickness H. So with $\delta\lambda$ and the four variables related, we can explore the mechanism of geological and physical parameters interplaying with the measured error of thermal conductivity.

Figure 3 shows the variation of the measuring error rate as the thermal conductivity is increased from 0.03 to 0.3 W/m · K. It can be observed that the measuring error rate did not vary with the increase of porosity, whatever the other parameters are. This indicates the measuring error rate is only related with the thermal conductivity λ, the permeability coefficient k and thickness H. From figure 3, it can be noticed that when the thicknesses are of 3 cm and 5 cm and the permeability coefficients are 10^{-2} m^2 and 10^{-5} m^2, the measuring error rate decreases with the increase of thermal conductivity; when the thickness is 1 cm or permeability coefficient is 10^{-15} m^2, the measuring error rate does not vary with thermal conductivity. This indicates that the critical values are represented by the thickness and permeability coefficient, the measured error of thermal conductivity decreasing with the increase of thermal conductivity. The critical values of thickness and permeability coefficient will be discussed in figures 4 and 5.

Figure 4 shows the variation of the measured error rate of thermal conductivity for fibrous porous materials as the thickness increases. It can be noticed that the measured error rate is increased with the increase of thickness. This indicates that the thermal conductivity calculated based on the assumptions of single component continuum materials and one dimensional heat transfer, deviates from the actual thermal conductivity more and more as the thickness increases. Figure 4, also shows that when the thickness is approximately lower than 1 cm, the variation of the measured error rate of thermal conductivity is minor enough to be ignored. This indicates that the critical value of thickness, mentioned above, is of 1 cm. Besides, when thickness

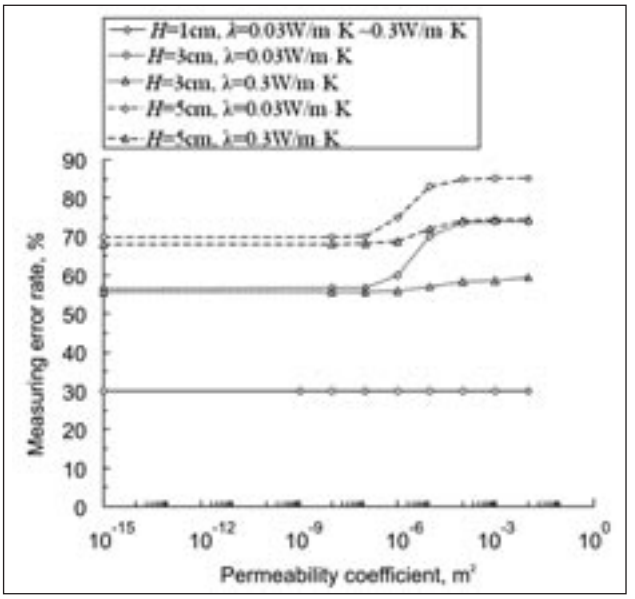

Fig. 5. Variation of the measured error rate with the increase of the permeability coefficient

is lower than 1 cm, not only the thermal conductivity but also the permeability coefficient are not related with the measured error rate. Therefore below thicknesses of 1cm, only the thickness influences the measured error of thermal conductivity. Another phenomenon can be observed from figure 4, respectively when thickness is 0.4 cm, the measured error rate is approximately 18%, while the thickness is 0.05 cm, the measured error rate is below 1%. This indicates that the measured thermal conductivity is close to the actual thermal conductivity only when testing fibrous porous material with very thin layer, such as normal textile.

Figure 5 shows the variation of the measured error rate as the permeability coefficient is increased. The permeability coefficient varies within a wide span, so the logarithmic coordinate was adopted for representing permeability coefficient. As can be noticed from figure 5, the measured error rate does not vary with the permeability coefficient when the thickness is 1 cm. At thicknesses of 3 cm and 5 cm, respectively, the permeability coefficient is higher than 10^{-4} m^2 or lower than 10^{-7} m^2, the measured error rate is not related with permeability coefficient; on the contrary, as the permeability coefficient is within the span of 10^{-7} m^2 to 10^{-4} m^2, the measured error rate increases with the increase of the permeability coefficient. This indicates that when the thickness is higher than 1 cm, the measured error of thermal conductivity is in piecewise function with permeability coefficient. Another conclusion drawn from figure 5 is that when the thickness is 3 cm and 5 cm, and respectively when the permeability coefficient is lower than 10^{-7} m^2, the measured error rate slightly varies with thermal conductivity; but when the permeability coefficient surpassed 10^{-7} m^2, the measured error rate decreases with the increase of thermal conductivity. This indicates that the critical value of permeability coefficient mentioned above is 10^{-7} m^2, thus only when the thickness is higher than 1 cm and permeability coefficient is higher than 10^{-7} m^2, the measured error of thermal conductivity decreases with the increase of thermal conductivity.

Observing from figures 3–5, the measured error rates are always positive whatever the other parameters are. This indicates that the thermal conductivity, calculated based on the single component continuum materials and one dimensional heat transfer, is higher than the actual thermal conductivity of fibrous porous materials. From figures 3–5, it can be noticed that the parameters λ, k, H are not simultaneously affecting the measured error rate, in some circumstance, only one or two of them being related with it. This provides the opportunity to separately discuss about the parameters in order to eliminate the measured error.

The effect of convective heat transfer rate

Under the experimental environment, the interior heat of fibrous porous materials can be transformed mainly in two ways, which are conduction and convection. The interior heat convection varies with different experimental environments, due to which the thermal conductivity of fibrous materials is not a constant. So in order to investigate the effect of interior convection on the thermal conductivity, the heat convection of fibrous porous materials under the experimental environment should be studied. But till now, there have been no reports about the convection effect under experimental environment on the thermal conductivity.

For investigating the effect of heat convection on the thermal conductivity, the assumption that no air flow exits in the body of the fibrous porous materials under the boundary conditions was made. Then the equation (3) is substituted with the following:

$$\frac{\partial^2(aT)}{\partial x^2}+\frac{\partial^2(aT)}{\partial y^2}+\frac{\partial^2(aT)}{\partial z^2}=0 \qquad (18)$$

Integral to the equations (3) and (18), the heat flux q_1' with convection and heat flux q_2' without convection can be derived, then $q_3' = q_1' - q_2'$, q_3' is the deviation caused by the convection in the body of fibrous porous materials. The affecting convection rate ζ, used for representing the response of measured thermal conductivity under the heat convection, is defined as follows:

$$\zeta=\frac{q_1'-q_2'}{q_1'}\cdot 100\% \qquad (19)$$

The convective effecting rate is obtained by calculating formula (19). For making a thorough investigation of the convection in the body of the fibrous porous materials, the variation of convective effecting rates with thermal conductivity under the different thickness and permeability coefficient are simulated, respectively, shown in figure 6.

Figure 6 shows the variation of the affecting convection rate as the thermal conductivity increases from 0.3 – 0.3 W/m · K at a thicknesses of 1 cm, 3 cm and 5 cm. The result is that the heat affecting convection rate decreases with the increase of thermal conductivity; it increases as thickness increases and decreases as the permeability coefficient decreases. When the thickness is 1 cm, the affecting convection rate is very low. These indicates that for the fibrous porous material of very thin layer, with thickness close to 1 cm

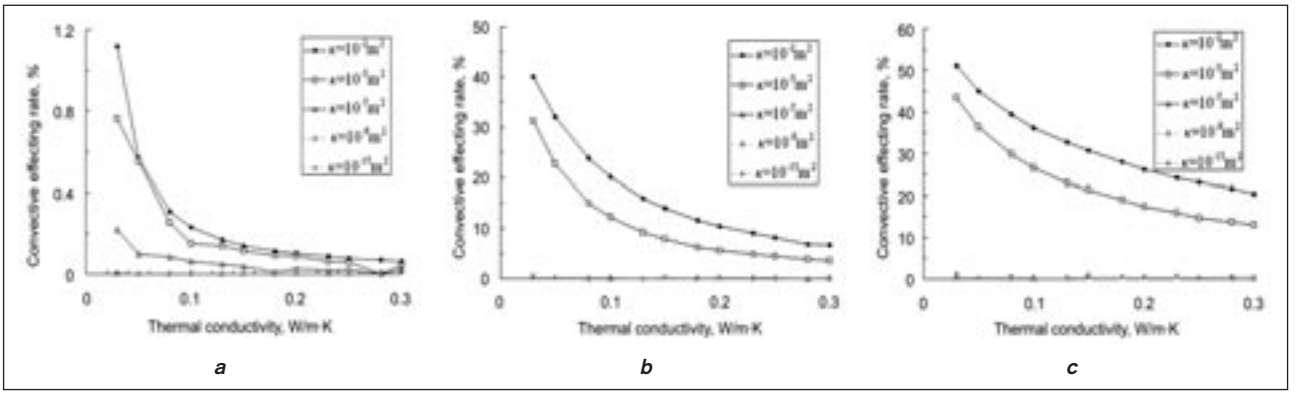

Fig. 6. Variation of the affecting convection rate as thermal conductivity increase:
a – Thickness 1 cm; ***b*** – thickness 3 cm; ***c*** – thickness 5 cm

or lower than 1 cm, the convection effect on the thermal conductivity is minor enough to be ignored, thus this time the interior heat transfer of fibrous porous materials can be considered as completely conductive, the measured thermal conductivities being completely conductive. In figure 6 *b, c*, it can be noticed that when the permeability coefficient is 10^{-2} m^2, the lowest value of the affecting convection rate is approximately 50%; when the permeability coefficient is 10^{-5} m^2, the lowest value of the convective coefficient is approximately 5%; when the permeability coefficient is reduced to 10^{-7} m^2, the affecting convection rate is below 1%. This indicates that when the permeability coefficients is close to 10^{-7} m^2 or lower than 10^{-7} m^2, the measured thermal conductivities of fibrous porous materials are completely conductive, due to the fact that the air flow within the body of fibrous porous materials is minor enough to be ignored.

EXPERIMENTAL PART

Different types of materials were selected for proving our analysis theory above, of which the measured error caused by the thickness is considered to be verified. We used two types of materials for manufacturing the wadding *A* and wadding *B* to different thicknesses. Due to the technical shortcoming of obtaining fabric *C* and nonwoven *D* in different thickness, the fabric *C* and nonwoven *D* of different layers were used, in order to substitute for the different thicknesses. The information regarding the samples is shown in table 2.

The KES-THERMO II equipment, which is produced in Japan, was used to measure the heat flux through the samples, then with the formula proposed by the equipment's manufacturer, i. e., $\lambda = \frac{q \cdot H}{\Delta T}$, the thermal conductivity could be measured, as shown in figure 7.

Table 2

THE SAMPLES INFORMATION				
Types of material	**Wadding *A***	**Wadding *B***	**Fabric *C***	**Nonwoven *A***
Material, mass fraction	70% camel hair+30% polyester	30% sheep hair+70% polyester	polyester	polyester
Thickness, cm	1.51, 2.72, 3.55, 4.84	1.42, 2.70, 3.94, 4.85	0.378 one layer	0.275 one layer
Permeability coefficient, m^2	$9.83 \cdot 10^{-9}$	$7.69 \cdot 10^{-9}$	$6.89 \cdot 10^{-13}$	$7.22 \cdot 10^{-10}$
Porosity, %	99.1	99.4	67.2	99.5

Figure 7 shows the variation of the measured thermal conductivities as thicknesses increase. The volume fraction of air contained in the wadding *A* or wadding *B* is approximately 99%, so the dominating heat transfer is medium.

Another major components of the fibrous porous materials is sheep and camel hair, the thermal conductivities of which are 0.0372 W/m · K and 0.052 W/m · K, besides, few polyesters are contained in the samples, the thermal conductivity of which is of 0.084 W/m · K . So depending on the ratios of the ingredients of the fibrous porous materials, their thermal conductivity should be close to that of the air and far from that of the fibers. Observing figure 7, it can be noticed that the thermal conductivities of wadding *A* or wadding *B* have already largely surpassed the conductivities of camel hair and sheep hair. With the growth of the thickness, some thermal conductivities of wadding *A* and wadding *B* are even greater than those of polyester, a phenomenon which is obviously against the physical law. From another point of view, this abnormality is fitted for our analysis in part 4.1, the conclusion being that thermal conductivity calculated on the assummptions of single component continuum materials and one dimension heat transfer, is greater than the actual thermal conductivity. Another phenomenon observed from figure 7 is that the fluctuation of the measured error rate against thicknesses is smaller in the case of fabric *C*

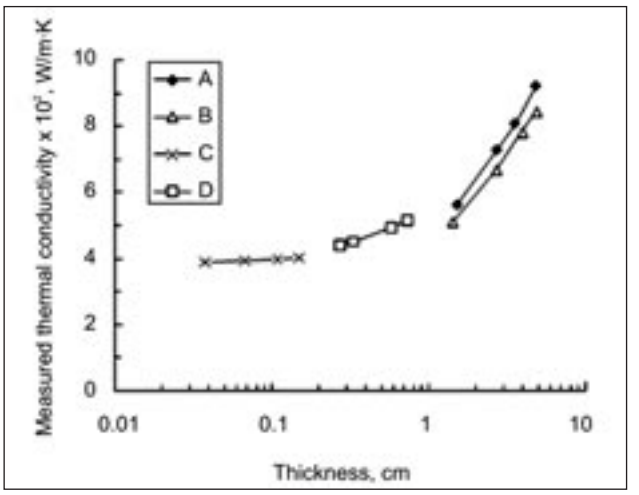

Fig. 47. Variation of the measured error rate with the increase of thichness

and nonwoven *D*, than in the case of wadding *A* and wadding *B*. The thicknesses of fabric *C* and nonwoven *D* being smaller than those of the wadding *A* and wadding *B*, this confirming our theory analysis, which claims that the measured error rate is in accordance with the thickness, in other words, the fluctuation of the measured error of thermal conductivity increases with the increase of thickness.

CONCLUSIONS

The guarded hot plate method is a universally used testing method for thermal conductivity. Due to heat loss and other reasons, some deviations occur during the test of thermal conductivity. The interior heat transfer mechanism of fibrous porous materials is very different from that of the single component continuum material due to the complexity of integral conductive and convective heat transfer. To explore the principle of the measured error of thermal conductivity of fibrous porous materials tested, a numerical simulation method was used and solved with finite volume method for studying the heat transfer through the body of fibrous porous materials tested, with the guarded hot plate. Based on the theory analysis and after experimental verification, the conclusions obtained are the following: the thermal conductivities of fibrous porous materials, which are calculated on the assumptions of single component continuum materials and one dimensional heat transfer, are higher than the actual thermal conductivities; the deviation of calculated thermal conductivity from actual thermal conductivity increases with the increase of thickness. This indicates that the assumptions of single component materials and one dimensional heat transfer are not appropriate for calculating the thermal conductivity of fibrous porous materials. When the thickness is higher than 1cm and permeability coefficient is higher than 10^{-7} m^2, the measured error of thermal conductivity decreases with the increase of thermal conductivity, but for thicknesses below 1 cm, the measured error of thermal conductivity only relates to thickness.

The measured error of thermal conductivity is in piecewise function with the permeability coefficient. When the permeability coefficient is higher than 10^{-4} m^2 or lower than 10^{-7} m^2, the permeability coefficient does not affect the measured error of thermal conductivity; on the contrary as the permeability coefficient is within the span of 10^{-7} m^2 to 10^{-4} m^2, the measured error of thermal conductivity increases with the increase of the permeability coefficient.

The effect of convection on the thermal conductivity of fibrous porous materials decreases with the increase of thermal conductivity; increases with the increase of thickness and decreases with the decrease of permeability coefficient. When the thicknesses of fibrous porous materials are very low, close to 1cm or lower than 1 cm, or the permeability coefficient is close to 10^{-7} m^2 or lower than 10^{-7} m^2, the heat transfer within the body of fibrous porous materials can be regarded as conductive and the measured thermal conductivity can be considered as completely conductive.

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DOCUMENTARE

Materii prime

MATERIALE COMPOZITE DIN MORCOVI

Compania ***Cellucomp*** a elaborat un nou material biocompozit de înaltă performanță, durabil, rezistent și cu greutate redusă. Noul material biocompozit este brevetat și are proprietăți mecanice deosebite față de polimerii armați cu fibră de sticlă (GFRP) și cei armați cu fibră de carbon (CFRP). Pentru producerea lui se folosesc metode de producție ecologice, care implică un consum scăzut de energie.

Curran – materialul, al cărui nume provine din cuvântul de origine celtică ce denumește morcovul, este o combinație între celuloza aflată în pereții celulari ai plantei și rășini speciale.

Utilizarea celulozei în fabricarea materialelor compozite este limitată din cauza faptului că rezistența, rigiditatea și duritatea acestora nu sunt destul de ridicate, comparativ cu cele ale fibrelor din sticlă sau carbon.

Până în prezent, producerea materialelor care să utilizeze pe deplin proprietățile nanofibrelor din componența celulozei, s-a dovedit a fi o provocare tehnică imposibilă.

Cellucomp a inventat și brevetat un procedeu prin care proprietățile nanofibrelor celulozice sunt utilizate la maximum. După mai mulți ani de experimentări, efectuate pe o gamă variată de materiale din plante, compania și-a optimizat tehnologiile de prelucrare a anumitor tipuri de legume, în special a morcovilor (fig. 1), a sfeclei de zahăr și a guliei suedeze.

Celuloza extrasă este combinată cu rășini speciale, care acționează pentru fixarea particulelor și care, după uscare, conferă impermeabilitate amestecului. Biocompozitele obținute au bune proprietăți de rezistență și de durabilitate și o greutate redusă.

Fig. 1

Materialele *Curran* sunt disponibile sub formă de folii și sunt compatibile cu diferite tipuri de rășini convenționale, precum cele epoxidice, cu poliuretanul și poliesterul.

Inițial, piața-țintă a companiei ***Cellucomp*** a fost cea a echipamentelor sportive, iar primele articole comercializate au fost undițele, în anul 2007. Undițele obținute din Curran sunt cu 25% mai ușoare decât cele din carbon, fiind adecvate atât pescuitului cu undițe ce implică aruncări pe rază scurtă, cât și pe rază lungă. Undițele, construite din Curran aplicat peste un miez ultrasubțire din carbon, au un coeficient ridicat de elasticitate, reprezentând o premieră mondială în domeniul proiectării undițelor (fig. 2).

Fig. 2

Materialul Curran are o largă aplicabilitate, de la industria de automobile la cea a aparatelor și bunurilor de consum.

Produsele fabricate din Curran sunt diferite de materialele biocompozite din gama fibrelor naturale destinate ranforsărilor din industria de automobile. În timp ce biocompozitele convenționale utilizează, de obicei, fibre cu un diametru de dimensiuni micrometrice și cu o lungime de câțiva centimetri, materialul elaborat de ***Cellucopmp*** folosește fibre celulozice de dimensiuni nanometrice.

Fibrele Curran au o rigiditate de 130 Gpa, o rezistență la rupere de până la 5 Gpa și o rezistență la deformarea sub sarcină de peste 5%, ceea ce face ca materialele compozite obținute din ele să aibă proprietăți unice, de înaltă performanță.

În prezent, ***Cellucomp*** elaborează o serie de alte produse pe bază de Curran.

Sursa: www.cellucomp.com

Surface treatments applied to textile materials and implications on their behaviour in wet conditions

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REZUMAT – ABSTRACT – INHALTSANGABE

Tratamente de suprafaţă aplicate materialelor textile şi implicaţiile acestora asupra comportării lor în mediu umed

Lucrarea prezintă rezultatele unor cercetări comparative privind modificarea comportării unor materiale textile în mediu umed, în urma aplicării unor tratamente fizico-chimice de suprafaţă. S-au utilizat trei variante de ţesături vopsite, din bumbac, poliamidă şi poliester. S-au aplicat tratamente în plasmă rece de înaltă frecvenţă, urmate de reacţii de grefare cu stiren şi tratamente chimice de hidrofobizare cu produse speciale. Proprietăţile hidrofobe ale materialelor obţinute au fost apreciate prin determinarea timpului de udare şi a unghiului de contact al picăturii de apă pe suprafaţă. Rezultatele au fost corelate cu permeabilitatea la vapori şi higroscopicitatea, determinate pentru probele martor şi variantele tratate. Eficienţa tratamentelor de suprafaţă a fost apreciată prin modificările cromatice ale probelor şi analiza aspectului acestora, folosind microscopia electronică cu baleaj.

Cuvinte-cheie: materiale textile, tratamente fizico-chimice, morfologia suprafeţei, permeabilitate, higroscopicitate, modificări cromatice

Surface treatments applied to textile materials and implications on their behavior in wet conditions

The paper presents the results of some comparative researches regarding the modification of textile materials behavior in wet conditions, subsequent to the applications of certain physical and chemical surface treatments. Three variants of dyed fabrics – cotton, polyamide and polyester – have been used. Surface treatments were applied in high-frequency cold air plasma, followed by grafting reactions with styrene and chemical treatments with special waterproofing products. The hydrophobic properties of the obtained materials were assessed by determining the absorption time and contact angle of water droplet on the surface. Results were correlated with the vapor permeability and hygroscopicity, determined for the control samples and the treated variants. The surface treatments effectiveness was assessed by samples color changes and the analysis of their morphology by scanning electron microscopy (SEM).

Key-words: textile materials, physical-chemical treatments, surface morphology, permeability, hygroscopicity, color changes

Oberflächenbehandlungen der Textilmaterialien und deren Einwirkung beim Nassverhalten

Die Arbeit präsentiert die Ergebnisse einiger Vergleichuntersuchungen über die Verhaltensveränderung einiger Textilmaterialien in Nassbedingungen, als Folge physisch-chemischer Oberflächenbehandlungen. Es wurden drei Varianten von gefärbten Gewebe verwendet, aus Baumwolle, Polyamide und Polyester. Es wurden kalte Plasmabehandlungen hoher Frequenz durchgeführt, gefolgt von Pfropfreakionen mit Stiren und chemische Hydrophobierungsbehandlungen mit speziellen Produkten. Die hydrophoben Eigenschaften der so erhaltenen Materialien wurden nach dem Bestimmen des Nassintervals sowie dem Kontaktwinkel des Wassertropfens auf der Oberfläche untersucht. Die Ergebnisse wurden mit der Dampfpermeabilität und der Hygroskopizität korreliert, welche für die Musterproben und die behandelten Varianten bestimmt wurden. Die Effizienz der Oberflächenbehandlungen wurde nach der chromatischen Veränderung der Proben und deren Aussehenanalyse untersucht, mit Hilfe der Rasterelektronenmikroskopie.

Schlüsselwörter: Textilmaterialien, physisch-chemische Behandlungen, Oberflächenmorphologie, Permeabilität, Higroskopizität, chromatische Veränderungen

Surface characteristics of the textile materials can be modified by treatments with various chemical agents, plasma treatments, Corona discharge, UV exposure etc. These treatments are carried out in order to improve the preexisting properties, or to create new properties. The consequence of such treatments is the change of the surface morphology, as well as the introduction of new functional groups [1–14].

Efforts to produce high quality, yet also low-cost products, determine the development of technologies with different objectives, such as: water repellent (waterproofing), oil repellent (oleo-phobic treatments), load reduction of static electricity, easy dirt removal etc. In the present paper, comparative studies have been carried out regarding the changes in the surface properties of certain textile materials, revealed through plasma treatments, grafting of styrene and by treatment with a hydrophobic/ oleo-phobic product [15–17].

EXPERIMENTAL PART

To develop the study cotton, polyamide and polyester fabrics have been used, all characteristics of the woven structure being presented in table 1.

The treatments were performed on white and dyed fabrics, as literature indicates the likelihood of their color change [22]. The cotton fabric was dyed with two types of dyes (C.I. Direct Black 103 and C.I. Reactive Red 4), polyester fabric with a dispersion dye (C.I. Disperse Red 74) and the polyamide fabric with an acid dye (C.I. Acid Red 85). Dyeing was carried out in the

Table 1

CHARACTERISTICS OF FABRICS					
Textile support	**Weight**, g/m^2	**Density,** yarns/cm		**Fineness**	
		warp	weft	warp	weft
Cotton, MB	104	305	240	*Nm* 140/2	*Nm* 40/1
Polyester, MPES	132	380	210	167/34 · 1 dtex	167/34 · 1 dtex
Polyamide, MPA	68	380	270	78/18 · 1 den	112/24 · 1 den

Table 2

TECHNOLOGICAL VARIANTS FOR PLASMA TREATMENT		
Treating variant	Time of activation in plasma, min.	Quantity of styrene used in grafting, cm^3
V_{1a}	10	0.25
V_{2a}	10	0.50
V_{3a}	10	1.00
V_{4a}	10	1.50
V_{5a}	10	2.00

following conditions: 3% dye at a liquor-to-goods ratio of 20:1, in stainless steel vessels in an infrared dyeing machine (Mathis "Polycolor P 4702") using the classic technology, indicated by the dye producing company. After dyeing, the samples were washed, dried and then subjected to the proposed surface treatments.

Treatments in cold high-frequency plasma

The samples were treated for 10 minute, in cold high-frequency plasma, and then grafted for 10 minute, with gaseous styrene in plasma at different concentrations (table 2).

To achieve the treatment, a laboratory installation was used with the following characteristics:

- air plasma 2–3 x 10^{-1} mbarr pressure;
- electrode tension 2.5 kV;
- electrical field force 100 V/cm;
- power 150 W;
- frequency 1.2 MHz;
- plasma temperature 34–35°C.

Hydrophobic/oleo-phobic treatments

The experiments have been carried out with the Tubiguard 66® product (a cationic perfluorinated resin – Bezema®) on three types of fabric (100% cotton, 100% polyester and 100% polyamide) in the following conditions:

- two minutes impregnation with X g/L Tubiguard 66® and 1 g/L acetic acid (table 3);
- 80% squeezing on a Benz pad;
- drying at 80°C and thermal treatment for 2 minutes at 150°C on a Minitherm Benz drying – heat setting – curing equipment.

The hydrophobic character of fabrics, due to the treatments performed, was assessed by determining the amount of absorbed water, W_{H2O}, the absorption time, t_{H2O}, and the contact angle [15–18]. For an overall characterization of surface modification, it was considered relevant the investigation of hygroscopicity, vapor permeability and air permeability [18–20].

The amount of water absorbed, W_{H2O}, was determined by the method of artificial rain [21], according to the following relation:

$$W_{H2O} = \frac{m_2 - m_1}{m_1} \quad [\%] \qquad (1)$$

where:

m_1 is the sample initial mass, g;

m_2 – the sample mass after the test, g.

The contact angle was determined by the goniometric method, measuring the angle between the water drop

Table 3

TECHNOLOGICAL TREATMENT VARIANTS WITH TUBIGUARD 66®	
Treating variant	Concentration, g/l
V_{1b}	10
V_{2b}	20
V_{3b}	30
V_{4b}	40
V_{5b}	50
V_{6b}	60

and the textile material surface. A special device built for this purpose was used.

The hydrophilicity was evaluated by determining the absorption time, W_{H2O}, of a distilled water drop placed on the fabric surface, according to AATC-39-1980 standard. For hygroscopicity assessment, 50 · 50 mm size samples were used, which were successively kept for 24 hours in a medium with standard atmosphere, φ = 65%, and moisture, φ = 100% [22, 23]. Vapor absorption capacity is determined by the following relation:

$$H_{abs} = \frac{M_u - M_c}{M_c} \cdot 100 \quad [\%] \qquad (2)$$

where:

M_u is the average mass of samples conditioned at relative humidity, φ = 100%, g;

M_c – the average mass of samples conditioned at relative humidity, φ = 65%, g.

The hygroscopicity index, i_H, is calculated by formula:

$$i_H = \frac{M_u - M_c}{S \cdot t} \quad [g/m^2 \cdot h] \qquad (3)$$

where:

S is sample surface, m^2;

t – duration for sample maintained in relative humidity, φ = 100%, h.

The capacity of fabrics to allow vapor passage was evaluated by calculating the vapor permeability index, μ, according to the established methodology:

$$\mu = \frac{\Delta M}{S \cdot t} \quad [g/m^2 \cdot h] \qquad (4)$$

$$\Delta M = M_0 - M_f \quad [g] \qquad (5)$$

where:

M_0 is the initial mass of the testing assemble (Herfeld container, water and sample), g;

M_f – the final mass of the assemble, g;

S – the sample surface subjected to vapor diffusion, m^2;

t – the time for sample maintained in diffusion conditions, h.

Another indicator considered relevant to characterize the treated materials is air permeability, P_a. The tests were conducted in accordance with SR EN ISO 9237, using an air flow-meter (type ATL-2) which allows the measurement of air flow through the fabric, in l/h. The computing formula is:

Table 4

TREATMENTS WITH STYRENE IN VAPOUR PHASE ON THE COTTON FABRICS							
Treating variant	Amount of water absorbed, %	Absorption time, s	Contact angle, degrees	Hygroscopicity		Permeability	
				H, %	i_H, g/m² · h	Vapor, g/m² · h	Air, m³/m² · h
V_{1a}	37.43	3 642	119	11.54	0.6463	14.44	48.21
V_{2a}	24.11	6 786	128	10.12	0.5668	15.18	46.19
V_{3a}	16.42	12 981	134	9.38	0.5253	15.49	44.10
V_{4a}	7.97	15 300	139	9.01	0.5047	15.88	43.82
V_{5a}	7.79	15 368	145	8.85	0.4957	16.10	42.92
M	73.26	2	0	12.82	0.7180	13.52	51.67

Table 5

TREATMENTS WITH STYRENE IN VAPOUR PHASE ON POLYESTER FABRICS							
Treating variant	Amount of water absorbed, %	Absorption time, s	Contact angle, degrees	Hygroscopicity		Permeability	
				H, %	i_H, g/m² · h	Vapor, g/m² · h	Air, m³/m² · h
V_{1a}	2.98	7 883	117	0.342	0.0076	16.19	27.68
V_{2a}	1.89	8 911	125	0.294	0.0064	16.87	26.64
V_{3a}	1.17	9 728	131	0.262	0.0057	17.08	25.40
V_{4a}	0.12	10 279	136	0.256	0.0054	17.24	24.46
V_{5a}	0.10	10 311	142	0.238	0.0051	17.38	24.12
M	11.13	138	10	0.380	0.0082	15.56	30.42

Table 6

TREATMENTS WITH STYRENE IN VAPOUR PHASE ON POLYAMIDE FABRICS							
Treating variant	Amount of water absorbed, %	Absorption time, s	Contact angle, degrees	Hygroscopicity		Permeability	
				H, %	i_H, g/m² · h	Vapor, g/m² · h	Air, m³/m² · h
V_{1a}	11.16	3 764	114	3.76	0.0580	15.32	16.28
V_{2a}	8.51	4 912	121	3.60	0.0556	15.75	15.78
V_{3a}	4.16	7 014	127	3.19	0.0493	15.96	14.91
V_{4a}	3.14	8 900	132	2.98	0.0474	16.48	14.48
V_{5a}	3.09	8 988	138	2.91	0.0461	16.90	14.44
M	16.09	1 020	70	4.08	0.0630	14.86	17.50

$$P_{a\Delta p} = \frac{q \cdot 10^{-3}}{60 \cdot A \cdot 10^{-4}} = \frac{q}{6A} \quad \left[\mathrm{m^3 / m^2 \cdot min.}\right] \qquad (6)$$

where:

q is the air flow passing through the tested fabric, l/h;

Δp – the pressure difference (in this case, Δp = = 10 mm water column);

A – the surface of the absorption hole, cm² (in this case, A = 11 cm²).

The surface morphology of treated and untreated samples has been studied by scanning electronic microscopy (SEM – Tesla BS 301).

The color of the samples has been measured by a spectrophotometer Spectraflash 300® produced by DATACOLOR, for the illuminant D 65/10. Using Micromath 2000® software, the color difference (ΔE*CIELAB) to standard (dyed samples, without chemical and physical-chemical treatments) was calculated.

RESULTS AND DISCUSSIONS

The results obtained for samples untreated and treated in plasma are presented in tables 4, 5, 6. Analyzing the experimental data, one can notice the higher values for the water absorption time and the contact angle, in the case of the three materials treated in plasma and grafted with styrene, compared to the untreated ones.

In absolute value, the most significant changes are obtained in the case of cotton fabrics, where the quantity of absorbed water for the variant V_{5a} of materials treated in plasma and grafted with styrene decreases almost 10 times, compared to the untreated cotton (reference sample) and slightly less, when compared to the untreated polyester and polyamide.

Table 7

EFFECT OF TUBIGUARD 66 TREATMENT ON POLYESTER FABRICS						
Treating variant	Amount of water absorbed, %	Contact angle, degrees	Hygroscopicity		Permeability	
			H, %	i_H, g/m^2 · h	Vapor, g/m^2 · h	Air, m^3/m^2 · h
V_{1b}	2.38	127	0.300	0.0067	16.76	28.24
V_{2b}	1.46	136	0.264	0.0059	17.54	27.46
V_{3b}	0.97	143	0.235	0.0052	17.78	26.94
V_{4b}	0.43	148	0.228	0.0050	17.93	24.59
V_{5b}	0.10	154	0.208	0.0046	18.03	23.81
V_{6b}	0.11	155	0.208	0.0046	18.03	23.72
M	11.3	10	0.360	0.0081	15.56	30.42

Table 8

EFFECT OF TUBIGUARD 66 TREATMENT ON POLYAMIDE FABRICS						
Treating variant	Amount of water absorbed, %	Contact angle, degrees	Hygroscopicity		Permeability	
			H, %	i_H, g/m^2 · h	Vapor, g/m^2 · h	Air, m^3/m^2 · h
V_{1b}	9.13	124	3.57	0.0595	16.15	16.05
V_{2b}	7.05	132	3.41	0.0534	16.62	15.36
V_{3b}	5.66	138	3.09	0.0484	16.84	14.83
V_{4b}	3.12	144	2.96	0.0463	17.39	14.22
V_{5b}	2.60	150	2.84	0.0445	17.82	13.08
V_{6b}	2.52	150	2.85	0.0446	17.82	12.55
M	16.09	70	4.02	0.0630	14.86	17.50

The values increase with the styrene concentration used for grafting and this behavior can be explained by hydrophobic characteristics of the polystyrene existent on the material surface. It is interesting that the contact angle value increases for all materials according to the amount of styrene used in grafting, and tends almost to the same value (varying slightly from 138 to 1 450, according to the nature of the nature of the textile material modified).

By increasing the amount of styrene used for grafting, the decrease of treated materials hygroscopicity, showed by hygroscopicity, *H*, and hygroscopicity index, i_H, is caused by the hydrophilic groups, decreasing accessibility in their chemical structure.

Increased vapor permeability for the samples grafted with styrene can be explained by the number reduction of groups capable to fix water or by their shielding with polystyrene grafted, which is chemically bound to the sample surface.

The air permeability decreasing reached by increasing the amount of monomer used in grafting can be explained by the presence of polymer deposited on the fibers surface, which causes the reduction of the interstices which allow the air to pass through the fabric.

The results obtained for the three fabrics, after the hydrophobic/oleo-phobic treatment, are presented in tables 7 to 9. As one can notice, the quantity of water absorbed by the three textile materials (polyester, polyamide and cotton) decreases as the concentration of the product used for treatment increases. This is explained by Tubiguard quantity reacting with fiber, respectively by the increase of hydrophobia produced by this through the chain $F_3C–(CF_2)_x$ – from the fluorine compound, which leads to water repellent and fatty substances rejection.

It is noted again that the most intense effect, in absolute value, is obtained for cotton (table 9), where, in the case of treatment with a solution of Tubiguard 66 concentration 30 g/l, a value of the water quantity absorbed by the material comparable with the untreated polyester and polyamide samples is achieved. At the maximum concentration used, a reduction exceeding 10 times the amount of water absorbed is achieved, as compared to the untreated sample.

In relative values, the most significant change of hydrophobia is recorded for polyester, the decrease of moisture retain being more than 110 times lower. The hydrophobic nature is reflected by the value of the distilled water drop contact angle on the fabric surface, as well. It was found that, for all the three textile materials, the contact angle values increased dramatically compared with the untreated material, as the fluorinated product concentration in the treatment baths increased. Maximum values were obtained for concentrations of 50 g/l. For higher concentrations the contact angle changes significantly.

Among the three fibers, the comparative analysis shows that the best results were obtained for cotton fabrics. It should be also noted that the results are

Table 9

EFFECT OF TUBIGUARD 66 TREATMENT ON COTTON FABRICS

Treating variant	Amount of water absorbed, %	Contact angle, degrees	Hygroscopicity		Permeability	
			H, %	i_H, g/m^2 · h	Vapor, g/m^2 · h	Air, m^3/m^2 · h
V_{1b}	31.02	128	10.52	0.5582	15.15	50.96
V_{2b}	20.01	139	9.21	0.4887	16.24	50.40
V_{3b}	13.63	146	8.54	0.4531	16.59	49.83
V_{4b}	8.50	152	8.19	0.4346	16.99	48.99
V_{5b}	6.51	158	7.99	0.4186	17.23	48.29
V_{6b}	6.39	159	7.98	0.4192	17.25	48.15
M	73.26	0	12.82	0.7180	13.52	51.67

Table 10

THE INFLUENCE OF GRAFTING CONDITIONS ON THE COLOR DIFFERENCE, ΔE^*

Grafting variant	Color difference, ΔE^*			
	Direct Black 103	Reactive Red 4	Acid Red 85	Disperse Red 74
V_{1a}	0.218	1.001	1.080	0.294
V_{2a}	0.344	1.376	1.402	0.558
V_{3a}	0.525	1.658	0.524	0.642
V_{4a}	0.864	2.366	1.237	1.187
V_{5a}	1.091	3.5582	3.046	1.273

Table 11

INFLUENCE OF PRODUCT TUBIGUARD 66® ON THE COLOR DIFFERENCE, ΔE^*

Variant	Color difference, ΔE^*			
	Direct Black 103	Reactive Red 4	Acid Red 85	Disperse Red 74
V_{1b}	0.706	0.445	1.202	2.016
V_{3b}	0.901	0.647	1.280	2.529
V_{4b}	1.003	0.859	1.378	2.538
V_{5b}	1.366	0.940	1.492	2.546
V_{6b}	1.872	1.338	1.529	2.652

perfectly consistent with those recorded after treatment in plasma, followed by grafting with styrene, for the three studied materials.

The hydrophobic and oleo-phobic nature of the materials treated with Tubiguard 66 is illustrated both by the high absorption times (>15 000 s) and by the spherical shape of water and oil drops on their surface. Also, as it can be noticed in the case of cotton fabrics, hygroscopicity decreases with the concentration increase of product used for treatment (Tubiguard 66). Regarding the vaporization coefficients, their values increase as hygroscopicity decreases, this showing that the treatment resulted in changes of the fibers structure, these becoming hydrophobic.

For materials made of synthetic fibers, polyester and polyamide (tables 6–8), hygroscopicity and hygroscopicity index decrease with the increase of product concentration in the treatment bath. Yet, vapor permeability increases by the increase in product quantity used, the highest values being achieved for the polyester fabrics. Also, air permeability decreases by increasing the concentration of product used for treatment. This can be explained by the decrease in the pore size due to fixation of the hydrophilicity treating product. In this sense, the lowest values were recorded in the case of polyamide fabrics with a higher density structure.

Regarding the influence of physical-chemical treatments for activation in plasma and grafting with styrene on color, the obtained results are presented in table 10. The data presented show that the color difference, ΔE^*, increases, versus the untreated dyed sample, by the increase of monomer amount used in grafting. This could be explained by the fact that, for the samples grafted with styrene, two main factors cumulate in the color change, namely – both the action of plasma, and the one of the polymer formed on the fiber surface. Regarding the dyestuff used for dyeing, the most significant color changes were obtained in the case of cotton fabrics dyed with Reactive Red 4 and in the case of polyamide dyed with Acid Red 85.

In the case of treatments with Tubiguard 66®, the color difference increases, for all dyes, as the product concentration in the impregnation baths increases (table 11). In the case of the same product concentration, the most significant color changes occur for the materials dyed with Direct Black 103 and Disperse Red 74.

The changes in the surface properties of cotton, polyester and polyamide samples have been revealed by means of scanning electron microscopy analysis and the resulted SEM images.

The analysis of plasma treated materials, in the absence of vinyl monomer, shows an increased fiber surface roughness, as a result of the material surface "corrosion" (fig. 1).

The action of cold plasma is reflected in the polymer destruction, accompanied by the generation of low-molecular products, even volatile, which can be easily removed from the fiber surface, creating visible artifacts. The mechanism of this destruction process lays at the basis of styrene or other vinyl monomer grafting. Thus, SEM images of plasma treated samples in the presence of styrene show the presence of vinyl polymer grafts. On the same line, by correlating electron microscopy analysis with the properties conferred by these plasma treatments, one can notice a good match and could conclude that the hydrophobic treatments of the

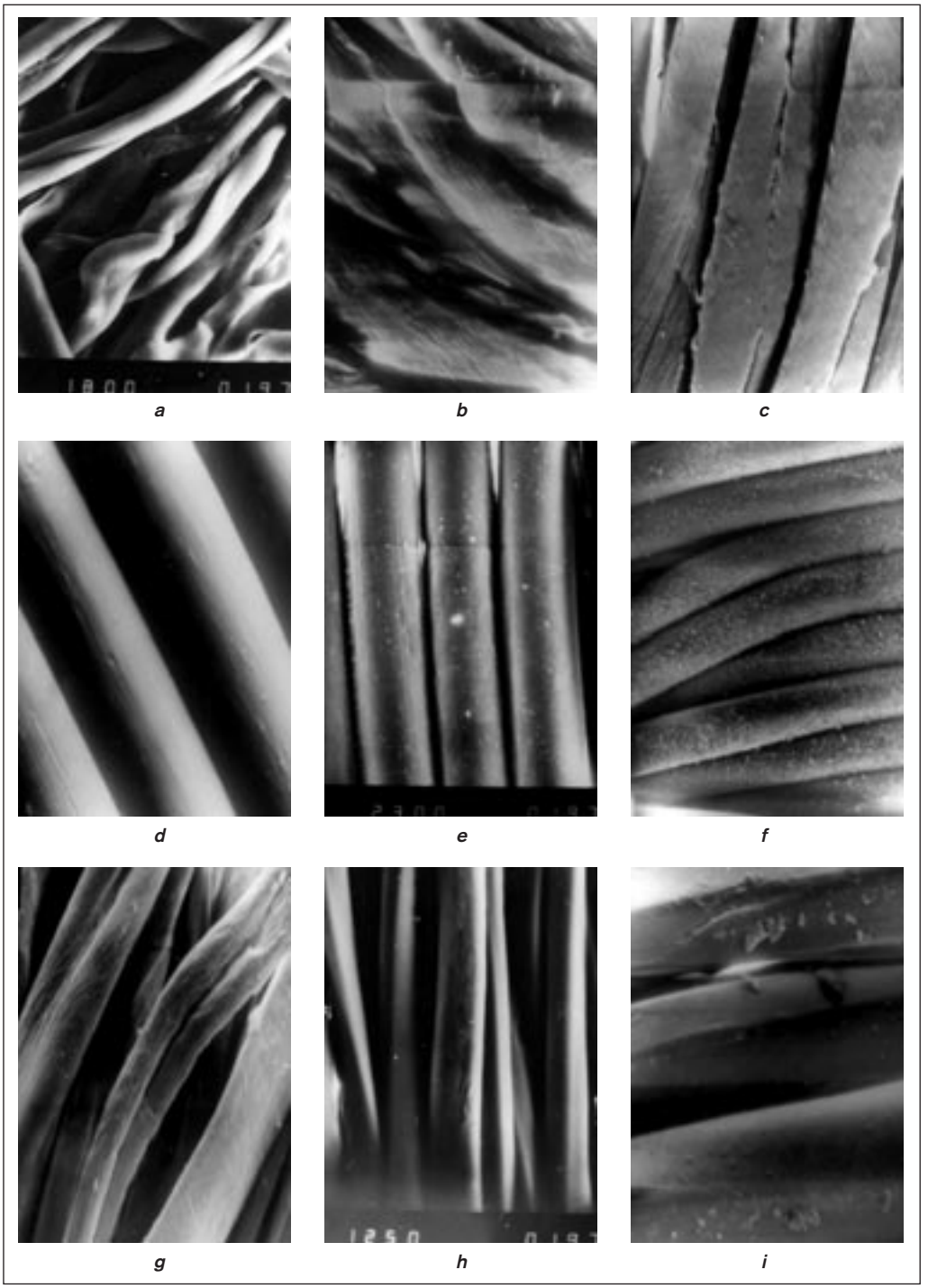

Fig. 1. SEM images if fibers grafted with styrene:
a – cotton – reference sample; ***b*** – cotton treated in plasma; ***c*** – cotton treated abd grafted in plasma; ***d*** – PA – reference sample; ***e*** – PA treated in plasma; ***f*** – PA treated and grafted in plasma; ***g*** – PES control sample; ***h*** – PES treated in plasma; ***i*** – PES treated and grafted in plasma

textile materials are the result of their grafting with polystyrene.

The SEM images of the textile materials treated with Tubiguard 66 show the presence of some roughness on the fiber surface, which can be attributed only to fluorinated products (fig. 2).

Once more, similarly to the material treated in plasma and grafted with styrene, there is a more uniform distribution on the surface of polyamide fibers, due to the product used for treatment.

CONCLUSIONS

- Plasma activation of the materials and plasma activation followed by grafting with styrene change the surface characteristics of the cotton, polyamide and polyester fibers;
- Grafting with styrene leads to increasing the hydrophobia of the three materials, the greatest differences between untreated and treated samples being noticed in case of cotton;
- The hydrophobic character for all the three textile materials increases as the concentration of fluorinated product in the treatment baths increases; maximum values are obtained for concentrations of 50 g/l;
- Physical-chemical treatments for grafting with styrene and chemical hydrophobic/oleo-phobic treatments lead to color changes of the materials dyed;

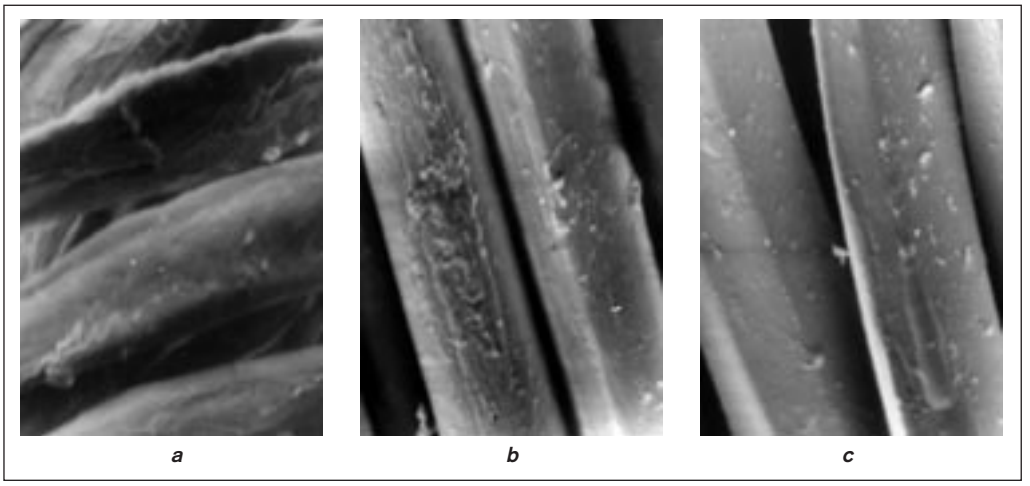

Fig. 2. SEM images of fibers treated with Tubigard 66:
a – cotton; ***b*** – polyamide; ***c*** – polyester

- The color difference increases with the concentration of monomer used in grafting and with the amount of product applied to the textile material in the impregnation baths;
- Color changes occurring in both physical-chemical (plasma followed by styrene grafting) and chemical (hydrophobic/oleo-phobic) treatments have close values, larger differences occurring in case of plasma treatments;
- The plasma treatments and grafting of textile materials with hydrophobic monomers will become an alternative for the classical treatments which are more expensive and not environmentally friendly.

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Biotechnologies for textile materials made of protein fibers. Enzymatic scouring of raw wool

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REZUMAT – ABSTRACT – INHALTSANGABE

Biotehnologii de tratare a materialelor textile din fibre proteice. Spălarea enzimatică a lânii brute

Experimentările de laborator cu produse enzimatice lipolitice, efectuate pe fibre din lână semigroasă, au avut în vedere găsirea unor soluții eficiente de spălare enzimatică a lânii brute, atât din punct de vedere al efectului de spălare și al protejării fibrei de lână în timpul acestui proces, cât și al impactului asupra indicatorilor apelor uzate. S-a urmărit influența utilizării în rețetele de spălare a lânii brute a unei anumite cantități de lipază, în combinație cu produse chimice uzuale de spălare, în vederea reducerii concentrației acestora. S-a studiat, de asemenea, modul în care adaosul de protează în flotele de spălare cu conținut de lipază acționează sinergetic pentru îmbunătățirea calității spălării.

Cuvinte-cheie: lipază, protează, spălare enzimatică, lână brută, protecția mediului

Biotechnologies for textile materials made of protein fibers. Enzymatic scouring of raw wool

Laboratory experimentations with lipolytic enzymatic products on the semi-fine wool fibers were aimed to find new efficient solutions for the enzymatic scouring of raw wool, both in terms of the scouring effect and wool fiber protection during scouring, and considering as well, the impact on the wastewaters pollution indicators. The influence exerted by the use of a lipase quantity combined with common scouring chemicals in the raw wool scouring recipes was monitored, in order to reduce their concentration. The influence of the protease addition in the lipase containing scouring baths was also studied, in order to point out whether this addition has a synergetic action over the scouring quality improvement.

Key-words: lipase, protease, enzymatic scouring, raw wool, environmental protection

Biotechnologien für die Behandlung der Textilmaterialien aus proteischen Faser. Enzymatisches Waschen der Rohwolle

Die Laboruntersuchungen mit lipolytischen enzymatischen Produkten, welche auf halbdicke Wollfaser durchgeführt wurden, hatten als Zweck die Bestimmung einiger effizienten Lösungen für das enzymatische Waschen der Rohwolle, sowohl vom Standpunkt des Waschеffektes und der Wollfaserprotektion während dieses Prozesses, als auch betreff des Impaktes auf die Abwasserindikatoren. Es wurde die Einwirkung einer bestimmten Menge von Lipase bei der Anwendung von Rohwoll-Waschrezepten untersucht, in Mischung mit generellen chemischen Waschprodukten, im Sinne der Reduzierung derer Konzentration. Es wurde gleichfalls die synergetische Wirkung des Zuschusses von Protease in den Waschflotten mit Lipaseinhalt für die Verbesserung der Waschqualität, untersucht.

Schlüsselwörter: Lipase, Protease, enzimatisches Waschen, Rohwolle, Umweltschutz

Raw wool contains a series of natural extraneous matter, whose distribution differs a lot in terms of wool fineness, varying between 5–40% for the greasy substances, 2–20% for suint, 5–40% for the mineral impurities and 0.5–12% for the vegetable impurities.

The greasy substances ("wool grease") are produced by the sebaceous glands and scouring is aimed to remove them up to a certain content, depending on the wool application (combed or carded), so that its further processing would not be negatively influenced.

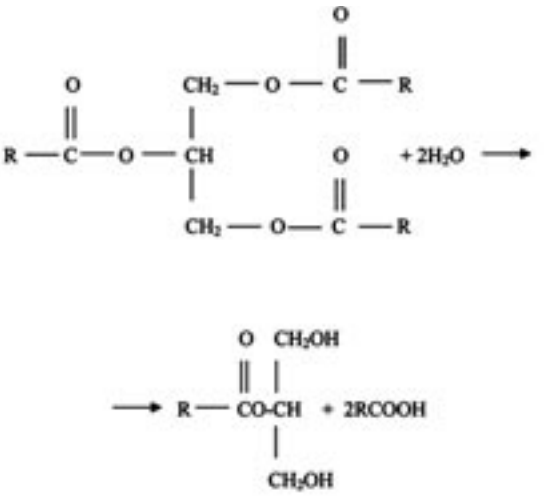
The traditional scouring procedure of raw wool is made on specific equipments (leviathans) made of successive scouring basins, containing aqueous solutions with high quantities of alkaline substances, such as sodium carbonate, and surfactants like soap or anionic or nonionic synthetic detergents based on fatty alcohols, at *p*H = = 9–10, or even 10.5–11 for the inferior wool class. The alkaline environment and the surfactants in the scouring baths have a negative influence over fiber integrity.

Analyses conducted on wool scoured in these conditions proved that, in strongly alkaline baths (*p*H = = 10–11), the first to be destroyed by surfactants are the nonpolar bonds (which mainly determine the proteins configuration, respectively their sensitivity to different reagents). The traditional scouring procedure is efficient in terms of wool greasy substances removal, yet, it generates wastewaters with a high content of alkali and surfactants.

Due to the ability of the lipolytic enzymes to hydrological divide the greasy substances from raw wool, the premises are already created for the achievement of some technically efficient solutions addressed to raw wool scouring, both from the perspective of the scouring effect and the wool fiber protection during scouring, and from the ecological viewpoint [1, 2].

Lipases hydrolyze esters of glycerin and of the fatty acids or different esters of the alcohols with a low molecular mass [3]. Lipase is an esterase, whose systemic name is triacyl-acyl-hydrolase (E.C. 3.1.1.3) [4].

Lipases catalyze with a high rate the lipids hydrolytic cleavage or the greasy substances cleavage, hydrolyzing the ester bonds from the triglycerides, in glycerin or fatty acids. The catalyzed reaction (1) can be illustrated as follows [5]:



Traditionally, lipases were obtained from the animal gastric or pancreatic juice. They can be now obtained

Table 1

CHARACTERISTICS OF THE ENZIMATIC EXPERIMENTED PRODUCTS

Enzymatic product	Commercial name	Activity*	Maximum activity at:	
			*p*H	Temperature, °C
Lipase	Lipomax CXT	1 000 DFU/g	9–10	55–60
Lipase	Lipase G 1 000	1 000 EU/g	6.5–7.5	40–50
Protease	Perizym AFW	22.93 U/ml	8.5–9	70

* *Notes: DLU* – lipolytic activity unit (Detergent Lipase Units), determined according to Genencor International internal standard; *EU* – esterase activity unit (Esterase Units), respectively the quantity of enzyme releasing 1 butyric acid micromole per minute, in the conditions of Genencor International Internal Standard; *U* – proteolytic activity unit, respectively the quantity of enzyme which, in standard conditions (37°C, *p*H = 7.8), hydrolyzes casein so that to release a quantity of soluble product per minute, giving the same color with the tyrosine mille-equivalent

of microbial cultures, as well through relatively simple procedures and especially less expensive [6].

The lipoprotein-lipases cleave selectively the lipoprotein bonds, meaning that the hydrophobic barrier will be also cleaved [3]. Lipases are enzymatic complexes formed of exo-lipases and endo-lipases [3, 7–8]. Unlike the other enzymes, the lipase shows specificity on the nature of the substrate onto which it will act, respectively on lipids, which are water insoluble. They are enzymes with a non-michaelian behavior.

In vivo, greases emulsification occurs at first, thus leading to the increase of the contact surface between the substrate lipo-soluble phase and the enzyme hydro-soluble one. Because of this, the initial reacting rate is dependent on the number of enzyme molecules absorbed at the interface, as it is generally low.

Further on, after the first generation of fatty acid molecules, the reaction rate increases, due to the faster emulsification of the substrate, the fatty acids molecules having hydrophobic areas and areas with hydrophilic properties with $RCOO^-$ groups. In this case, the rate of the substrate hydrolytic cleavage is not depending on the triglycerides concentration, but on the size of the contact surface at the interface of the two phases, namely on the emulsification degree.

In the following, characteristics of some lipases are shown, depending on the source they were achieved from [3, 9]:

- *Aspergillus niger, M* = 45 000 Da, optimal *p*H = 8.0, $T = 40^oC$;
- *Penicillium roqueforti, M* = 12 000 Da, optimal *p*H = 6.0–8.0, $T = 30–40^oC$;
- *Mucor javanicus, M* = 36 000 Da, optimal *p*H = = 5.5–8.5, $T = 30–45^oC$;
- *Pseudomonas fluorescens, M* = 36 000 Da, optimal *p*H = 5.0–9.0, $T = 40–65^oC$.

The use of lipases in the textile applications constitutes at present a topic for some studies conducted on global level. In the context of wool processing, the interest related to the lipolytic enzymes is relatively recent, with no solutions elaborated up to present for the industrial scale [10].

The lipases main application possibilities in the textile industry are:

- enzymatic degradation of suint and the greasy substances present in the raw wool;
- mild removal of the cotton impurities (waxes) without alkali;
- combined treatments of enzymatic desizing, by means of amylase/lipase, for the cotton woven fabrics, whose warp yarns were sized with starch-based recipes and tallow-based (triglycerides) lubricants.

Lipases combination with other textile enzymes is simple, due to the wide temperature and *p*H intervals for which they show an optimal activity. Lipases meant for textile applications have the optimal activity within the temperature range included between 50°C and 80°C, and 6 to 9 *p*H values.

ENZYMATIC PRODUCTS USED

For experimentations, lipolytic and proteolytic enzymatic products were used, in experimental and commercial variants, supplied by acknowledged producers:

- *Lipomax CXT* – Genencor International/Denmark (enzymatic product based on alkaline lipase obtained by genetic modification of Pseudomonas alcaligenes, encapsulated in a nonionic substance (polyethyleneglycol);
- *Lipase G 1000* – Genencor International/Denmark (lipolytic experimental product obtained of Rhizomucor miehei, selected and encapsulated in crystallized sodium chloride). It shows specificity for esters of the fatty acids with a short chain and hydrolyzes the natural greases;
- *Perizym AFW* – Textilchemie & Dr. Perty/Germany (catalytic product obtained by combining more types of protease, having a high specificity for the protein fiber).

The main physical-chemical characteristics of the enzymatic products specified are shown in table 1.

TREATING PROCEDURE

The scouring experiments conducted on raw wool were directed to monitor the influence over the scouring quality, exerted by the use of a lipase quantity, with or without additional protease content, in combination with the common scouring chemical products (the purpose being the one to reduce the quantity of chemical products used).

Scouring tests were performed on semi-fine wool fibers to settle the efficiency of the enzymatic scouring process, as well as to determine the influence of the enzymatic products over the wool, as compared to the classical scouring process based on commonly used chemical products (Na_2CO_3 and non-ionic surfactants). The codification of the experimented variants is given in table 2, while the parameters of the scouring process using lipolytic and proteolytic enzymes are presented in tables 3 and 4.

CODIFICATION OF THE EXPERIMENTED VARIANTS	
Variant code	**Description**
M	Semi-fine wool fiber, classically treated with Na_2CO_3 and surfactant in the first 3 scouring bath (control sample)
V_1	Semi-fine wool fiber scoured with a 2 g/L Lipomax CXT addition in the 3rd scouring bath and an approximate 44% reduction of the surfactant and an approximate 33% reduction in the Na_2CO_3
V_2	Semi-fine wool fiber scoured with a 4 g/L Lipomax CXT addition in the 3rd scouring bath and an approximate 44% reduction of the surfactant and an approximate 33% reduction in the Na_2CO_3
V_3	Semi-fine wool fiber scoured with a 4 g/L Lipomax CXT addition in the 3rd scouring bath and an approximate 66% reduction of the surfactant and an approximate 33% reduction in the Na_2CO_3
V_4	Semi-fine wool fiber scoured with a 4 g/L Lipomax CXT addition in the 3rd scouring bath and 3 g/l Lipomax CXT in the 4th bath, with an approximate 66% reduction of the surfactant and an approximate 33% reduction in the Na_2CO_3
V_5	Semi-fine wool fiber scoured with a 4 g/L Lipomax CXT addition in the 2nd bath and 4 g/l Lipomax CXT in the 3rd bath, with an approximate 94% reduction of the surfactant and 66% reduction of the Na_2CO_3
V_6	Semi-fine wool fiber scoured with a 1 g/L Perizym AFW addition in the 2nd bath, 4 g/L Lipomax CXT addition in the 3rd bath and 4g/L Lipomax CXT addition in the 4th bath, with an approximate 94% reduction of the surfactant and 66% reduction of the Na_2CO_3
V_7	Semi-fine wool fiber scoured with a 2 g/L Lipase G 1000 addition in the 3rd bath, with an approximate 45% reduction of the surfactant and 33% reduction of the Na_2CO_3
V_8	Semi-fine wool fiber scoured with a 4 g/L Lipase G 1000 and a 1 g/L Perizym AFW addition in the 3rd bath, with an approximate 45% reduction of the surfactant and a 33% reduction of the Na_2CO_3

RESULTS AND DISCUSSIONS

To assess the scouring efficiency, the content of greasy substances (metilen chloride extraction) was determined – according to SR 7690:1993, for the enzymatic scouring variants, as compared to the classically scoured control sample.

To assess the influence of the enzymatic products over the fiber characteristics, a series of physical mechanical indices (cystine-cysteine content – in conformity with SR ISO 2913:1996, solubility in alkali – in conformity with SR ISO 3076:1996) were determined, which are able to provide indications on possible fiber damage. The tinctorial test (Pauly) was also conducted [11, 12] to relieve the state of the cuticle (scaly) layer, after the scouring in the presence of lipolytic products, in comparison with the classical scouring variant.

Results achieved are included in table 5 and figures 1 and 2. The values obtained for the greases content (fig. 1) lead to the following conclusions:

- For the enzymatic scouring variants, the additional quantity of lipase in the 3rd and 4th scouring bath allows the decrease of the surfactant quantity up to 94% and of the sodium carbonate up to 66%, thus obtaining lower values of the greases content than in the case of the classically scoured variant (variants V_4 and V_5);
- Protease addition (variants V_6 and V_8) is not leading to the scouring efficacy improvement;
- There is no significant difference in the performance of products tested (Lipomax CXT and Lipase G 1000), for the same work conditions (variant V_2, as compared to variant V_7). The greases content resulted after scouring is similar.

As we analyze the solubility in alkali (fig. 2), we note the following:

- In the presence of lipase, all the scouring variants lead to lower values of the wool solubility in alkali, in comparison with the classically scoured variant, with no enzyme addition;
- The more we decrease the surfactant and the soda ash concentration and increase the lipase quantity, the lower it gets the wool solubility in alkali (even in a high 9.5 *p*H), pointing the fact the lipase hydrolyzes the wool greases with no damage on its structure;
- The tinctorial behavior of the samples subjected to Pauly test proves a minimal influence of the scouring variants V_5 and V_6, for which a light yellow color was obtained, identical to the one of the non-scoured raw wool sample.

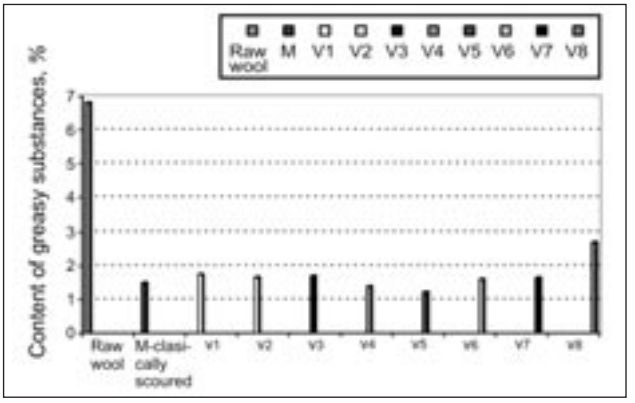


Fig. 1. Efficiency of raw wool scouring with lipolytic enzymatic products

IMPACT OF THE RAW-WOOL SCOURING ENZYMATIC TREATMENT OVER THE WASTEWATERS INDICATORS

In order to assess the load grade of the wastewaters (baths resulted from the raw wool scouring with lipolytic enzymes), the following treatment variants were selected:

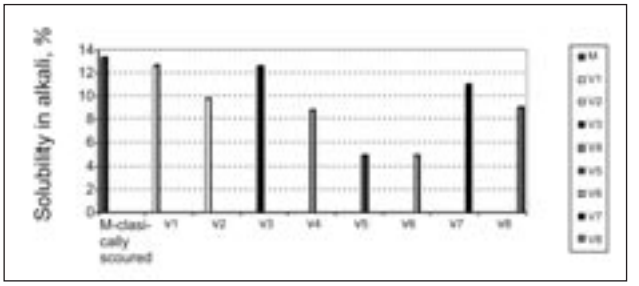


Fig. 2. Enzymatic treatment influence over the fiber integrity

RAW WOOL SCOURING VARIANTS WITH THE LIPOMAX CXT LIPOLYTIC ENZYMATIC PRODUCT, WITH OR WITHOUT THE ADDITION OF PERIZYM AFW PROTEASE (*Hm* = 1:70)

Variant	Scouring bath no.	Megapal VA, g/L	Na_2CO_3, g/L	Lipomax CXT, g/L	Perizym AFW, g/L	*p*H	Temperature, °C
M	1	0.5	2	–	–	9.5–10	45
	2	4.5	2	–	–	10	50
	3	4	2	–	–	10	50
	4	–	–	–	–	8	45
	5	–	–	–	–	7.5	35–40
	6	–	–	–	–	7	30
V_1	1	0.5	2	–	–	9.5–10	45
	2	4.5	2	–	–	10	50
	3	–	–	2	–	9.5	52
	4	–	–	–	–	8	45
	5	–	–	–	–	7.5	35–40
	6	–	–	–	–	7	30
V_2	1	0.5	2	–	–	9.5–10	45
	2	4.5	2	–	–	10	50
	3	–	–	4	–	9.5	52
	4	–	–	–	–	8	45
	5	–	–	–	–	7.5	35–40
	6	–	–	–	–	7	30
V_3	1	0.5	2	–	–	9.5–10	45
	2	4.5	2	–	–	10	50
	3	–	–	4	–	9.5	52
	4	–	–	–	–	8	45
	5	–	–	–	–	7.5	35–40
	6	–	–	–	–	7	30
V_4	1	0.5	2	–	–	9.5–10	45
	2	2.5	2	–	–	10	50
	3	–	–	4	–	9.5	52
	4	–	–	3	–	9.5	50
	5	–	–	–	–	8	45
	6	–	–	–	–	7.5	35
V_5	1	0.5	2	–	–	9.5–10	45
	2	–	–	4	–	9.5	52
	3	–	–	4	–	9.5	50
	4	–	–	–	–	8	45
	5	–	–	–	–	7.5	35–40
	6	–	–	–	–	7	30
V_6	1	0.5	2	–	–	9.5–10	45
	2	–	–	–	1	9.5	52
	3	–	–	4	–	9.5	50
	4	–	–	4	–	8	45
	5	–	–	–	–	7.5	35–40
	6	–	–	–	–	7	30

- Variant S_1 – raw wool scouring with lipolytic enzymatic products. The scouring tests were conducted on a semi-fine wool fiber, with the lipolytic enzymatic product Lipase G 1000. The work conditions for the variant S_1 are the ones shown in table 6.
- Variant S_2 – raw wool scouring according to a classical recipe. The work conditions used for this variant are the ones presented in table 7.

In order to assess the loading level of the wastewater resulted from the raw wool scouring with the lipolytic enzyme Lipase G 1000, the following indicators were determined: chemical oxygen demand (COD), according to the potassium dicrhromate method – SR ISO 6060:

1996; biochemical oxygen demand (BOD), 5 days after – according to SR EN 1899-2:2002, content of petroleum ether extractible substances – according to SR 7587:1996 and the solid residue – according to STAS 6953:1981.

The wastewaters coming from the enzymatic treatment of the textile materials were compared to the ones obtained by the raw wool classical scouring [12, 13]. The results achieved are shown in table 8.

RESULTS AND DISCUSSIONS

Results of the wastewaters indicators, achieved for the raw wool enzymatic scouring (for which 66% of the surfactant concentration and 33% of Na_2CO_3 concentration was reduced, as compared to the classical recipe), show a lower influence of the enzymatic treatment over the effluents:

- The BOD value is 44.7% lower than the one of the classically scoured variant;

Table 4

RAW WOOL SCOURING VARIANTS WITH THE LIPASE G 1000 LIPOLYTIC ENZYMATIC PRODUCT, WITH OR WITHOUT THE ADDITION OF PERIZYM AFW PROTEASE (*Hm* = 1:70)

Variant	Scouring bath no.	Megapal VA, g/L	Na_2CO_3, g/L	Lipase G 1000, g/L	Perizym AFW, g/L	*p*H	Temperature, °C
V_7	1	0.5	2	–	–	9.5–10	45
	2	4.5	2	–	–	10	50
	3	–	–	4	–	6.5	45
	4	–	–	–	–	–	45
	5	–	–	–	–	–	35–40
	6	–	–	–	–	–	30
V_8	1	0.5	2	–	–	9.5–10	45
	2	4.5	2	–	–	10	50
	3	–	–	4	1	7	45
	4	–	–	–	–	–	45
	5	–	–	–	–	–	35–40
	6	–	–	–	–	–	30

Table 5

PHYSICAL-CHEMICAL CHARACTERISTICS OF THE CLASSICALLY AND ENZYMATICALLY SCOURED RAW WOOL

Treated variants	Determined characteristics	
	Content of greasy substances, %	Solubility in alkali, %
Raw wool	6.8	13.32
Classically scoured control sample	1.5	12.6
V_1	1.75	9.74
V_2	1.66	12.59
V_3	1.7	8.76
V_4	1.39	5
V_5	1.22	5
V_6	1.6	11.03
V_7	1.65	9.04
V_8	2.7	–

Table 6

WORKING PARAMETERS FOR THE RAW WOOL ENZYMATIC SCOURED

Variant	Scouring bath no.	Megapal VA, g/L	Na_2CO_3, g/L	Lipase G 1000, g/L	*p*H	Temperature, °C
S_1	1	0.5	2	–	9.5–10	45
	2	2.5	2	–	10	50
	3	–	–	4	9.5	52
	4	–	–	3	9.5	50
	5	–	–	–	8	45
	6	–	–	–	7.5	35

- The COD value is 44% lower than the one of the classically scoured variant;
- The content of petroleum ether extractible substances is 67.7% lower than the one of the classically scoured variant;
- The solid residue is comparable to the one of the classically scoured variant;
- The BOD/COD ratio, providing data on the chemically degradable fraction of organic matter, is amounted to 0.74, for both treatments, indicating that the biodegradable fraction of organic matter is large.

Note: A value of the BOD/COD ratio ranging between 0.3-0.5 denotes a difficult bacterial attack and an important fraction of bio-resistant organic matter present in the wastewater.

CONCLUSIONS

The raw wool scouring tests conducted at laboratory level on semi-fine wool fibers, by using the Lipomax CXT and Lipase G 1000 lipolytic enzymes, with or without an additional content of protease Perizym AFW, in combination with the commonly used chemical scouring products, led us to the following conclusions:

- The addition of a lipase quantity in the 3rd and the 4th scouring bath, in the case of the enzymatically scoured variants, allows an up to 94% decrease of the surfactant quantity and an up to 66% decrease of the sodium carbonate, thus obtaining values of the greases content lower than for the classically scoured variant;

Table 7

WORKING PARAMETERS FOR THE RAW WOOL SCOURED ACCORDING TO A CLASSICAL RECIPE					
Variant	**Scouring bath no.**	**Megapal VA,** g/L	**Na_2CO_3,** g/L	***p*H**	**Temperature,** °C
S_1	1	0.5	2	9.5–10	45
	2	4.5	2	10	50
	3	4	2	10	50
	4	–	–	8	45
	5	–	–	7.5	35–40
	6	–	–	7	30

Table 8

ASSESSED WASTEWATERS INDICATORS		
	Raw wool scouring	
Determined indicators	**Sample S_1 enzymatic**	**Sample S_2 classical**
BOD, ml/L	585.3	1 060.2
COD, mgO_2/L	785.2	1 404.6
Petroleum ether extractible substances, mg/L	40	124
Solid residue, mg/L	3 334	3 242

- The performance of the tested lipolytic enzymes proved no significant differences for the same work conditions; the greases content after scouring was similar;
- In the presence of lipase, all the scouring variants lead to lower values of wool solubility in alkali, as compared to the classically scoured variant with no enzyme addition;
- The more we decrease the surfactant and soda ash concentration once with the increase in lipase quantity, the wool solubility to alkali becomes lower, pointing the fact the lipase hydrolyzes the wool greases causing no damage to its structure;
- The protease addition is not generating an improvement of the scouring efficiency;
- The tinctorial behavior of the samples subjected to the Pauly test reveals a minimal influence of some enzymatic scouring variants, for which a light yellow color was obtained, identical to the one of the non-scoured raw wool sample.

Corroborating the values obtained for the greases content with the values obtained for the solubility in alkali, the best variant in terms of the scouring efficiency and of the fiber protection is the one with a 4 g/L Lipomax CXT addition in the 2nd bath and a 4 g/L Lipomax CXT addition in the 3rd bath, for which the surfactant concentration had an approximate 94% decrease, and the Na_2CO_3 concentration an approximately 66% decrease.

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Influence of finishing treatments on the physical-mechanical characteristics of cotton knitted fabrics

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REZUMAT – ABSTRACT – INHALTSANGABE

Influența tratamentelor de finisare asupra caracteristicilor fizico-mecanice ale tricoturilor din bumbac

Lucrarea prezintă modificările caracteristicilor fizico-mecanice ale tricoturilor obținute din fire de bumbac, cu structură glat, apărute în urma operațiilor de finisare aplicate acestora. S-a ajuns la concluzia că condițiile de vopsire au o influență hotărâtoare asupra caracteristicilor lor fizico-mecanice: desimea pe orizontală, desimea pe verticală, lungimea firului din ochi, grosimea tricotului, masa unității de suprafață, forța și alungirea la rupere, extensibilitatea și efectul de piling. Experimentările s-au efectuat folosind metoda regresiei multiple, variabilele independente fiind durata procesului de vopsire, temperatura de vopsire și pH-ul băii de vopsire, iar variabilele dependente forța de rupere și alungirea la rupere.

Cuvinte-cheie: tricoturi, bumbac, structură glat, caracteristici fizico-mecanice, finisare, metoda regresiei multiple

Influence of finishing treatments on the physical-mechanical characteristics of the cotton knitted fabrics

The paper presents changes of the physical and mechanical characteristics for the cotton flat knitted fabrics occurring after the finishing operations applied. It was concluded that dyeing conditions have a decisive influence on the physical-mechanical properties of the knitted fabrics, such as: wale density, course density, yarn length in the loop, knitted fabric thickness, mass per unit surface, break force and elongation, stretching and the pilling effect. Experimentations were conducted by using the multiple regression method, the independent variables representing the duration of the dyeing process, dyeing temperature and dyeing bath pH, and the dependent variables represent the breaking force and the breaking elongation.

Key-words: knits, cotton, flat knitting, physical-mechanical characteristics, finishing, multiple regression method

Die Wirkung der Veredlungsbehandlungen auf die physisch-mechanischen Eigenschaften der Baumwollgewirke

Die Arbeit stellt vor die Modifizierung der physisch-mechanischen Eigenschaften der Flachgewirke aus Baumwollgarnen, als Folge der angewendeten Veredlungsoperationen. Man ist auf die Schlussfolgerung gekommen, dass die Färbungsbedingungen eine entscheidende Rolle auf die physisch-mechanischen Eigenschaften haben: Horizontaldichte, Vertikaldichte, Maschenlänge, Maschendicke, Oberflächeneinheitsmasse und Bruchdehnung, Dehnungsvermögen und Pilleffekt. Die Untersuchungen wurden anhand der mehrfachen Regression durchgeführt, indem unabhängige Variablen die Zeitdauer des Färbeprozesses, die Färbungstemperatur sowie der pH der Flotte sind, und abhängige Variablen Bruchkraft und Bruchdehnung.

Schlüsselwörter: Gewirke, Baumwolle, Flachwirken, physisch-mechanische Eigenschaften, Veredlung, Mehrfache Regressionsmethode

In a previous article the results regarding the influence of washing degreasing treatments on the structural and functional characteristics of knitted fabrics from dyed blended yarns were presented. It was concluded that the fibre composition of yarns used in treatment has a decisive influence on the main characteristics of plain-weft-knitted fabrics such as: horizontal stitch density, vertical stitch density, the loop length, thickness, mass per unit area, breaking force and elongation, extensibility and pilling effect. The purpose of this work is to establish the influence of dyeing conditions on the plain-weft-knitted fabrics made of 100% cotton yarns. The experiments were performed using a multiple regression method, taking as:

- independent variables:
  - X_1 – dyeing time process, minutes;
  - X_2 – dyeing temperature, °C;
  - X_3 – *p*H dyeing bath;

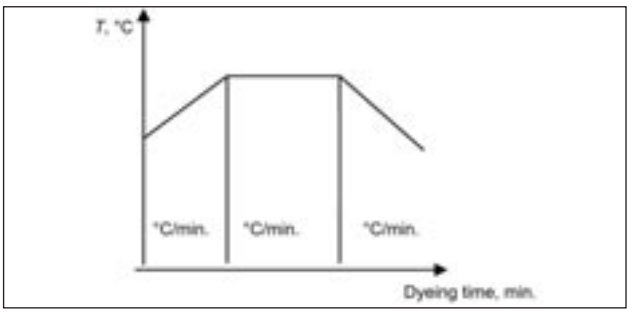


Fig. 1. Dyeing diagram with direct dyes on cotton knits

- dependent variables:
  - Y_1 – breaking force;
  - Y_2 – elongation.

EXPERIMENTAL PART

For this study raw knitted samples made of 100% cotton yarns were used and subjected to dyeing operations using a direct dyestuff (C.I. Direct Red). The dyeing bath composition: 3% dyes; 10% sodium chloride; bath ratio 1:20; *p*H alkaline – accomplished with sodium carbonate (Na_2CO_3).

A Mathis Polycolor dyeing installation was used, according to the following chart (fig. 1).

Codification of independent variables is presented in table 1 and the experimental plan and the physical-mechanical characteristics values of knitted fabrics in table 2. The dyed samples, subsequently dried without tensioning, were tested in order to determining their breaking force and elongation, using a dynamometer type Tinius Olsen DTK H5, the results were listed in table 2.

Table 1

CODIFICATION OF INDEPENDENT VARIABLES						
Variable	Code	Level				
		–1.682	–1	0	+1	+1.682
Dyeing time, min.	X_1	30	36	45	54	60
Dyeing temperature, °C	X_2	80	84	90	96	100
*p*H dyeing bath	X_3	6	6.5	7	7.5	8

Table 2

DYEING EXPERIMENTAL PLAN AND PHYSICAL-MECHANICAL CHARACTERISTICS VALUES OF KNITTED FABRICS

Nr. exp.	X_1, min.	X_2, °C	X_1, *p*H	Breaking force, Y_1, N		Breaking elongation, Y_2, %	
				Ş	R	Ş	R
1	–1	–1	–1	343.2	216.3	68.2	141.0
2	+1	–1	–1	341.2	222.3	69.0	141.0
3	–1	+1	–1	341.4	220.8	65.5	146.0
4	+1	+1	–1	344.4	220.0	65.9	143.8
5	–1	–1	+1	348.4	219.8	60.7	143.1
6	+1	–1	+1	347.6	212.3	66.2	141.1
7	–1	+1	+1	345.2	222.8	62.9	146.9
8	+1	+1	+1	342.4	222.0	61.8	145.8
9	–1.682	0	0	344.8	219.8	63.6	144.0
10	+1.682	0	0	348.8	222.8	63.4	144.6
11	0	–1.682	0	341.4	218.3	61.1	148.6
12	0	+1.682	0	341.0	222.3	60.2	147.5
13	0	0	-1.682	349.6	215.2	60.4	144.6
14	0	0	+1.682	344.8	214.5	61.2	141.0
15	0	0	0	345.2	230.8	69.0	142.8
16	0	0	0	343.6	232.3	69.1	143.0
17	0	0	0	344.0	236.2	69.3	143.0
18	0	0	0	344.0	236.2	69.3	143.0
19	0	0	0	344.0	236.2	69.3	143.0
20	0	0	0	344.0	236.2	69.3	143.0

Based on these data, using a factorial rotatable central program of order II, the following regression equations were obtained, in order to determine the correlations between the knitted fabrics characteristics marked with Y_1, Y_2 and the three variables considered for the study.

RESULTS AND DISCUTIONS

Breaking force on the wale direction, Y_1

The regression equation (1) describing the relationship between this characteristic and variables considered for the study is of the form:

$$Y = 344.2128 + 0.3023\,X_1 - 0.5868\,X_2 + 0.4156\,X_3 + 0.375\,X_1X_2 - 0.575\,X_1X_3 - 1.225\,X_2X_3 + 0.7216\,X_1^2 - 1.258\,X_2^2 + 0.863\,X_3 \quad (1)$$

This equation is represented in figures 2–5. The variation of knitted fabrics breaking force on the wale direction depending on dyeing time – X_1, temperature – X_2 and *p*H – X_3 is presented in figure 1.

It is noted that the knitted fabrics breaking force on the wale direction decreases as the time value of 344.4 N increases, followed by a continuous growth of this characteristic. Regarding the variation of knitted fabrics breaking force, depending on dyeing temperature, slow growth of this characteristic that occurs as the temperature increases can be noticed, followed by a decrease. The same can be noticed for breaking force variation of knits depending on *p*H of the dyeing bath. Considering that the desired breaking force value is the maximum, the optimal values of the studied variables are:

- dyeing time +1.682, with real value of 60 minutes;
- temperature –1, with real value of 84°C;
- *p*H +1.682, with real value of 8.

Regression equation confirms the interaction of three variables: X_1X_2 – time-temperature, X_1X_3 – time-*p*H and X_2X_3 – temperature-*p*H.

Figure 3 *a,b* presents the interaction between dyeing time and temperature on the wale direction of the knitted fabrics breaking force, where it can be seen that, in order to obtain the maximum values of breaking force, the dyeing time can be shorter or longer, but the temperature has to be close to the value 0 (90°C).

Figure 4 *a,b* shows the graphic representation of the variation of knitted fabrics breaking force values depending on dyeing time and *p*H, and, as it can be noticed, the good results are performed in the centre of dyeing time and *p*H value.

In figure 5 *a,b* the interaction between dyeing temperature and *p*H of the knitted fabrics breaking force is

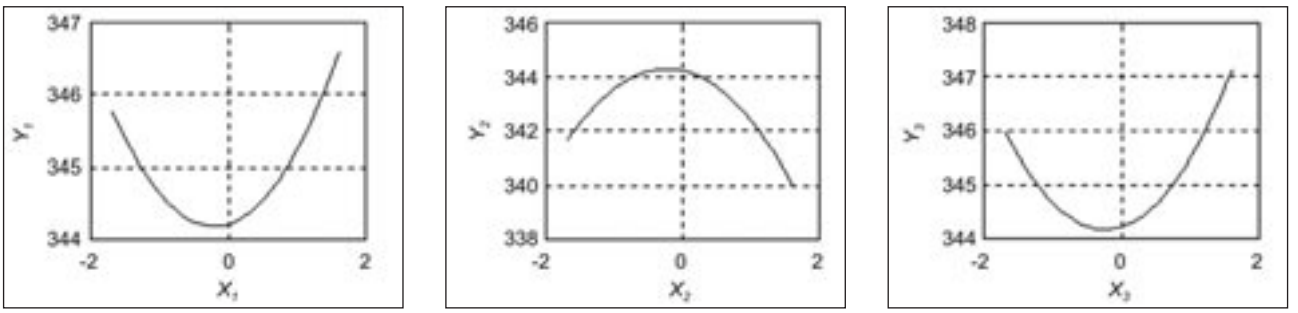
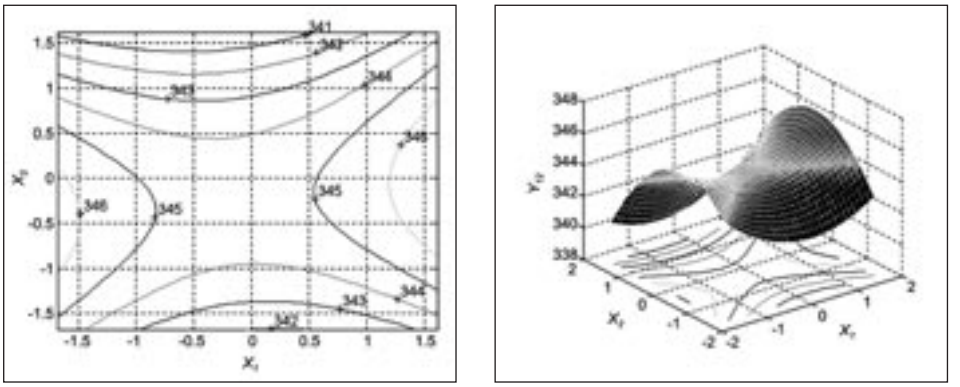
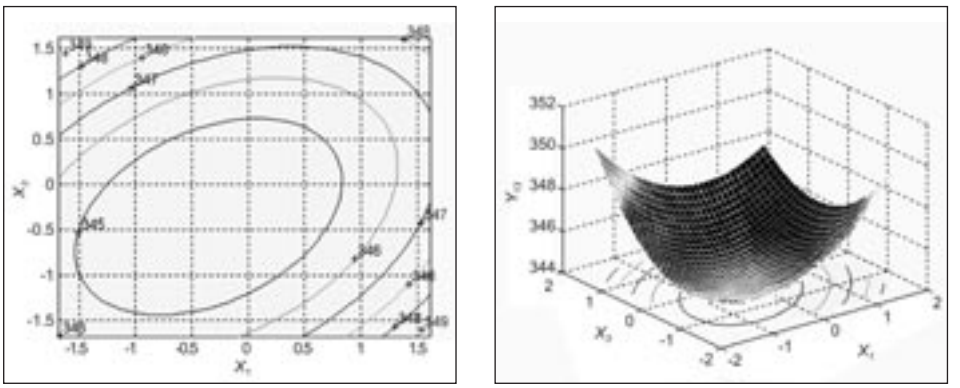


Fig. 2. The variation of knitted fabrics breaking force on the wale direction depending on dyeing time, temperature and *p*H



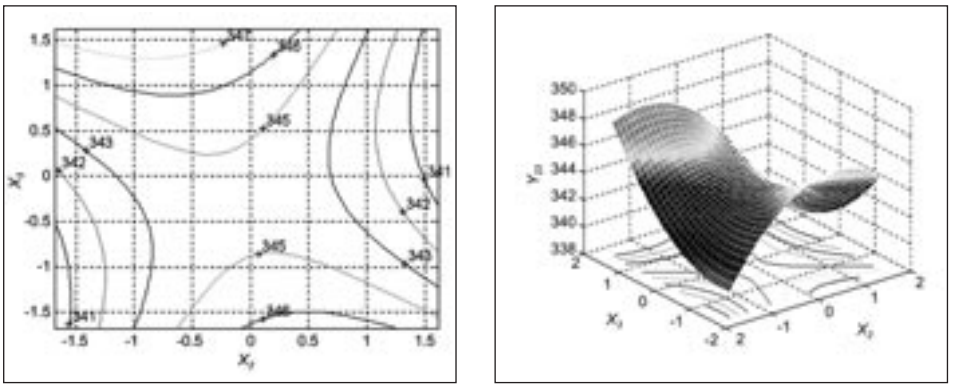
a *b*

Fig. 3. The variation of breaking force depending on dyeing time and temperature:
a – in plane; ***b*** – in space



a *b*

Fig. 4. The variation of breaking force depending on dyeing time and *p*H:
a – in plane; ***b*** – in space



a *b*

Fig. 5. The variation of breaking force:
a – in plane; ***b*** – in space

presented, hence the conclusion that, in order to obtain the maximum values of breaking force, the dyeing temperatures must be close to value 0 (90°C) and the *p*H values must be minimum or maximum.

Breaking force on the row direction, Y_1

The regression equation (2) describing the relationship between this characteristic and the variables considered for the study is of the form:

$$Y = 234.6275 + 0.1425\,X_1 + 1.5674\,X_2 - 0.2536\,X_3 - 0.0125\,X_1X_2 - 1.6875\,X_1X_3 + 1.3125\,X_2X_3 - 4.3852\,X_2^{\frac{1}{2}} - 4.7387\,X_2^{\frac{2}{2}} - 6.6653\,X_2^{\frac{3}{2}} \quad (2)$$

Graphic representations of the influence of the three variables on this characteristic (the knitted fabrics breaking force on the row direction) are given in figures 6–9. Thus, figure 6 shows the variation of knitted fabrics breaking force on the row direction depending on dyeing time, temperature and *p*H dyeing bath.

One can notice that the breaking force increases with the increase of dyeing time up to the value of 234 N, followed by a decrease of this characteristic. This can be observed for the breaking force variation depending on dyeing temperature and *p*H as well, except that in this case after a continuous increase of this characteristic with the dyeing temperature, a slow decrease of the breaking force follows.

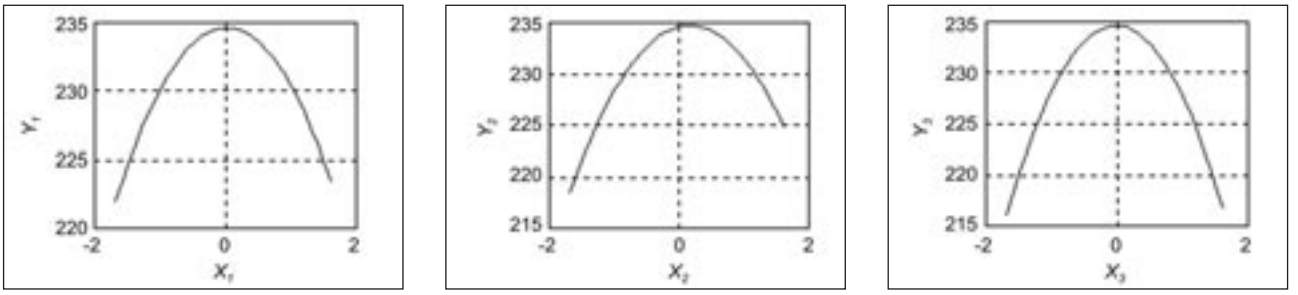
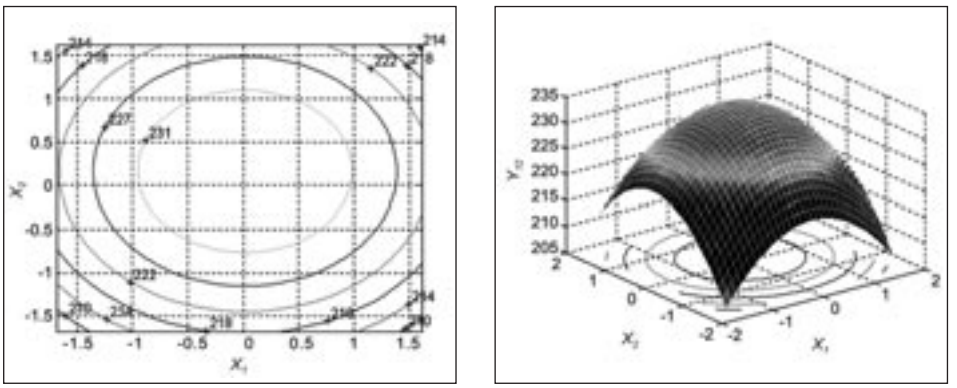
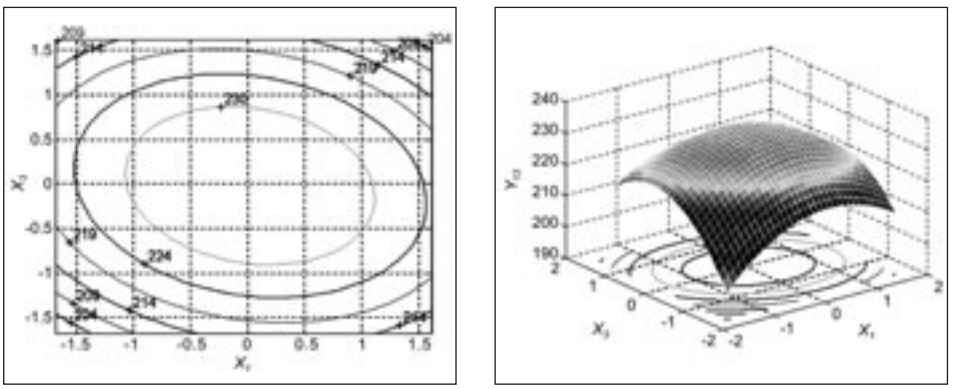


Fig. 6. The variation of knitted fabrics breaking force on the row direction depending on dyeing time, temperature and *p*H



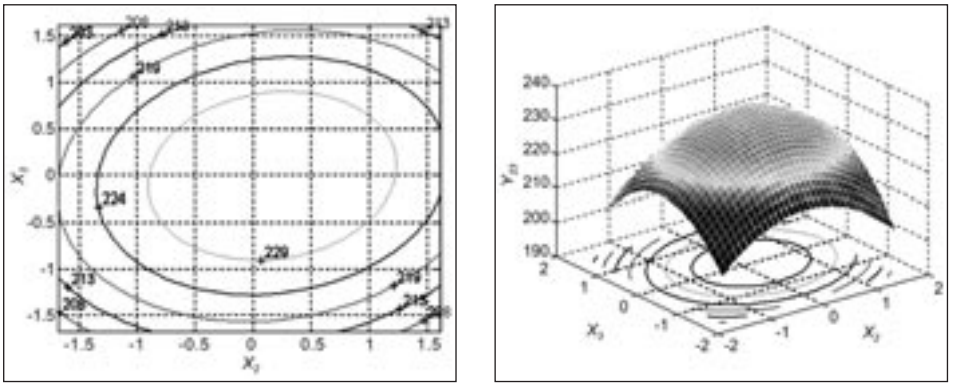
a ***b***

Fig. 7. The variation of breaking force depending on dyeing time and temperature:
a – in plane; ***b*** – in space



a ***b***

Fig. 8. The variation of breaking force depending on dyeing time and *p*H:
a – in plane; ***b*** – in space



a ***b***

Fig. 9. The variation of breaking force depending on dyeing temperature and *p*H:
a – in plane; ***b*** – in space

After the dyeing process intended to have as result maximum breaking force values, which can be seen in figure 6, the following optimal values of the variables considered for the study were registered: dyeing time = 45 minutes (0), temperature = 96°C (1) and *p*H = 7 (0). From the regression equation one can notice the inter-

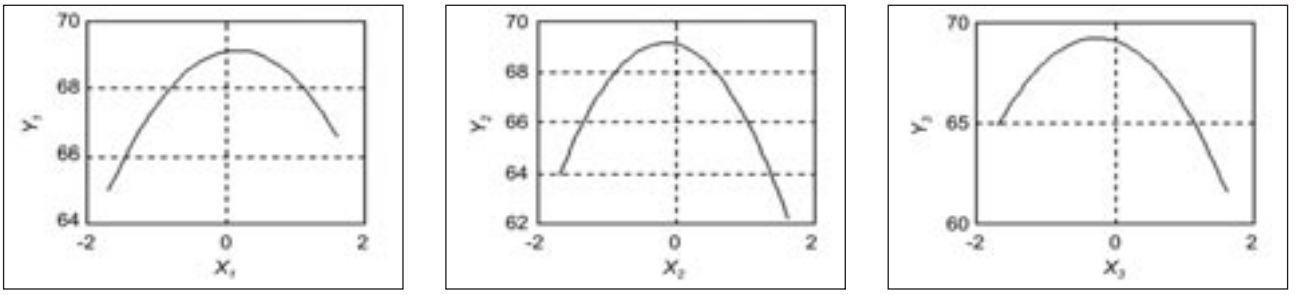
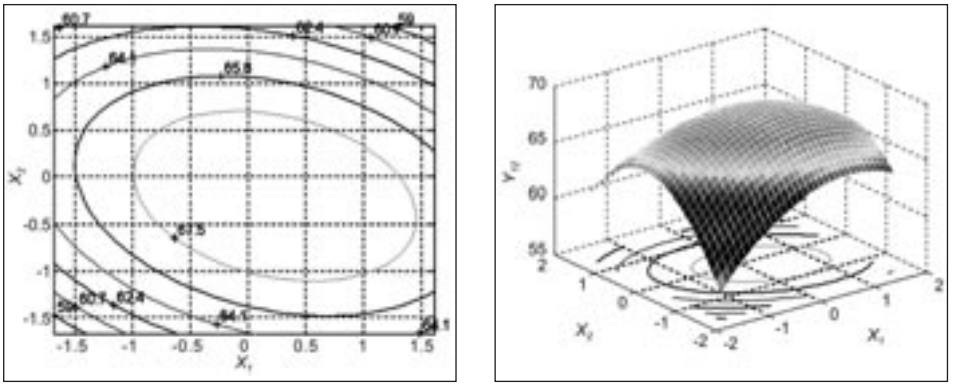
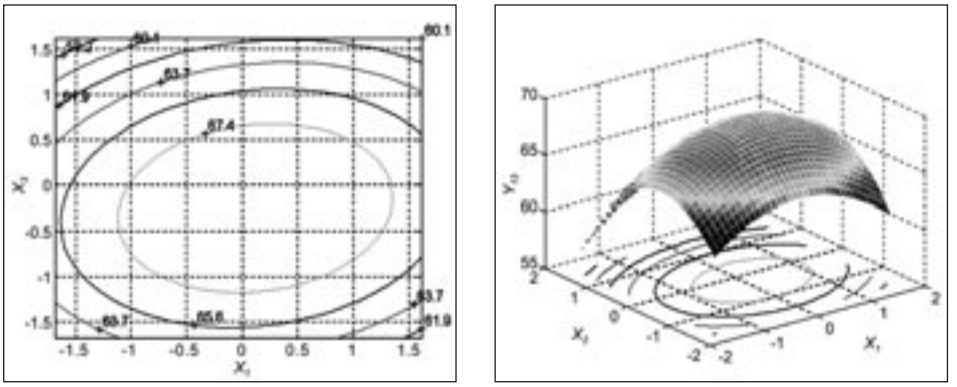


Fig. 10. The variation of knitted fabrics breaking elongation on the wale direction depending on dyeing time, temperature and *p*H



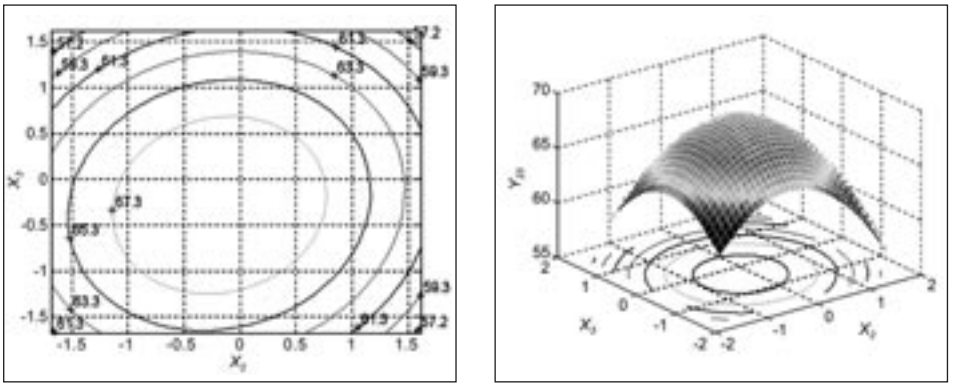
a ***b***

Fig. 11. The variation of breaking elongation depending on dyeing time and temperature:
a – in plane; ***b*** – in space



a ***b***

Fig. 12. The variation of breaking elongation depending on dyeing time and *p*H:
a – in plane; ***b*** – in space



a ***b***

Fig. 13. The variation of breaking elongation depending on dyeing temperature and *p*H:
a – in plane; ***b*** – in space

actions between of three parameters: time-temperature, time-*p*H and temperature-*p*H.

Figures 7–9 show the graphic representation of the variation of breaking force values depending on dyeing time-temperature, time-*p*H and temperature-*p*H, where it is noticeable that the best results can be achieved in

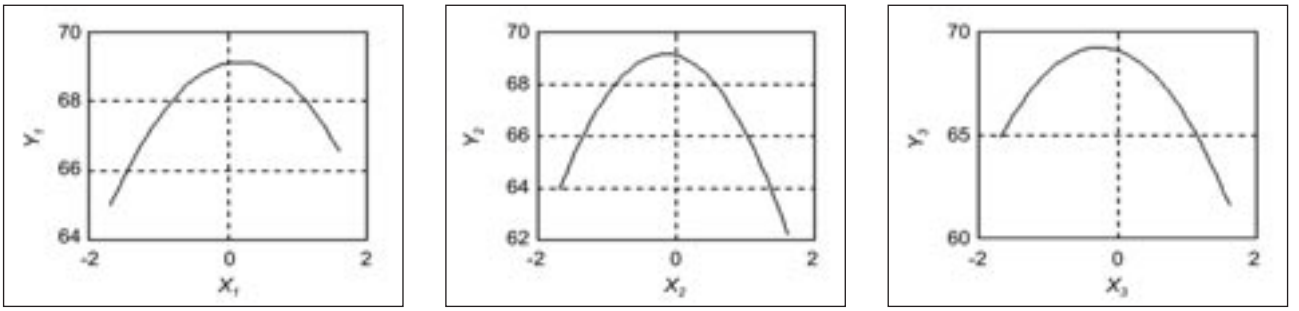
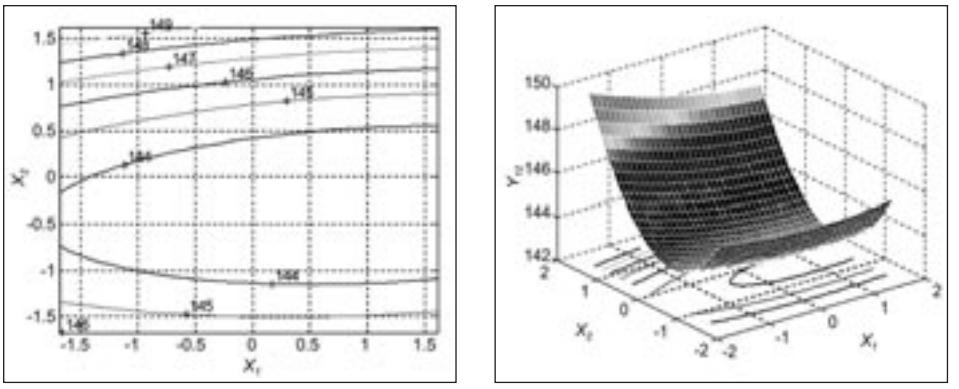
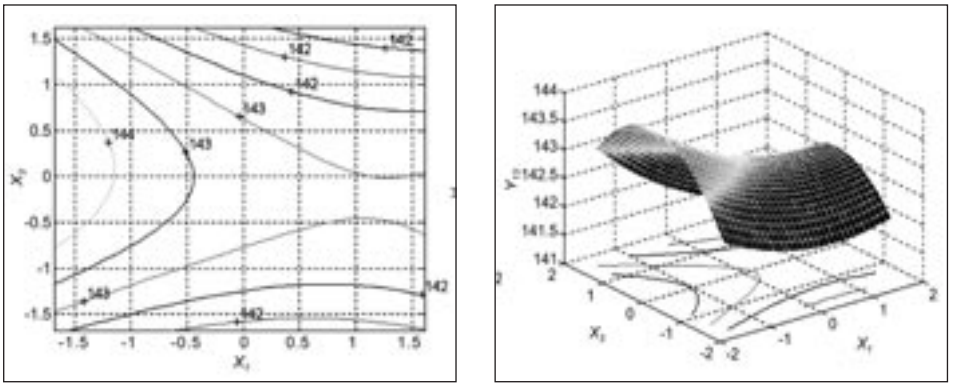


Fig. 14. The variation of knitted fabrics breaking elongation on the row direction depending on dyeing time, temperature and *p*H



a ***b***

Fig. 15. The variation of breaking elongation depending on dyeing time and temperature:
a – in plane; ***b*** – in space



a ***b***

Fig. 16. The variation of breaking elongation depending on dyeing time and *p*H::
a – in plane; ***b*** – in space

the centre of dyeing time, temperature and *p*H values.

Breaking elongation on the wale direction, Y_2

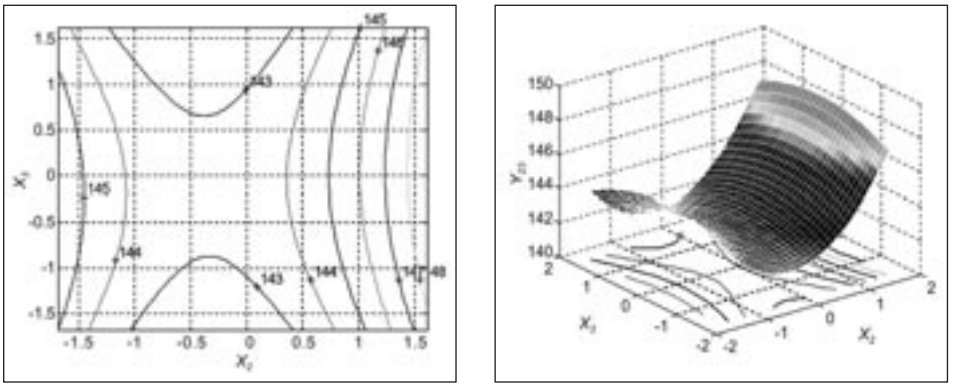
The regression equation (3) that establishes the relationship between this characteristic and the studied variables is expressed as:

$$Y = 69.1018 + 0.3854\ X_1 - 0.701\ X_2 - \\ - 1.1419\ X_3 - 0.875\ X_1X_2 + \\ + 0.4\ X_1X_3 - 1.2178\ X_2^1 - 2.2253\ X_2^2 - \\ - 2.1723\ X_2^3 \quad (3)$$

In figure 10 the variation of knitted fabrics breaking elongation on the wale direction depending on dyeing time, X_1, temperature, X_2 and *p*H, X_3 is presented.

Thus, it can be seen that breaking elongation increases as time increases up to 69.3%, followed by a slow decrease of this characteristic. In variation of breaking elongation, depending on dyeing temperature, it can be noticed that with the increase of this variable the breaking elongation increases also, the maximum value being registered near the centre of experimental field, 90°C. After this increase a continuous decrease of this characteristic follows. A slow increase of breaking elongation occurs with increasing of *p*H dyeing bath, followed by a decrease of this characteristic. To obtain the maximum value of the knitted fabrics breaking elongation, the optimal values of variables considered for this study are: dyeing time = 54 minutes (–1), temperature = 84°C (–1) and *p*H = 6.5 (–1).

The interaction of these three variables X_1X_2, X_1X_3 and X_2X_3 (time – temperature, time – *p*H and temperature – – *p*H) can be observed from the regression equation it. Figures 11–13 are graphic representations of the variation of the breaking elongation values on the wale direction depending on dyeing variables: time – temperature, time – *p*H and temperature – *p*H. Analyzing



a *b*

Fig. 17. The variation of breaking depending on dyeing temperature and *p*H::
a – in plane; ***b*** – in space

these graphics it can be concluded that the best results are achieved in the central areas of the dyeing time, temperature and *p*H values.

Breaking elongation – row direction – *Y2*

The regression equation (4) describing the relationship between this characteristic and the variables considered for this study is of the form:

$$Y = 143.0392 - 0.3142\,X_1 + 1.0473\,X_2 - 0.0594\,X_3 - 0.1625\,X_1X_2 - 0.1125\,X_1X_3 + 0.0875\,X_2X_3 + 0.1229\,X_2^1 + 1.4485\,X_2^2 - 0.4074\,X_2^3 \quad (4)$$

Graphic representations of the influence of the three variables on this characteristic (knitted fabrics breaking elongation on the row direction) are presented in figures 14–17. Figure 14 shows the variation of knitted fabrics breaking elongation on the row direction depending on dyeing time – X_1, temperature – X_2 and *p*H – X_3. The breaking elongation decrease slowly with the increase of the dyeing time up to 142.8%, while dyeing temperature increase leads to a continued increase of breaking elongation, an increase of knitted fabrics breaking elongation depending on *p*H dyeing bath up to 143.1% can be noticed, followed by a continuous decrease of this characteristic.

The result is that the optimal values of studied variables are: dyeing time = 30 minutes (–1.682), temperature = = 100°C (+1.682) and *p*H = 7 (0). The regression equation presented in figures 15–17 confirms the interactions of the three variables X_1X_2, X_1X_3 and X_2X_3.

The variation of knitted fabrics breaking elongation on the row direction depending on the interactions of two variables is drawn in figures 15 and 17. From the interaction between dyeing time and temperature presented in figure 15 *a, b,* it can be noticed that, in order to obtain the maximum values of knitted fabrics breaking elongation on the row direction, the dyeing process can be performed at lower or higher temperatures, but in a short dyeing time.

Figures 16 and 17 present the graphics for the variation of breaking elongation depending on dyeing temperature and *p*H and the variation of this characteristic depending on dyeing temperature and *p*H. It can be noticed that, in order to obtain maximum values of knitted fabrics breaking elongation on the row direction, the dyeing process should be performed at *p*H values as close to the centre of experimental field, at high temperatures and at small dyeing times.

CONCLUSIONS

The variation of breaking force and elongation for plain-weft-knits made of 100% cotton yarns with plain structure subjected to dyeing operation were analyzed, changing the following parameters: time, temperature and *p*H dyeing bath.

The optimum values of these parameters were specified, so that the cotton integrity support might be protected during the dyeing treatments.

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Efectele coloranților azoici asupra sănătății

DACIANA ILICA LEUCEA

REZUMAT – ABSTRACT – INHALTSANGABE

Efectele coloranților azoici asupra sănătății

Lucrarea prezintă rezultatele unor cercetări privind influența coloranților și pigmenților azoici, utilizați în vopsirea și imprimarea textilelor, asupra sănătății și, în special, asupra posibilității declanșării unor reacții alergice sau a cancerelor. Modul de formare a aminelor cancerigene este considerat a fi factorul major care determină carcinogenitatea unui anumit colorant. În majoritatea cazurilor, alergiile au fost cauzate de folosirea coloranților de dispersie pe materiale sintetice, destinate producerii de articole textile sau jucării, care prezentau o rezistență scăzută a culorii. Gradul de extracție a coloranților din materialele textile variază în funcție de numărul ciclurilor de purtare/spălare, de nivelul pH-ului, de aciditatea/alcalinitate transpirației etc. În producerea coloranților, o alternativă la aminele aromatice o constituie folosirea aminelor aromatice sulfonate, la care efectul genotoxic lipsește sau, în general, este foarte scăzut.

Cuvinte-cheie: coloranți azoici, pigmenți azoici, amină aromatică, efect alergenic, potențial cancerigen

The effects of azo dyes on human health

This paper presents the results of certain researches regarding the influence of azo dyes and pigments used in the dyeing and printing of textiles, on health and, in particular, on the possibility of triggering certain allergic reactions or cancer. The development of carcinogenic amines is believed to be the major factor determining the carcinogenic potential of a given dye. In most cases, allergies have been caused by the utilization of dispersion dyes on synthetic materials, meant for the manufacturing of textile items or toys which exhibited a low colour resistance. The dye extraction degree from textile materials varies depending on the number of washing/wear cycles, the pH level, the perspiration acidity/alkalinity, etc. In the manufacture of dyes, an alternative to aromatic amines is the use of aromatic sulphonate amines, which do not have a genotoxic effect, or, if they do it is, in general, very low.

Key-words: azo dyes, azo pigments, aromatic amine, allergenic effect, carcinogenic potential

Gesundheitseffekte der Azo-Farbstoffe

Die Arbeit stellt vor Forschungergebnisse betreff der Gesundheitswirkung der Azo-Farbstoffe und Pigmente beim Färben und Drucken der Textilien und insbesondere die Möglichkeit der Erscheinung alergischer Reaktionen oder Krebskranheiten. Die Bildungsweise der krebserregenden Amine wird als Hauptfaktor der Krebsverursachung für einen bestimmten Farbstoff betrachtet. In meisten Fällen wurden die Allergien von der Anwendung der Dispersionsfarbstoffen auf synthetische Materialien verursacht, welche der Produktion von Textilartikel oder Spielzeuge bestimmt sind, die einen schwachen Farbwiderstand aufwiesen. Der Extraktionsgrad der Farbstoffe aus Textilmaterialien variiert in Abhängigkeit der Trag-/Waschzyklen, des pH-Wertes, des Sauren/Alkalinen Charakters des Schweisses etc. In der Farbstoffproduktion wird eine Alternative zu den aromatischen Aminen durch die Anwendung der sulfonierten aromatischen Aminen gebildet, bei denen der genentoxische Effekt fehlt oder generell sehr gering ist.

Schlüsselwörter: Azo-Farbstoffe, Azo-Pigmente, aromatische Amine, Allergieeffekt, Krebserregendes Potential

Având în vedere ponderea mare a utilizării coloranților și pigmenților azoici în vopsirea și imprimarea textilelor, în ultimii ani s-a pus un accent deosebit pe studierea potențialului cancerigen al acestor coloranți.

Coloranții azoici reprezintă cea mai numeroasă clasă de coloranți organici folosiți în finisarea produselor obținute din fibre naturale, regenerate și sintetice. Având în vedere un potențial efect cancerigen al anumitor coloranți din această grupă, se impune o analiză a proprietăților acestora și a influenței lor asupra sănătății oamenilor și a mediului înconjurător.

Coloranții azoici reprezintă o grupă de compuși chimici care conțin unul sau mai multe grupări azoice, având o legătură dublă între doi atomi de azot: –N=N–. Coloranții azoici pot fi împărțiți în coloranți azoici solubili în mediul de aplicare și pigmenți azoici insolubili în mediul de aplicare. De asemenea, coloranții azoici pot fi solubili în apă (hidrofilici) sau în grăsimi.

Proprietatea caracteristică a pigmenților este solubilitatea extrem de scăzută în solvenți organici, ceea ce face ca, în timpul procesului de vopsire, aceștia să rămână aproape în totalitate în formă solidă [1].

Coloranții azoici sunt obținuți prin așa-numitul proces de „diazotizare“. În prima fază, amina aromatică este transformată, la temperatură scăzută și în prezența nitritului de sodiu sau a acidului clorhidric, într-un compus de diazoniu. Pentru a forma colorantul [2], acest compus reacționează apoi cu un component de cuplare, care poate fi un fenol, un naftol sau o amină (1):

(1)

Datorită posibilității alegerii componentelor diazo și a celor de cuplare, gama de coloranți azoici realizați este foarte largă. Numărul de combinații este în creștere, deoarece un colorant poate conține unul sau mai mulți compuși azoici.

REZULTATELE CERCETĂRII

Au fost efectuate cercetări detaliate privind influența coloranților azoici asupra sănătății și, în special, asupra posibilității declanșării unor reacții alergice sau a cancerelor. Mai puțin a fost abordată problema producerii de malformații și iritații. În lucrarea „Coloranții azoici în textile și jucării“ este prezentat un studiu, efectuat de către cercetătorii de la Institutul Danez de Tehnologie, în perioada 1990–1996, referitor la cazurile de alergii provocate de coloranții azoici (tabelul 1).

Trebuie menționat faptul că, în majoritatea cazurilor, alergiile au fost cauzate de folosirea coloranților de dispersie pe materiale sintetice, care prezentau o rezistență scăzută a culorii. În toate situațiile, alergiile au

Tabelul 1

CAZURI DE ALERGII PROVOCATE DE COLORANȚII AZOICI		
Denumirea colorantului	**Număr de cazuri**	**Referințe**
Black Acid 48 C165005 CAS 1328-24-1	3	Balato, N. ș.a., 1990
Acid Red 118	1	Seidenari, S. ș.a., 1995
Acid Red 359	2	Seidenari, S. ș.a., 1995
Acid Yellow 61	5	Seidenari, S. ș.a., 1995
Basic Brown 1 CI 21010 CAS 8005-78-5	2	Balato, N. ș.a., 1990
Bismark Brown	1	Lisboa, C. ș.a., 1994
Black Acid 48	4	Seidenari, S. ș.a., 1991
Black Base 1	9	Seidenari, S. ș.a., 1991
Direct Orange 34	8	Seidenari, S. ș.a., 1995
Disperse Black 1 CI 11365	17	Seidenari, S. ș.a., 1991; Lisboa, C. ș.a., 1994 Shehade, S.A. and Beck, M.H., 1990; Sousa-Basto, A. and Azenha, A., 1994
Disperse Black 2 CI 11255 CAS 6232-57-1	5	Lisboa, C. ș.a., 1994; Sousa-Basto, A. and Azenha, A., 1994
Disperse Blue 1 CI 64500 CAS 2475-45-8	3	Hausen, B.M., 1993
Disperse Blue 106	15	Lisboa, C. ș.a., 1994; Hausen, B.M. ș.a., 1991, 1993; Nakagawa, M. ș.a., 1996
Disperse Blue 124	59	Balato, N. ș.a., 1990; Seidenari, S. ș.a., 1991; Hausen, B. M. ș.a., 1993; Nakagawa, M. ș.a., 1996
Disperse Blue 3 C 161505 CAS 2475-46-9	4	Seidenari, S. ș.a., 1991
Disperse Blue 35 CAS 12222-75-2	9	Balato, N. ș.a., 1990; Seidenari, S. ș.a., 1991; Lisboa, C. ș.a., 1994
Disperse Blue 85	1	Brown, R., 1990
Disperse Brown 1	2	Brown, R., 1990; Nakagawa, M. ș.a., 1996
Disperse Orange 1 CI 11080 CAS 2581-69-3	1	Shehada, S.A. and Beck, M.H., 1990
Disperse Orange 13	2	Brown, R., 1990; Nakagawa, M. ș.a., 1996
Disperse Orange 3 CI 11005 CAS 730-40-5	39	Balato, N. ș.a., 1990; Seidenari, S. ș.a., 1991; Lisboa, C. ș.a., 1994; Shehade, S.A. and Beck, M.H., 1990; Sousa-Basto, A. and Azenha, A., 1994; Nakagawa, M. ș.a., 1996
Disperse Orange 76	14	Balato, N. ș.a., 1990; Seidenari, S. ș.a., 1991
Disperse Red 1 CI 11110 CAS 2872-52-8	46	Brown, R., 1990; Balato, N. ș.a., 1990; Seidenari, S. ș.a., 1991; Lisboa, C. ș.a., 1994; Hausen, B.M. ș.a., 1993; Shehade, S.A. and Beck, M.H., 1990; Sousa-Basto, A. and Azenha, A., 1994; Nakagawa, M. ș.a., 1996
Disperse Red 17	28	Balato, N. ș.a., 1990; Seidenari, S. ș.a., 1991; Lisboa, C. ș.a., 1994; Shehada, S.A. and Beck, M.H., 1990; Sousa-Basto, A. and Azenha, A., 1994; Nakagawa, M. ș.a., 1996

Tabelul 1 (continuare)

Denumirea colorantului	**Număr de cazuri**	**Referințe**
Disperse Red 7	1	Sousa-Basto, A. and Azenha, A., 1994
Disperse Yellow 3 CI 11855 CAS 2832-40-8	41	Balato, N. ș.a., 1990; Seidenari, S. ș.a., 1991; Lisboa, C. ș.a., 1994; Hausen, B.M. ș.a., 1993; Shehade, S.A. and Beck, M.H., 1990; Sousa-Basto, A. and Azenha, A., 1994
Disperse Yellow 54	3	Seidenari, S. ș.a., 1991
Disperse Yellow 9 CI 10375 CAS 6373-73-5	12	Brown, R., 1990; Seidenari, S. ș.a., 1991
p-aminoazobenzen (CI Solvent Yellow 1) CI 11000	28	Seidenari, S. ș.a., 1991; Shehade, S.A. and Beck, M.H., 1990
p-aminofenol	9	Seidenari, S. ș.a., 1991
p-dimetilaminoazobenzen	19	Balato, N. ș.a., 1990; Seidenari, S. ș.a., 1991

apărut numai din cauza folosirii coloranților, nu și a pigmenților. Ele nu au apărut la muncitorii implicați în procesul de producție, ci doar la consumatori. Cazurile analizate s-au referit la pantaloni, tricouri și bodyuri, jambiere, căptușeala perucilor, lenjeria de corp, lenjeria de pat, curele pentru ceasuri de mână.

Așa cum reiese din tabelul 1, în cele mai multe cazuri, alergiile au fost provocate de Disperse Black 1, Disperse Blue 106, Disperse Blue 124, Disperse Orange 3, Disperse Orange 76, Disperse Red 1, Disperse Red 17, Disperse Yellow 3, Disperse Yellow 9, p-aminoazobenzen și p-dimetilaminoazobenzen.

Totodată, există posibilitatea ca o persoană să fie alergică la unul sau mai mulți coloranți din aceeași categorie. De exemplu, persoanele care sunt alergice la Disperse Blue 124 pot fi alergice și la Disperse Blue 106, pentru că ambii aparțin grupei azo-azoil-fenilendiamine. Dintre grupele în interiorul cărora pot avea loc reacții încrucișate [4] sunt:

- grupa de aminoazobenzen – Disperse Red 1, Disperse Red 17, Disperse Brown 1 etc.;
- grupa de parafenilendiamine – parafenilendiamină și Disperse Orange 3;
- grupa de benzotiazol-azoil-parafenilendiamine – Disperse Red 153.

Caracterul carcinogen al coloranților azoici poate fi privit sub două aspecte:

- carcinogenitatea colorantului ca atare;
- carcinogenitatea aminelor aromatice, care pot produce degradarea produsului prin scindarea reductivă a grupării azoice.

Deoarece nu toți coloranții azoici au fost testați suficient, nu se poate afirma cu exactitate dacă aceștia ar trebui sau nu considerați cancerigeni. În tabelul 2 sunt prezentați coloranții care ar trebui considerați cancerigeni, iar în tabelul 3 sunt prezentați acei coloranți azoici care, probabil, nu sunt cancerigeni pentru oameni. Conform clasificării realizate de IARC – International Agency for Research on Cancer, World Health Organization, aceștia sunt incluși în grupa 4, folosită pentru agenți și mixturi, prin urmare nu există evidențe clare privind riscul cancerigen la oameni și nici rezultate

Tabelul 2

COLORANȚI AZOICI CONSIDERAȚI CANCERIGENI PENTRU OAMENI			
Denumirea colorantului	**Gradul de evidență**	**Observații**	**Referințe**
(4-dimetilamino)-azobenzen-1-naftalen	2B	Trei studii pe șoareci masculi au demonstrat apariția de tumori pe ficat și stomac	Longstaff, 1983
(4-dimetilamino)-azobenzen-1-naftalen	2A	Administrat pe cale orală, produce tumori pe ficat la șoareci	Longstaff, 1983
Acid Dye CI 16155 (diazo-component: 2,4,5 trimetilanilina)	2B	S-au raportat tumori pe ficat la șoareci	Longstaff, 1983
Acid Red 114 CI 23635	2B	Este legat, probabil de formarea metabolitului 3,3-dimetilbenzidina (apare în tabelul 4)	IARC, 1993
Acid Red 26	2B	Este cancerigen la șoareci; dacă este administrat pe cale orală, provoacă tumori pe ficat	Longstaff, 1983, Burg and Charest, 1980
Direct Black 38	2A	Conținut mare de benzidină	IARC, 1982
Direct Blue 15	2B	Probabil este legat de formarea metabolitului 3,3-dimetilbenzidina (apare în tabelul 4)	IARC, 1993
Direct Blue 6	2A	Conținut mare de benzidină	IARC, 1982
Direct Brown 95	2A	Conținut mare de benzidină	IARC, 1982
Solvent Yellow 1	2A	Produce tumori pe piele, la șoareci	Longstaff, 1983
Solvent Yellow 2	2A	Produce tumori pe piele, la șoareci	Longstaff, 1983
Solvent Yellow 3	1	Produce tumori pe ficat, prin aplicarea pe piele la șoareci, iar prin aplicări topice la șoarecii gestanți produce tumori generației F_1	Longstaff, 1983

Tabelul 3

COLORANȚI AZOICI NECANCERIGENI PENTRU OAMENI		
Denumirea colorantului	**Gradul de evidență**	**Referințe**
Acid Orange 10	Rezultate negative la dihori până la 12 luni, iar la șoareci pe toată durata vieții	Burg și Charest, 1980
Acid Orange 20	Rezultate negative la șoareci pe toată durata vieții, iar la șobolani pentru 2 ani	Burg și Charest, 1980
Acid Red 14	Rezultate negative la șoareci pe toată durata vieții, iar la șobolani pentru 12 luni	Burg și Charest, 1980
Acid Red 27	Rezultate negative la șoareci pe toată durata vieții, iar la șobolani pentru 64–78 de săptămâni	Burg și Charest, 1980
Pigment Yellow 12 (pur)	Nu s-au semnalat tumori, la testările efectuate pe un grup de 100 de șoareci, timp de 2 ani	Longstaff, 1983, Burg și Charest, 1980
Pigment Yellow 16 (pur)	Nu s-au semnalat tumori, la testările efectuate pe un grup de 100 de șoareci, timp de 2 ani	Longstaff, 1983
Pigment Yellow 83 (pur)	Nu s-au semnalat tumori, la testările efectuate pe un grup de 100 de șoareci, timp de 2 ani	Longstaff, 1983
Solvent Orange 7	Rezultate negative, subcutanat și oral, la șobolani pentru 65 de săptămâni, iar la șoareci pentru 52 de săptămâni	Burg și Charest, 1980
Solvent Yellow 5	Nu au provocat tumori la șoareci, șobolani, hamsteri și câini	Longstaff, 1983, Burg și Charest, 1980
Solvent Yellow 6	Rezultate negative la șobolani – pentru perioade de 65 de săptămâni–2 ani, și la câini – pentru 1 an	Burg și Charest, 1980

pozitive privind efectul cancerigen în cazul experimentelor efectuate pe animale. Dacă un colorant nu este specificat în cadrul tabelelor 3 sau 4, înseamnă că nu au fost găsite suficiente date care să permită includerea lui în grupa de agenți cancerigeni sau necancerigeni. Desigur, acest lucru scoate în evidență faptul că, la un mare număr de coloranți azoici, încă nu s-a stabilit cu certitudine efectul cancerigen.

Datele din tabelele 2 și 3 au fost selectate din diferite reviste științifice, de către cercetătorii de la DTI, și prezentate în lucrarea „Coloranții azoici în textile și jucării". Grupele de agenți cancerigeni stabilite de IARC sunt:

- *grupa 1* – agentul este cancerigen la oameni. Modalitatea de expunere determină efecte carcinogene la oameni.
- *grupa 2A* – agentul este probabil cancerigen la oameni. Modalitatea de expunere determină efecte care, probabil, sunt carcinogene la oameni.
- *grupa 2B* – agentul este posibil cancerigen la oameni. Modalitatea de expunere determină posibile efecte carcinogene la oameni.

Coloranții azoici pot forma amine aromatice prin scindarea reductivă a uneia sau a mai multor grupări azoice. Cel mai adesea, produsele de degradare a aminelor

AMINE AROMATICE CONSIDERATE CARCINOGENE		
Denumirea aminei	**CAS**	**Gradul de evidenţă a carcinogenităţii/genotoxicităţii**
2-Naftilamina	91-59-8	grupa 1 IARC/genotoxic
3,3'-Diclorobenzidina 91-94-1 grupa 2B IARC/genotoxic		
3,3'-Dimetoxibenzidina	119-90-4	grupa 2B IARC/genotoxic
3,3'-Dimetilbenzidina	119-93-7	grupa 2B IARC
4,4'-Metilendis(2-cloroanilina) (MOCA)	101-14-4	grupa 2A IARC /deosebit de genotoxic
4,4'-Metilendianilina	101-77-9	grupa 2B IARC /genotoxic
4,4'-Metilendi-o-toluidina	838-88-0	grupa 2B IARC
4,4'-Oxidianilina	101-80-4	grupa 2B IARC
4,4'-Tiodianilina	139-65-1	grupa 2B IARC
4-Aminobifenil	92-67-1	grupa 1 IARC/genotoxic
4-Cloro-o-toluidina	95-69-2	grupa 2A IARC/genotoxic
4-Metoxi-m-fenilendiamina	615-05-4	grupa 2B IARC
4-Metil-m-fenilendiamina (2,4-diaminotoluen)	95-80-7	grupa 2B IARC
6-Metoxi-m-toluidina (5-metil-o-anisidina)	120-71-8	grupa 2B IARC
Benzidina	92-87-5	grupa 1 IARC/genotoxic
o-Aminoazotoluen	97-56-3	grupa 2B IARC
o-Anisidina	90-04-0	grupa 2B IARC
o-Toluidin	95-53-4	grupa 2B IARC /genotoxic
p-Aminoazobenzen (p-(fenilazo)-anilina)	60-09-3	grupa 2B IARC
p-Cloroanilina	1106-47-8	grupa 2B IARC /genotoxic

aromatice sunt similare cu cele din care a fost obținut colorantul azoic, însă există și unele excepții. Dacă, în procesul de producție, o amină aromatică este utilizată în calitate de component diazo, produsul de descompunere va fi aceeași amină aromatică, ceea ce nu se întâmplă în cazul folosirii ei în calitate de component de cuplare.

Modul de formare a aminelor cancerigene este considerat a fi factorul major care determină carcinogenitatea unui anumit colorant. Un exemplu tipic sunt coloranții pe bază de benzidină, care sunt metabolizați de către așa-numita benzidină carcinogenă umană, clasificați de LKRC în grupa 1. Coloranții pe bază de benzidină au fost plasați de IARC în grupa 2A (probabil cancerigeni la oameni). Aminele aromatice care pot fi considerate cancerigene sunt redate în tabelul 4. Lista nu este completă, atâta vreme cât există o multitudine de amine aromatice care se pot forma în timpul descompunerii coloranților azoici și nu toate aminele aromatice au fost testate suficient pentru a permite o evaluare a efectului lor cancerigen.

Toate aminele aromatice din tabelul 4 se regăsesc și în lista stabilită de Germania, cu excepția o-anisidinei și p-aminoazobenzenului. 5-nitro-toluidina, CAS 99-55-8, și 2,4,5-trimetilanilina, CAS 137-17-7 se află, de asemenea, pe lista de amine interzise în Germania, dar au fost clasificate de IARC doar în grupa 3.

Alternative de substituire a aminelor interzise

În producerea coloranților, o alternativă la aminele aromatice o constituie folosirea aminelor de aromatice sulfonate. Jung, R., Steinle, D. & Anliker, R. [5] au realizat un studiu comparativ între aminele aromatice sulfonate și cele nesulfonate, din punct de vedere al genotoxicității și carcinogenității. S-a ajuns la concluzia că, la aminele aromatice sulfonate, efectul genotoxic lipsește sau, în general, este foarte scăzut.

Modul de expunere la coloranții azoici

În cazul produselor finite, expunerea la oricare dintre coloranții azoici sau la produsul lor de degradare, cum ar fi aminele aromatice, poate avea loc prin ingerare (de exemplu, la copiii care își bagă jucăriile în gură) sau prin contactul direct cu pielea (prin frecare sau extragerea prin transpirație).

Acidul gastric

La examinarea conținutului de amine aromatice din mostrele textile, este important să se creeze condiții de expunere a acestora cât mai apropiate de cele reale. Se știe că, în general, în intestine există un mediu acid, iar unul similar se poate obține cu ajutorul unei soluții de acid clorhidric cu un *p*H de 1,5.

Transpirația

Transpirația este secretată de două glande sudoripare: cele ecrine (minore) – care se găsesc în zonele fără păr și, în principal, în palmă, și cele apocrine (majore) – care se află în zonele cu păr și, în special, în zona axilară. Transpirația ecrină este clară, apoasă și fără miros, în timp ce cea apocrină, din cauza acțiunii bacteriilor, devine repede tulbure, vâscoasă, uneori gălbuie și fluorescentă, alteori albăstruie sau aproape neagră și urât mirositoare.

*p*H-ul transpirației

În cazul unui flux redus, transpirația ecrină este acidă, din cauza conținutului mare de secreție de acid lactic. Prin creșterea fluxului, acesta se transformă în alcalină, din cauza secreției de bicarbonat. *p*H-ul, în cazul unui nivel scăzut al fluxului de transpirație, se situează între 5 și 7, în timp ce, la un nivel ridicat, acesta este 7–8.

Transpirația apocrină, din cauza conținutului ridicat de amoniac, este mai puțin acidă decât cea ecrină. Transpirația copiilor este, în general, mai puțin acidă decât cea a adulților și are un *p*H de 6-8 [6].

Nivelul de amine aromatice din mostrele analizate

În cadrul cercetărilor realizate de DIT, unele mostre au fost supuse unor tratamente de simulare a unui mediu gastric acid, altele de simulare a unei transpirații acide/ alcaline. Îmbrăcămintea purtată în cazul simulării unei transpirații alcaline abundente, pentru un timp îndelungat, a prezentat cel mai mare număr de constatări pozitive, indicând cea mai mare expunere la amine aromatice. S-a găsit anilină în 13 din cele 59 de mostre, cu niveluri cuprinse între 0,4 și 160 mg/kg de material. Cel mai ridicat conținut de anilină s-a constatat la o pânză din bumbac, de culoare roz.

S-a constatat că, în 17 din cele 59 de mostre, au fost prezente aminele aromatice, la care s-a făcut referire în tabelul 4. Cel mai scăzut nivel a fost de 0,1 mg o-toluidină la un kg de material textil, iar cel mai ridicat nivel, de 70 mg o-toluidină, a fost înregistrat la un pulover tricotat, cu guler înalt, care se îmbracă peste cap, de culoare oliv închis. La același pulover, s-a constatat și un conținut ridicat de alte amine, prezentate în tabelul 4, precum și de anilină și p-fenilendiamină, care sunt alergenice. De asemenea, s-a găsit benzidină sau izomeri ai acesteia, la nivelul de 300 mg/kg, în două mostre – o pijama de mătase, de culoare burgundz, și o pereche de pantaloni scurți, din bumbac, de culoare maro.

Cercetătorii suedezi au testat 11 articole de îmbrăcăminte pentru copii și au găsit un conținut foarte mare de coloranți azoici în două din cele 11 mostre. Raportul nu specifică identitatea și cantitatea coloranților azoici, dar indică faptul că au fost găsite anumite amine aromatice, care se pot forma din unii coloranți azoici.

În plus, a mai fost găsită o cantitate de 27 mg de crom/kg, peste 0,22 mg plumb/kg și 37 mg de cupru/kg într-un pantalon de nou-născut. S-a constatat, de asemenea, un *p*H ridicat, de peste 8, în 5 din cele 11 mostre. Cel mai mare *p*H a fost de 9,4 [7]. La aceste valori mari ale *p*H-ului, pentru ca aminele aromatice să fie extrase nu mai este necesar ca transpirația să fie alcalină. Expunerea poate surveni doar prin stropirea materialelor cu apă sau cu mâncare.

Durata expunerii

În mod normal, textilele pentru îmbrăcăminte, în cazul în care ele nu au fost spălate de consumator înaintea purtării, prezintă un grad scăzut de expunere la prima purtare.

Încălțămintea este supusă unei expuneri constante, atâta vreme cât aceasta se spală foarte rar. Deoarece picioarele pot transpira foarte mult și pot furniza un mediu alcalin, există mari șanse de extragere a aminelor aromatice din oricare dintre coloranții azoici utilizați în materialul din care este realizată încălțămintea.

În 1997, Asociația Toxicologică și Ecologică a Producătorilor de Coloranți și Pigmenți Organici – ETAD, a realizat un studiu asupra gradului de extracție a coloranților din materialele textile, în cazul unei durate de utilizare mai mari decât cea normală. Această durată a fost stabilită la 50 de cicluri de purtare-spălare pentru fiecare bucată de material textil. În cadrul experimentului, pentru vopsirea mostrelor din materiale poliamidice, au fost aleși trei coloranți de dispersie – Disperse Yellow 3, Disperse Blue 3 și Acid Red 114. Acesta a fost cel mai neinspirat studiu de caz, atâta vreme cât vopsirea în profunzime a materialelor poliamidice cu acest tip de coloranți determină o rezistență scăzută. Studiul a arătat că migrarea măsurată a fost mult mai mică decât cea prevăzută. Expunerea externă preconizată, în g/kg/zi, este reprezentată astfel (1):

$$\frac{1\,m^2(\text{material textil})}{70\ kg\ (\text{greutatea persoanei})} \cdot D \cdot 50 \cdot 0,001 \cdot 10^6\,g/kg/zi \quad (1)$$

unde:

D	reprezintă	greutatea materialelor vopsite;
50	–	numărul de purtări;
0,001	–	valoarea de migrare implicită (0,1%/zi).

Studiile au arătat că, la o vopsire de referință, rezultă o rezistență de aproximativ 4, la un nivel mediu de expunere de 1 g/ kg corp/ zi. Nivelul mediu prevăzut al expunerii, calculat pe baza valorii migrării de 0,1% a colorantului, poate fi la fel de mare ca și 500 g/ kg corp/ zi. Valoarea măsurată de migrare a colorantului se micșorează la un număr de purtări mai mare decât cel normal.

Cauzele discrepanțelor mari dintre valoarea măsurată și cea preconizată ale expunerii ar putea fi alcalinitatea agenților de spălare, aminele aromatice libere și surplusul de coloranți, care sunt extrași foarte rapid.

CONCLUZII

Reglementările din Danemarca conțin o listă de coloranți azoici, care pot descompune aminele aromatice interzise în Germania și Olanda. O listă similară nu este inclusă, însă, și în reglementările din Germania, în care nu se specifică coloranții sau pigmenții interziși, ci doar aminele aromatice interzise. Motivul îl constituie faptul că se utilizează o mulțime de coloranți și nu ar fi posibil și nici justificabil să se demonstreze suspiciunile, pentru fiecare caz, prin experimente pe animale [3].

În concordanță cu data de baze AQIURE din 1993, aproximativ 100 de coloranți azoici, interziși prin reglementările germane, sunt prezenți încă pe piață. În Europa de Est mai există încă producători de coloranți azoici, posibil nocivi pentru sănătatea umană, și nu poate exista un control al coloranților produși în aceste țări, deoarece nu există statistici referitoare la aceste produse. Aceasta înseamnă că există riscul folosirii unor coloranți azoici în componența cărora există una sau mai multe amine aromatice interzise de Uniunea Europeană.

Motivele pentru care coloranții azoici dețin marea majoritate în domeniul coloranților utilizați în industria textilă sunt numărul vast de nuanțe și aplicații disponibile, precum și proprietățile de rezistență conferite. Din cauza numărului mare de coloranți azoici utilizați, nu este posibilă realizarea unei liste exacte cu cei interziși. În plus, de multe ori, este dificilă aflarea de la producători

a informației referitoare la componența chimică a produselor lor, deoarece ei preferă ca aceste date să rămână confidențiale. Utilizarea coloranților azoici și a pigmenților azoici, în principal, depinde de schimbările survenite în tendințele modei și de apariția constantă pe piață a unor coloranți noi, odată cu dispariția celor vechi. Aceasta înseamnă că ar fi dificil de menținut o listă cu acei coloranți azoici care sunt interzișii.

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Fig. 1

Fibrele Dyneema sunt utilizate deja în producerea de mănuși rezistente la tăiere, însă TST Sweden este prima companie care va utiliza aceste fibre în realizarea de echipamente de protecție, primind licența de utilizare a brandului Dyneema.

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Nivelul de protecție poate fi reglat prin varierea numărului de straturi Dyneema, utilizate la oricare dintre echipamentele individuale. Acest lucru înseamnă că echipamentele TST pot fi adaptate cu precizie nivelului de protecție solicitat pentru fiecare tip de activitate.

Echipamentele tradiționale sunt disponibile doar pentru niveluri ridicate de protecție (2 000 bari). Cu alte cuvinte, pentru multe dintre aplicațiile ce solicită protecția împotriva jeturilor de apă, aceste echipamente depășesc nivelul specificațiilor.

În prezent, echipamentele realizate din fibre Dyneema permit adaptarea la un anumit scop. Prin urmare, este posibilă producerea de echipamente particularizate, din perspectiva nivelului de protecție solicitat, optimizând în acest fel confortul purtătorului și manevrabilitatea.

Sursa: www.tst-sweden.com; www.dyneema.com

NANOFIBRE BIOMIMETICE PENTRU REGENERAREA OSOASĂ

Cercetătorii de la ***Institutul de Tehnologie Stevens*** și-au propus elaborarea unei game de nanofibre biomimetice cu conținut de colagen și fosfat de calciu, care vor ajuta la formarea fenotipului de celulele osoase.

Având în vedere faptul că, în fiecare an, în Statele Unite se constată aproximativ 6,2 milioane de fracturi osoase, apărute în urma traumatismelor sau a bolilor, se pune tot mai acut problema creării unor țesuturi osoase destinate chirurgiei reconstructive și transplanturilor.

Standardele actuale referitoare la țesuturile utilizate în reconstrucția osoasă pot avea ca rezultat fuziunea rapidă a oaselor, însă rezistența mecanică a acestora este limitată – de multe ori din cauza lipsei de țesut osos cortical, care este dificil de recoltat fără durere. Finanțat de Fundația americană National Science, dr. Wang Hongjun, profesor în cadrul Departamentului de Chimie, Biologie și Inginerie Biomedicală al Institutului de Tehnologie Stevens, împreună cu colaboratorii săi, au dezvoltat o nouă abordare „bottom-up" pentru reconstrucția țesutului osos, care oferă posibilitatea formării osului cortical în mod ierarhic. În plus, cercetările au fost direcționate spre:

- abordări practice în formarea elementelor asemănătoare osteonului, prin utilizarea nanofibrelor biomimetice și a osteoblastelor;
- folosirea fosfatului de calciu cu conținut de colagen, sub formă de gel, pentru formarea țesutului osos;
- o nouă abordare pentru obținerea țesuturilor osoase asemănătoare celor de tip cortical, prin asamblarea structurilor asemănătoare osteonului într-un singur segment osos, construit prin fuzionare.

În cadrul Proiectul de cercetare *„Realizarea biomimetică a formațiunilor osoase asemănătoare osului cortical cu structură ierarhică"*, vor fi dezvoltate platforme robuste, controlabile și eficiente pentru crearea țesuturilor cu structuri ierarhice complexe destinate chirurgiei reconstructive și a transplanturilor. Biomimetica reprezintă studiul și dezvoltarea sistemelor sintetice care imită formarea, funcția sau structura substanțelor, materialelor, mecanismelor și proceselor biologice.

Echipa de cercetare de la Institutul de Tehnologie Stevens îmbină eficient celulele, metodele și materialele utilizate cu factorii biochimici și fizico-chimici necesari pentru reconstrucția sau înlocuirea porțiunilor de țesut deteriorate. Spre deosebire de cercetările de ultimă oră, axate pe elaborarea de țesuturi osoase cu un grad ridicat de porozitate, cercetările efectuate de dr. Wang se axează pe construcția osului cortical, acesta fiind principalul element cu capacitate portantă.

Pentru crearea unui țesut osos cortical compactat, voluminos, asemănător celui cortical, dr. Wang utilizează o metodă bazată pe nanotehnologie, având o abordare modulară, obținută prin sintetizarea unităților repetitive asemănătoare osteonului și fuzionarea acestora.

Încorporarea nanofibrelor în construcția țesutului osos, pentru formarea unor unități biomimetice mici și repetitive de țesut osos cortical oferă importante avantaje din punct de vedere al suprafețelor mari și al porilor interconectați pentru transportul nutrienților și penetrarea celulară. Un alt avantaj este acela că noul țesut oferă un micromediu biomimetic de formare a țesutului osos prietenos celulei și de mare importanță pentru reconstrucția defectelor osoase complexe.

În următorii ani, echipa de cercetare de la Institutul de Tehnologie Stevens își planifică înregistrarea unor progrese substanțiale în domeniul încorporării nanobiomaterialelor în reconstrucția țesutului osos, pentru crearea unui os cortical cu structură ierarhică și complexitate funcțională.

„*Bogăția mediului intelectual stabilit de dr. Wang și echipa sa reprezintă o sursă de inspirație pentru studenții și absolvenții care participă la transformările benefice ale cercetării de ultimă oră și aplicațiile complexe ale acesteia*" – a declarat dr. Philip Leopold, director al Departamentului de Chimie, Biologie Chimică și Inginerie Biomedicală, din cadrul Universității Stevens.

Smarttextiles and Nanotechnology, octombrie 2010, p. 5

Tricotaje

PERFORMANȚĂ DE TOP ÎN PRODUCEREA MATERIALELOR TRICOTATE PENTRU ARTICOLE SPORT

În ultimii ani, cercetătorii de la ***Institutul Hohenstein***/Germania au fost preocupați de dezvoltarea unor materiale tricotate destinate îmbrăcămintei sport, cu proprietăți termofiziologice și funcționale îmbunătățite (fig. 1).

În acest scop, au fost selectate 34 de materiale tricotate, care au fost testate din perspectiva caracteristicilor lor termofiziologice. Au fost utilizate mostre din diferite tipuri de fibre – poliester, poliamidă, polipropilenă, lână și bumbac, cu o greutate pe unitatea de suprafață ce a variat între 100 și 329 g, cu diverse finisaje de suprafață și cu variate structuri ale tricotului – glat, pichet.

Eșantioanele reprezentative special selectate au fost testate în cadrul unor încercări controlate, ce au implicat participarea unor voluntari, care să le poarte într-o încăpere cu climat controlat. Pentru testarea proprietăților termofiziologice, adică a modului în care sunt transportate prin material căldura și umiditatea, în vederea asigurării unui confort sporit, au fost utilizate mostre care imită pielea, iar pentru evaluarea rezultatelor testării s-au folosit note de la 1 (foarte bun), la 6 (nesatisfăcător).

În ceea ce privește confortul, s-a constatat că aproape toate materialele tricotate destinate articolelor sport, care au fost supuse investigării, au primit calificative de la satisfăcător la foarte bun. În cazul a nouă mostre, notele acordate s-au situat între 1 și 1,5 (foarte bun), iar fibrele chimice s-au dovedit a avea bune proprietăți în ceea ce privește transferul de umiditate și modul de uscare.

Fig. 1

În schimb, mostrele din fibre naturale realizate din lână și bumbac s-au situat în capul listei, în ceea ce privește retenția transpirației. La compararea mostrelor din materiale textile, din poliamidă, cu sau fără o finisare hidrofilă, s-a constatat că finisarea hidrofilă a avut un efect negativ asupra nivelului de confort, deoarece materialul a avut nevoie de mai mult timp pentru a se usca. Totuși, aplicarea unei finisări hidrofile mostrelor realizate din fibre polipropilenice sau dintr-un amestec bumbac-propilenă a prezentat rezultate mai bune din perspectiva confortului, datorită faptului că nu s-au lipit așa de mult de piele.

Proiectul de cercetare a fost finanțat de Ministerul Federal al Economiei și Tehnologiei (BMWi), prin intermediul Federației Asociațiilor de Cercetare Industrială.

www.hohenstein.de

Confecții

NOI ETICHETE DESTINATE ÎMBRĂCĂMINTEI MILITARE, PENTRU PREVENIREA MIROSULUI NEPLĂCUT AL CORPULUI

Compania ***Odegon Technologies*** a dezvoltat o nouă etichetă, care utilizează nanotehnologia pentru a stoca moleculele de miros ale corpului și care se aplică pe articolele de îmbrăcăminte din domeniul militar, în zona axilei (fig. 1).

Fig. 1

Eticheta a fost realizată dintr-o țesătură cu dimensiunile de 7 x 4 cm, care înglobează o plasă tridimensională din carbon activ. Producătorii de îmbrăcăminte livrează eticheta fie aplicată direct pe confecții, fie sub forma unei benzi, care se poate aplica de către utilizatori, acasă, prin presare cu fierul de călcat.

Etichetele sau benzile moi, fără conținut de produse chimice, fără miros, inerte, nonalergene și prietenoase mediului vor rămâne în acel loc pe toată durata de viață a îmbrăcămintei, având performanțe durabile, indiferent de numărul de spălări. Realizată din carbon nanoporos, suprafața materialului activ din interior are o serie de ridicături și adâncituri, atfel încât, dacă ar fi întinsă, aceasta ar avea dimensiunile unui teren de tenis. Moleculele de miros ale corpului sunt absorbite și stocate în nanostructurile materialului, prin forțele Van der Waals, până când îmbrăcămintea va fi curățată, iar moleculele vor fi eliminate.

„*Odegon este o realizare importantă pentru industria serviciilor și pentru milioanele de oameni de pretutindeni, care vor să pună capăt mirosului neplăcut al corpului. Etichetele Odegon au fost descoperite în timpul elaborării unor materiale pentru filtre speciale, destinate protejării personalului militar împotriva unor agenți chimici și gaze letale... Dacă materialul îndeplinește cerințele costumului CRBN, acesta va combate mirosul neplăcut al corpului. Analizând potențialul materialului Odegon pe diferite piețe și în diverse aplicații, s-a constatat un interes semnificativ din partea societăților de îmbrăcăminte corporative și a celor de retail, în special pe piețele din Asia și Europa*" – afirma Tom Rawlings, director general al Odegon Technologies.

Sursa: www.odegon.com

IN MEMORIAM...

La puțin timp după împlinirea venerabilei vârste de 90 de ani, s-a stins din viață ing. ***MAURIȚIU ȚUPERMAN****, specialist de elită în domeniul textilelor, cercetător științific principal, conferențiar universitar asociat și consilier al ministrului industriei ușoare.*

S-a născut în anul 1920, în București. După absolvirea liceului „Mihai Viteazul", a urmat cursurile Școlii Superioare de Textile, iar în 1949 și-a echivalat studiile la Facultatea de Textile din Institutul Politehnic București și a obținut titlul de inginer în specialitatea mecanică – filatura de bumbac. A lucrat la „Filatura de bumbac" – din București, „Textila românească Găvana" – din Pitești și „Filatura românească de bumbac" – din București, ca șef de secție, șef de serviciu, inginer șef. În anul 1951, a început să lucreze la Institutul de Cercetări Textile, fiind printre primii cinci salariați ai noului institut. După ce a fost transferat la Ministerul Industriei Ușoare – în funcția de consilier, iar apoi la Institutul de Proiectări pentru Industria Ușoară – ca șef de proiect, a revenit la Institutul de Cercetări Textile – ca șef de secție în cercetarea-prelucrarea fibrelor și firelor textile. Pe lângă activitatea de coordonare a unor colective de cercetare de interes național, a desfășurat o largă activitate de cercetare în cadrul unor numeroase proiecte de cercetare pe teme legate de prelucrarea fibrelor, firelor și țesăturilor din bumbac. În paralel, a desfășurat o bogată activitate didactică, fiind asistent, șef de lucrări și conferențiar la Facultatea de Textile din Institutul Politehnic București. A publicat numeroase articole în reviste de specialitate și a fost coautor la „Filatura de bumbac". De asemenea, a participat cu referate și comunicări științifice la manifestările organizate CNIT și AGIR.

Pentru contribuțiile aduse în activitate de cercetare științifică, de organizare și dezvoltare a industriei textile românești, inginerului Maurițiu Țuperman i-au fost conferite numeroase Diplome de onoare, Diplome de excelență și decorații.

Biroul Executiv al Consiliului AGIR i-a conferit titlurile onorifice de Membru de Onoare Emerit și de Expert Consultant Magistru al SIT-AGIR, precum și Diploma și Medalia AGIR pentru merite deosebite aduse la creșterea prestigiului AGIR și a profesiei de inginer.

Cerc. șt. dr. ing. ***EMILIA VISILEANU***
Președinte SIT-AGIR
Director general al I.N.C.D.T.P.

Recent a trecut în neființă cel ce a fost dipl. ing. ec. ***STELIAN SIMION GHERMAN****, specialist de înaltă clasă în filarea fibrelor de lână și tip lână.*

S-a născut în anul 1929, în comuna Clopotiva, județul Hunedoara. După terminarea liceului „Decebal" din Deva și, respectiv, „Aurel Vlaicu" din Orăștie, a urmat cursurile Institutului Politehnic – Facultatea de Textile-Pielărie, specialitatea Mecanică – Filatura de lână. A lucrat la „Industria lânii" din Timișoara – ca maistru, șef de secție, șef de producție, inginer șef, iar apoi la „Fabrica de pălării" din Timișoara, până în anul 1970, când a fost transferat la „Combinatul textil" din Arad – în funcția de inginer principal și consilier al directorului general. În perioada 1967–1969 a dat Examenul de Stat la cursurile postuniversitare din cadrul Institului Politehnic din București a obținut diploma de inginer economist. Între anii 1972–1989, a lucrat la „Centrala industriei lânii" din București, în funcțiile de consilier al directorului general, șef al serviciului tehnic, șef al serviciului programarea producției, șef al secției de cercetare, șef al serviciului de specializare-cooperare – șef de producție filaturi, șef al serviciului de organizare. În anul 1989 s-a pensionat. Începând cu anul 1992, a activat cu contract civil de muncă la o reprezentanță din București, pentru un grup de firme franceze, în calitate de consilier.

Ing. Gherman a fost membru al organizațiilor inginerești – ASIT, CNIT, AGIR și SIT-AGIR. A participat cu referate și comunicări științifice la numeroase seminarii, simpozioane și conferințe. A colaborat la elaborarea „Manualului inginerului textilist", fiind revizor tehnico-științific la secțiunea de filatură de lână.

În tinerețe a practicat sportul, iar în ultimii ani ai vieții a devenit un mare iubitor al muzicii clasice, un nelipsit audient al concertelor de la Ateneul Român și Opera Română.

Datorită contribuțiilor aduse în cadrul Societății Inginerilor Textiliști din România, Consiliul de conducere al SIT-AGIR i-a conferit Diplome de onoare și Diplome de excelență, precum și titlul profesional onorific de de expert consultant magistru al SIT-AGIR.

Cerc. șt. prof. ing. ***ARISTIDE DODU***
Președinte de onoare al SIT-AGIR
Membru de onoare al ASTR și AGIR

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