The behavior in finishing of textile materials made of man-made fibers containing ZnO in blends with cotton

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REZUMAT – ABSTRACT

Comportamentul la finisare al materialelor textile realizate din fibre artificiale cu conținut de ZnO în amestecuri cu bumbac

Obiectivul acestui studiu a fost de a investiga comportamentul la finisare al materialelor textile realizate din fibre artificiale cu conținut de ZnO în amestec cu bumbac. Au fost studiate posibilitățile de reducere a concentrației agenților chimici considerați a fi agresivi pentru fibrele textile funcționale, temperatura și durata proceselor, precum și numărul operațiilor tehnologice efectuate, astfel încât calitatea vopsirii să nu aibă de suferit, iar probele vopsite să fie acceptabile din punctul de vedere al uniformității și al rezistenței vopsirii. Pentru a evidenția influența auxiliarilor chimici utilizați în operațiile de finisare, temperatura procesului, pH-ul și durata tratamentului asupra caracteristicilor fizico-chimice și fizico-mecanice ale fibrelor funcționale, s-au aplicat diferite metode de tratare preliminară și vopsire în diferite variante experimentale. Pentru evaluarea performanţelor tratamentelor preliminare din punctul de vedere al gradului de alb și al hidrofiliei, țesăturile au fost testate înainte și după efectuarea tratamentelor preliminare. În scopul determinării eficienţei tratamentelor realizate, ţesăturile finite (tratate preliminar și vopsite) au fost testate din punctul de vedere al diferenței de culoare și al rezistenței vopsirii. Țesăturile finite au fost, de asemenea, caracterizate din punctul de vedere al principalelor caracteristici fizico-chimice și fizico-mecanice: masa, rezistența la tracțiune, rezistența la rupere, permeabilitatea la vaporii de apă, permeabilitatea la aer. Analiza SEM a fost utilizată pentru a investiga morfologia de suprafață a țesăturilor tratate. Activitatea antibacteriană a probelor tratate a fost testată împotriva tulpinii test Staphylococcus aureus.

Cuvinte-cheie: fibre funcționale, tratament de finisare preliminară, proprietăți fizico-mecanice-chimice, activitate antibacteriană

The behavior in finishing of textile materials made of man-made fibers containing ZnO in blends with cotton

The objective of this study was to investigate the behavior in finishing of textile materials made of man-made fibers containing ZnO in blends with cotton. It has been studied the possibilities of reducing the concentration of the chemical agents considered to be aggressive for the functional textile fibers, the temperature and the duration of the processes, as well as the number of technological operations performed, so that the dyeing quality will not suffer and the dyed samples to be acceptable from the uniformity and fastness point of view. To highlight the influence of the chemical auxiliaries used in finishing operations, the process temperature, pH and the treatment duration on the physical-chemical and physical-mechanical characteristics of the functional fibers, various methods of preliminary treatment and dyeing were applied in different experimental variants. In order to assess the preliminary treatments performance from whiteness degree and hydrophilicity point of view, the fabrics were tested before and after preliminary treatments. Finished fabrics (preliminary treated and dyed) were tested for the efficiency of the performed treatments in terms of color difference attributes and color fastness. The finished fabrics were also characterized in terms of the main physical-chemical and physical-mechanical characteristics: mass, tensile strength, tearing strength, water vapor permeability, air permeability. SEM analysis was used to investigate the surface morphology of treated fabrics. Antibacterial activity of treated samples was tested against the Staphylococcus aureus test strain.

Keywords: functional fibres, preliminary finishing treatment, physical-mechanical-chemical properties, antibacterial activity

INTRODUCTION

Textile industry continuously searches for new technologies in order to accomplish the consumers' demands. Especially in recent years, new developments allowed the production of functional and smart textiles which are capable of sensing changes in environmental conditions or body functions and responding to these changes. As a consequence, the number of bio functional textiles with an antimicrobial activity has increased considerably over the last few years [1–3]. Several major classes of synthetic (quaternary ammonium compounds, silver, polyhexamethylene biguanides (PHMB), triclosan) or natural antimicrobial agents (chitosan, plant extracts, essentials oils) are used in the textile industry in order to control the bacterial growth efficiently and to keep its durability [4–9].

Antimicrobial agents can be applied to textile by different methods such as pad-dry-cure, coating, spraying and foam techniques. It can also be applied directly by adding the antimicrobial agent into the spinning fiber solutions. Development of antimicrobial fibers is based on the process of incorporating active antimicrobial agents into the fiber in its manufacturing stage and this technology is rather increasing today, mainly being supported by fiber manufacturers [10]. Some commercial bioactive fibres with antimicrobial agents and finishing products are: SeaCellR Active, a cellulose-base fibre; MicroFresh®, SoleFresh® and Guard-Yarn®, polyester or nylon yarns with Alpha San®, a zirconium phosphate-based ceramic ion-exchange resin containing silver; Trevira Bioactive®: polyester fiber with silver incorporated prior to the extrusion process; SmartSilver®, wool fibers with silver added by typical exhausting dyeing methods and other finishing silver-based products like Smart Silver™, SilpureR, Sanitized®, AlphaSan® and Ultra-Fresh. Cotton fibers are also being commercialized under a pre-treatment with ReputexR (PHMB attached to cotton) and more recently, polyamide with PHMB is sold as Purista®. Moreover, polyamide and polyester fibers treated with Tinosan AM 100®, cellulose acetate yarns named Silfresh®, Microban® textile and Irgaguard® and Irgacare® products, all contain triclosan as antimicrobial agent [11].

Smartcel™ Sensitive fibers developed by Smartpolymer (Germany) are Lyocell fibers that have integrated ZnO into their matrix for skin protection and skin care, produced without aggressive chemical agents for the skin [12]. Textiles materials containing these novel zinc fibers allow natural, soft and pure skin care with an additional antibacterial and odor-reducing effect. The antibacterial effect of zinc is based on the "oligo dynamic effect" which describes a disruption in the antibacterial metabolism.

For the finishing of textile materials made of functional fibers have to pay special importance on every technological process involved in the technological flow of finishing, so that the additives contained in the fiber structure, giving them functionality, are not eliminated by the finishing, dyeing or final finishing procedures applied. In this respect, the specific objective of this study was to investigate the behavior in finishing of textile materials made of man-made fibers containing ZnO in blends with cotton. It has been studied the possibility of reducing the concentration of the chemical agents considered to be aggressive for the functional textile fibers, the temperature and the duration of the processes, as well as the number of technological operations performed, so that the dyeing quality will not suffer and the dyed samples to be acceptable from the uniformity and fastness point of view.

EXPERIMENTAL WORK

Materials

For laboratory experiments 80% cotton/20% Smartcel™ Sensitive (with ZnO content) plain weave fabric with 290 yarns/10cm in warp and 230 yarns/10cm in weft, 246 g/m^2 weight was used. For preliminary finished operations Kemapon PC/LF (Kem Color S.p.a, Italy) or Imerol JFS (Clariant) was used as wetting and detergent agents. Kemaxil Liq (Kem Color S.p.a., Italy) was used as stabilizer agent for H_2O_2 and Sequion 48/98 (Giovanni Bozzeto S.p.a, Italy) was used as a dispersant and sequestrant for calcium, magnesium and iron ions. Sirrix SB (Clariant), a multi-action anionic product, has been used as a dispersant for fatty impurities, oxygen active generator, activator and stabilizer for H_2O_2. For pre-treatment operations performed at low temperatures, Imerol LTB (Archroma) was used, a chemical product especially designed for the low temperature bleaching process, having superior wetting and removal properties of fatty impurities, oils, accidental pigments and high emulsifying properties of impurities, and as H_2O_2 stabilizing agent was used the product formulated for low temperature process, Stabilizer LTB (Archroma).

Preliminary treatments

To highlight the influence of the chemical auxiliaries used in finishing operations, the process temperature, pH and the duration treatment applied, on the physical-chemical and physical-mechanical characteristics of the functional fibers, various methods of preliminary treatment and dyeing were applied, in different experimental variants. Laboratory experiments were performed on the jigger (Roaches-England) laboratory apparatus at 1:10liquor ratio (material: liquor ratio), as follows:

• **Strong alkaline pre-treatment-bleaching in successive phases (classical process)** (**Code V_1**): Alkaline treatment – Bath 1 ® 2 g/L Kemapon PC/LF, 2 g/L Sequion 48/98, 6 ml/L NaOH 38°Be, 3 g/L trisodium phosphate, 90 minutes, 98°C; Bath 2 ® Bleaching: 1g/L Kemapon PC/LF, 2 ml/L Kemaxil Liq, 4 ml/L NaOH 38ºBe, 20 ml/L H_2O_2 30%; 60 minutes, 98°C;

• **Mild alkaline pre-treatment-bleaching in successive phases (mild process)** (**Code V_2**): Alkaline treatment – Bath 1 ® 2 g/L Kemapon PC/LF, 2 g/L Sequion 48/98, 2 g/L Na_2CO_3; 45 minutes, 98°C; Bath 2 ® Bleaching 1g/L Kemapon PC/LF, 2 mL/L Kemaxil Liq, 2.3 mL/L (pH = 12) NaOH 38ºBe, 20 mL/L H_2O_2 30%; 45 minutes, 95ºC;

• **Pre-treatment in single phase with a multiple action chemical auxiliary (mild process) (Code V_3):** 0.5 g/L Imerol JSF, 0.8 g/L Sirrix SB, 2.6 g/L NaOH 38ºBe, 6.5 g/L H_2O_2 30%; 30 minutes, 95ºC;

• **Preliminary treatment – dyeing in single phase with a multiple action chemical auxiliary** (preliminary treatment followed by dyeing without intermediate rinsing) (**Code V_4**): Bath 1 ® 0.5 g/L Sirrix SB, 0.8 g/L, 2.6 g/L NaOH 38° Be, 6.5 g/L H_2O_2 30%; 30 minutes, 95ºC ® Bath evacuation, without intermediate rinsings ® Bath 2 ® 0.2 mL Sirrix NE, 0.35 g/L Bactosol ARL liq. (catalase) ® adding of appropriate chemicals and dyes for dyeing operation;

• **Pre-treatment in a single-phase with low concentration of chemical auxiliary special formulated for low temperature processes** (**Code V_5**) at: 0.5 g/L Imerol LTB, 1g/L Stabilizer LTB, 3 g/L NaOH 38°Be, 4 g/L H_2O_2 30%; 40 minutes, 80°C;

• **Pre-treatment in a single-phase with higher concentration of chemical auxiliary special formulated for low temperature processes** (**Code V_6**): 1 g/L Imerol LTB, 1 g/L Stabilizer LTB, 6 g/L NaOH 38°Be, 4 g/L H_2O_2 30%; 40 minutes, 80°C.
After the preliminary treatment operations the samples were successively rinsed with water at 90°C, 70°C, 40°C and cold rinsing, except for V_4V.

Dyeing operation

After the preliminary treatments, the dyeing operation has been performed by using the following dyeing recipe: 1.5% Drimaren Gelb CL-2R, 70 g/L NaCl (added in two portions), 20g/L Na_2CO_3 (added in two portions). After dyeing operation the samples were rinsed as follows: warm rinsing at 60°C, soaping with 1 g/L Kemapol SR (Kemcolor) at 90°C, 20 minutes, rinsing at 80°C, 40°C, 30°C and cold, 10 minutes each rinsing, followed by drying at room temperature.

Methods

Physical-chemical and physical-mecanical characteristics

In order to asses the preliminary treatments performance, the 80% cotton/20% Smartcel™ Sensitive fabrics were tested before and after preliminary treatments in terms of whiteness degree (SR EN ISO 105-J01:2003) and from hydrophilicity point of view by determining the wettability (drop test method according with SR 12751/1989 standard) and the water absorbency (capillarity test according with SR 6146/1989 standard). Finished fabrics (preliminary treated and dyed) were tested for the efficiency of the performed treatments in terms of color difference attributes (SR ISO 105 J03: 2001) and color fastness to washing (SR EN ISO 105-C10:2010), acid and alkaline perspiration (SR EN ISO 105-E 04: 2013) and light (SR EN ISO 105-B02: 2003).
The finished fabrics were also characterized in terms of the main physical-chemical and physical-mechanical characteristics, respectively: mass (SR EN 12127-2003), density (SR EN 1049-2: 2000-Method A, B), tensile strength (SR EN ISO 13934-1/2013), tearing strength (SR EN ISO 13937-3: 2002), water vapor permeability (STAS 9005: 1979), air permeability (SR EN ISO 9237: 1999).

Antibacterial testing

The antibacterial activity of the dyed samples and pre-treated in different variants was qualitatively determined according with the ISO 20645: 2004 (E) standard method, by using of cultures in liquid medium replicated at 24 hours of ATCC 6538 *Staphylococcus aureus* strains (Gram-positive). For determination, the samples were cut in circular shape with a diameter of 2 cm and subsequently disposed in the middle of Petri plates. The culture medium was poured into two layers in Petri plates, lower layer consists of culture medium free from bacteria and the upper layer being inoculated with the test bacteria, then incubated at 37°C and analyzed after 48 hours.

Scanning Electron Microscopy (SEM)

The surface morphology of treated samples in different variants was investigated by a FEI Quanta 200 Scanning Electron Microscope with a GSED detector, at 2000 x magnification and accelerating voltage of 12.5 kV – 20 kV.

Energy Dispersive X-ray analysis (EDX)

EDX was used to identify the presence of Zn in textile materials. The analysis was made with a FEI Quanta 200 Scanning Electron Microscope coupled with EDX detector. The detector has the ability to convert the X-ray energy emitted by the samples into voltage signals that are specific to different chemical elements.

RESULTS AND DISCUSSIONS

Whiteness degree

The values obtained for the whiteness degree of 80% cotton/20% Smartcel™ Sensitive fabrics preliminary treated in different variants, are shown in table 1.
From the series of experimental variants is highlighted the alkaline pre-treatment – bleaching in successive phases (classical and mild process) (V_1 and V_2) for which higher values of the whiteness degree were found. Lower values of the whiteness degree are obtained for the preliminary treatments in single phase using the multiple action chemical auxiliary (Sirrix SB) (V_3) and also for the low temperature processes (Imerol LTB) (V_5).
Tha values obtained for the hydrophilicity of 80% cotton/20% Smartcel™ Sensitive fabrics after the applied preliminary treatments are shown in table 2.
From the table 2 it can be seen that the hydrophilicity is good for all applied preliminary treatment (below

Table 1

WHITENESS DEGREE

Code	Whiteness degree (Berger)	Whiteness degree (CIE)
V_1	75.58	76.16
V_2	73.25	74.40
V_3	55.55	56.41
V_5	45.99	45.82
V_6	60.36	60.05

Table 2

HYDROPHILICITY FOR 80% COTTON / 20% SMARTCEL™ SENSITIVE FABRICS PRELIMINARY TREATED IN DIFFERENT VARIANTS

Hydrophilicity	Raw	Code				
		V_1	V_2	V_3	V_5	V_6
Wettability, drop test [s]	> 600	< 1	< 1	2	< 1	< 1
Water absorbency [%]	-	58.52	52.44	44.09	45.75	51.65

1 second according to drop test and water absorbency between 44% and 58% respectively). Slightly lower values of hydrophilicity is obtained for the fabric treated in single phase with the multiple action chemical auxiliary (mild process) (V_3), for which a wettability of 2 seconds and 44% water absorbency have been obtained.

Color measurements

Colour differences attributes obtained for 80% cotton/ 20% Smartcell^{TM} Sensitive fabrics preliminary treated in different variants and dyed with Drimaren Gelb CL-2R are presented in table 3. As reference, the sample treated by strong alkaline treatment – bleaching in successive phases (classical process) (Code V_1) was used. The values obtained reveal significant color differences between the analyzed samples. This behavior is due to the fact that the applied treatments provide a differentially removal of the natural impurities of cotton (pectin, waxes, pigments) depending on: the chemical auxiliaries used in the process, the main process parameters (pH, temperature, treatment duration), the number of operations and rinsings performed with water at high temperature, with implicit influences on whiteness degree and hydrophilicity. In conclusion, the dye uptake varies from one variant to another, being influenced by all these factors. Compared to the reference samples treated by strong alkaline pre-treatment – bleaching in successive phases (classic process) (V_1), the total color difference (DE*) of pre-treated samples in different variants ranging between 2.14 and 5.30, which corresponds to a total color difference of ½ up to 2½ tons compared to the reference. Analyzing the obtained data, it is possible to appreciate that the classical treatment in successive phases, considered the reference standard, is the most effective in terms of removing the impurities of cotton and implicitly in terms of dye adsorption.

Table 3

COLOR DIFFERENCES ATTIBUTES FOR FABRICS PRELIMINARY TREATED IN DIFFERENT VARIANTS AND DYED

Code	Color differences				
	DL*	DC*	DH*	DE*	Mark
V_1	Refference				
V_2	–0.25	2.10	–0.33	2.14	4–5
V_3	–2.13	4.62	–1.49	5.30	3–4
V_4	–2.73	3.41	–1.66	4.67	3

Color fastness

Regardless of the pre-treatment method, applied prior to dyeing operation, color fastness to washing, acid and alkaline perspiration, dry and wet rubbing are very good, with marks obtained for change of shade and staining of the multi-fiber standard between 4–5/5 (table 4).

The main physical-mechanical characteristics are present in the table 5. Analyzing the obtained results,

Table 4

COLOR FASTNESS

Code	Washing				Acid perspiration				Alkaline perspiration				Rubbing	
	Color change	Color staining			Color change	Color staining			Color change	Color staining			Dry	Wet
		CO	PA	W		CO	PA	W		CO	PA	W		
V_1	4-5	4-5	4-5	4-5	4-5	4-5	4-5	5	4-5	4-5	4-5	5	4-5	4-5
V_2	4-5	4-5	4-5	4-5	4-5	5	4-5	5	4-5	4-5	4-5	5	4-5	4-5
V_3	4-5	4-5	4-5	4-5	4-5	5	4-5	5	4-5	4-5	4-5	5	5	4-5
V_4	4-5	4-5	4-5	4-5	4-5	4-5	5	5	4-5	4-5	5	5	5	4-5
V_5	4-5	4-5	4-5	4-5	4-5	4-5	5	5	4-5	4-5	5	5	5	4-5
V_6	4-5	4-5	4-5	4-5	4-5	4-5	5	5	4-5	4-5	5	5	5	4-5

Table 5

PHYSICAL-MECHANICAL CHARACTERISTICS

Code	Mass [g/cm^2]	Density [No counts/10cm]		Tensile strength [N]		Elongation at break [%]		Tearing strength [N]	
		Weft	Warp	Weft	Warp	Weft	Warp	Weft	Warp
Raw fabric	246	290	230	1788	717	21.8	11.30	71.3	38.9
V_1	260	310	236	1716	787	26.8	18.26	33.1	15.67
V_2	260	324	238	1823	786	26.5	19.51	31.9	15.50
V_3	261	314	232	1778	780	28.0	18.92	54.4	35.4
V_4	265	306	240	1892	818	38.0	15.0	61.2	39.5
V_5	265	310	237	1879	627	32.1	25.9	44.9	34.5
V_6	263	312	234	1757	671	30.4	18.05	38.3	19.36

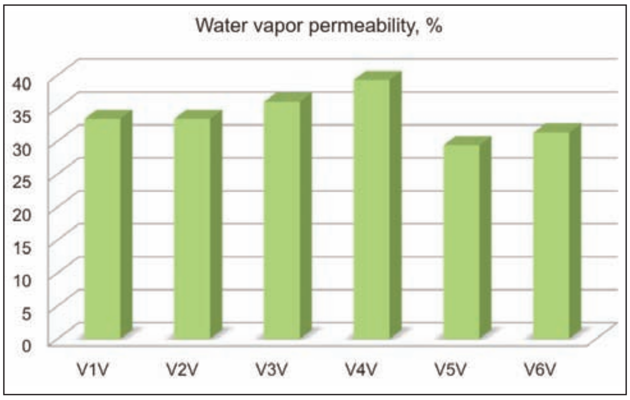

Fig. 1. Water vapor permeability of fabrics treated in different variants

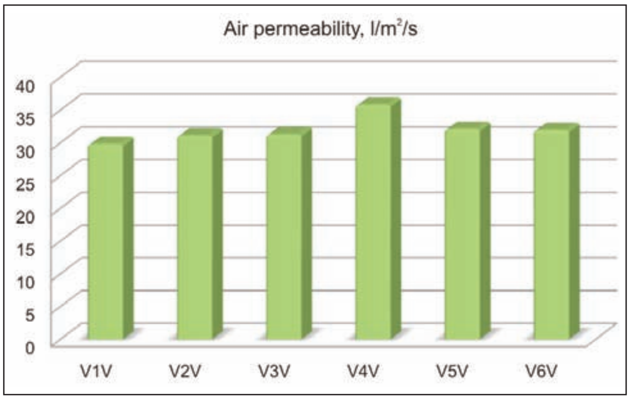

Fig. 2. Air permeability of fabrics treated in different variants

it can be observed that, during the finishing process, the fabric contraction took place, leading to the increasing of the mass (g/m^2) and density in the warp and weft direction, compared to the raw fabric, without significant differences between variants. This behavior is normal for the finishing processes of cotton fabrics carried out in aqueous medium and high temperatures. Tensile strength does not show significant changes after finishing treatments, just only small variations (decreases or increases) ranging from 0.5 to 5.8 % as compared to the raw fabric, but these variations can be considered negligible.

Tearing strength shows decreases values in the case of classic treatment variant – hot alkaline treatment in the presence of NaOH (V_1) and also for the mild alkaline treatment in the presence of Na_2CO_3 (V_2), with more than 50% decreases of values in warp and weft direction. From the applied treatments, the preliminary treatment with the multiple action chemical auxiliary followed by dyeing without intermediate rinsings (V_4) affects the least the tearing strength of the treated samples.

The air and the water vapor permeability recorded for the 80% cotton/20% Smartcell™ Sensitive fabrics have certain variations between the experimented finishing variants, in close relation with the fabrics density. Higher values of this caracteristics is obtained for the fabric pre-treated with the multiple action chemical auxiliary followed by dyeing without intermediate rinsings (V_4) (figures 1–2), for which the lowest value of the density in weft direction is obtained.

SEM-EDX

Electronic images recorded at a magnification of 2000 x for textile materials treated in different variants are shown in figure 3. Analyzing the obtained images can be observed that applied preliminary treatments do not change differentially the surface of functional man-made cellulosic fibers.

Quantification of the Zn content existing in the matrix of Smartcell™ Sensitive fibers is shown in table 6.

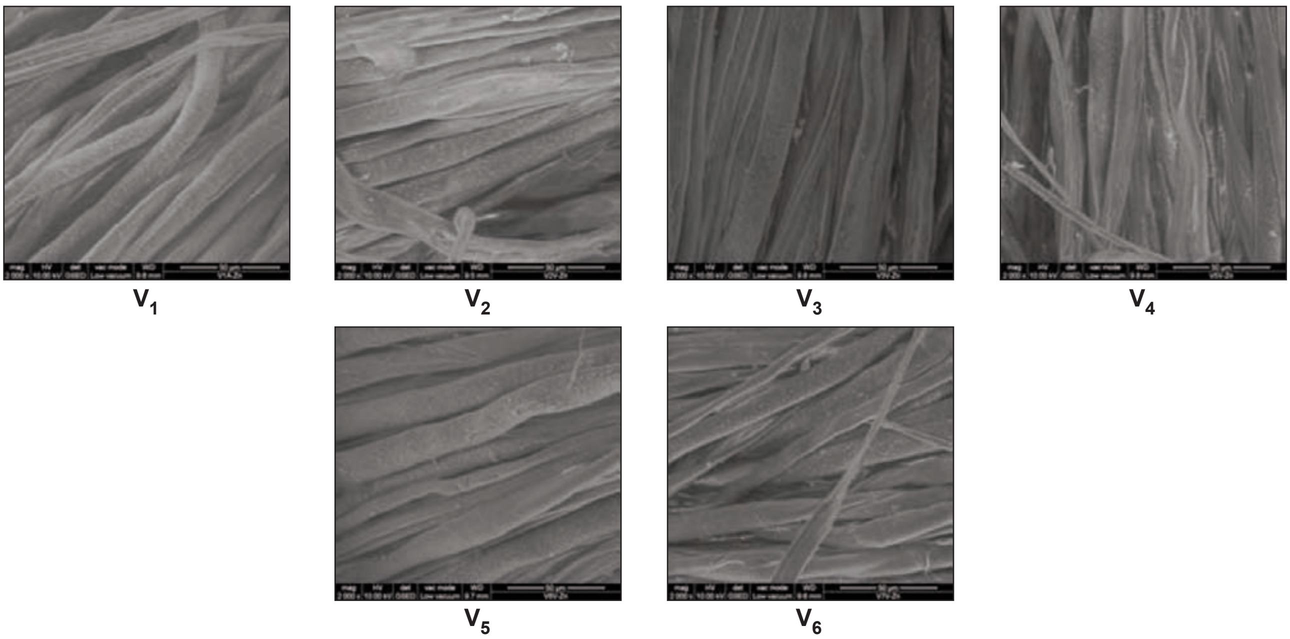

Fig. 3. SEM images of textile materials treated in different variants

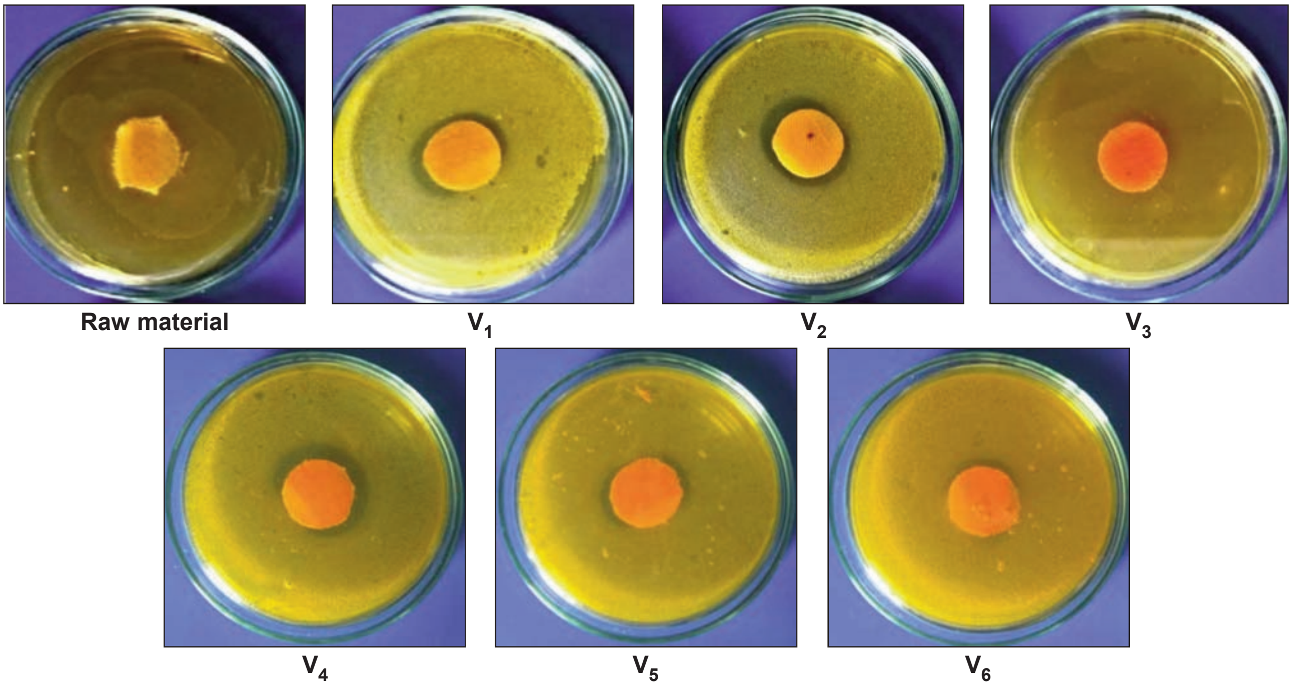

Fig. 4. Images of Petri plates after 48 h incubation

Table 6

Zn CONTENT							
Zn content		Code					
	Raw	V_1	V_2	V_3	V_4	V_5	V_6
Wt %	11.39	1.32	9.92	9.63	7.62	2.34	5.59
At %	2.59	0.30	2.28	2.21	1.71	0.51	2.10

Analyzing the obtained data, it can be appreciated that regardless of the finishing applied variant, the Zn presence was identified in the percentage of mass between 1.32% and 9.63%. It should be noted, however, that the appreciation of the Zn content is qualitative and by this method it was not possible to make a definite differentiation between the experimented variants.

Antibacterial activity

Images of Petri plates after 48 h incubation are shown in figure 4.

The results obtained from the evaluation of antimicrobial activity for the treated samples in different experimental variants are shown in table 7.

From the evaluation of the results obtained for all the variants. an antibacterial effect against the *Staphylococcus aureus* test strain is observed. In the case of the raw fabrics (unfinished) and the samples treated according to the pre-treatment in single phase with a multiple action chemical auxiliary Sirrix SB (code V_3), the inhibition zone or the *Staphylococcus aureus* test strain in the whole medium of the culture was not observed, thus completely inhibiting the growth and inhibition of the test microorganism.

Table 7

ANTIMICROBIAL ACTIVITY		
Sample	Inhibition zone [mm]	Evaluation
Raw material	-	Satisfactory effect
V_1	2.5	Satisfactory effect
V_2	4	Satisfactory effect
V_3	-	Satisfactory effect
V_4	5	Satisfactory effect
V_5	2	Satisfactory effect
V_6	4	Satisfactory effect

CONCLUSIONS

Laboratory experiments have highlighted that the hydrophilicity obtained after pre-treatment is very good for the all experimental variants, slightly lower values of hydrophilicity is obtained for the fabric pre-treated in single phase with the multiple action chemical auxiliary by a mild process (V_3). The strong alkaline treatment – bleaching in successive phases (V_1 – classical process), being considered the reference standard process, is the most effective in terms of removing the impurities of cotton and implicitly in terms of hydrophilicity, whiteness degree and dyes adsorption. However, tearing strength shows significant decreases values in the case of classical treatment variants – hot alkaline treatment in the presence of NaOH (V_1) or mild alkaline treatment in the presence of Na_2CO_3 (V_2), with over 50% decreases of values in warp and weft direction. The preliminary treatment affecting the least tearing strength of the treated sample is the one who uses the multiple action chemical auxiliary in the first process step being followed by the dyeing, without intermediate rinsings between technological operations (V_4). In terms of tensile strength, there are no significant differences between experimented finishing treatments, yet existing small variations (decreases or increases) compared with the raw fabric, being considered negligible. Color fastness to washing, acid and alkaline perspiration, dry and wet rubbing fastness are very good, with marks obtained for all the samples between 4–5/5. An antibacterial effect against the *Staphylococcus aureus* test strain is observed for all finished samples, with or without in inhibition zone.

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