ANTI-ALLERGIC CELLULOSE SUPPORT AT THE EPIDERMIS–ENVIRONMENT INTERFACE

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This paper is part of a study on the development and design of some cellulose articles intended for certain cutaneous allergic diseases: contact dermatitis (CD) and atopic dermatitis (AD). The study is based on the utilization of 100% cotton supports grafted with a cyclodextrin product, namely monochlorotriazinyl-beta-cyclodextrin (MCT-β-CD), for the inclusion of some natural active principles (AP), such as *Viola tricoloris Herba* (VTH), menthol (Ml) and propolis (Pr) ,with anti-allergic properties. The cellulose support addressing patients with cutaneous affections realizes a system for the transdermal controlled release of the AP from the cavities of the cyclodextrin grafted on the cellulose support, to the hypoderm level of the skin. In order to highlight the grafting with cyclodextrin reactive product and the inclusion of anti-allergic AP, the cellulose support was characterized by Fourier transform infrared spectroscopy (FTIR) measurements and nitrogen elemental analysis, while the morphological formations were rendered evident through scanning electron microscopy (SEM). The humidity profile and conduction were determined by the utilization of a liquid miming the perspiration transfer through the textile material treated with anti-allergic AP, as compared to a standard.

Keywords: cellulose textile, anti-allergic properties, cyclodextrin, active principles, moisture management tester

INTRODUCTION

In recent years, the utilization of textiles with medical properties has reached a high demand in the medical field, for both personal care and public health.[1] The prevalence of allergies nowadays represents a challenge for the realization of textile materials with medical functionality.

Textiles with anti-allergic properties represent a novelty. The research objective consists in designing and manufacturing a knitted fabric from 100% cotton fibers addressing patients with cutaneous allergic manifestations, such as contact dermatitis (CD) and atopic dermatitis (AD).

Research attempts have been made worlwide focusing on cutaneous affections, and hypo-allergic or antimicrobial articles have been produced, intensifying the dermal comfort without the precise target of anti-allergic therapy. For example, hypo-allergic seat covers have been made for Volvo automobiles and antimicrobial products based on silver particles were realized. Also, a soft handle textile has been produced increasing comfort and dealing gently with the skin with itchiness, hypersensitivity or with urticaria type problems, using certain agents that inhibit the microorganisms development or create a microclimate in the textile material/skin system, with the role of nourishment, hydration and regeneration of a healthy skin.[2-7]

Allergic diseases

The prevalence of allergies, defined as immunologically mediated hypersensitivity, is increasing worldwide. It has been estimated that more than 20% of the population of the developed countries suffer from a form of allergic diseases, such as allergic asthma, allergic rhinitis/allergic conjunctivitis, atopic dermatitis, etc. Children are prone to bear the greatest burden of allergic diseases. For instance, 80% of the children from developed countries are allergic, even if only 5-15% of them have a medical record. The most common allergic conditions in children and teenagers are food allergies, contact dermatitis and asthma.[8,9]

In Romania, studies show that the prevalence of atopic dermatitis, for instance, remains constant. The only diseases with an increasing tendency are asthma, allergic rhinitis and food allergy.[10]

Detailed knowledge of the pathological manifestations is in straightforward correspondence with the criteria for designing a textile article designed for allergic patients.

CD is characterized by redness, itching, appearance of vesicles and, in a more chronic form, scaly desquamation, resulting from exposure to environmental chemicals (cosmetic products, metals like nickel, dyes, synthetic and natural fibers, especially wool fibers and so on). The location and morphological characteristics of the lesions are determined by the causative exposure. CD can be classified as: irritant contact dermatitis and allergic contact dermatitis. It is estimated that CD accounts for 4-7% of dermatological consultations.[11,12]

AD – also known as eczema or atopic eczema – is an inflammatory, chronically relapsing, non-contagious and extremely pruritic skin disease.[13] AD is today the most common, chronic inflammatory skin disease among children in developed countries. The AD is a disease of civilization, its cumulative prevalence varying from 20% in northern Europe and the USA to approximately 5% in Mediterranean countries. The eczematous disease creates a favorable medium for the development of *Staphylococcus aureus*, which is present in 95% of patients with AD.[14]

In the topic dermatologic therapy, the textile supports are used as a barrier against the acarids present in domestic dust (which are allergic vectors), or as humid compresses in the amelioration of itchiness and of the discomfort of eczematous locations.[15,16]

The present research aims at producing a cotton support with anti-allergic action, making use of the anti-allergic properties of some natural products (in our case AP), such as *Viola tricoloris Herba* (VTH), menthol (Ml) and propolis (Pr).[17,18] The study had as an objective the realization of a clean and absorbing textile material, soft handle and antiseptic, for which the finishing stages and working receipts have been established. The conformation and specific size of AP have been established,[19] in order to allow their penetration into the hydrophobic cavity of the cyclodextrin compound. The lethal dose and the therapeutic concentration of AP were determined by *in vivo* tests; a hierarchy was established concerning the efficacy of the natural products used, as well as the physiological aspects which intensify the efficiency of the therapy with natural AP. The procedure of assembling the textile support parts was determined, in order to produce pyjamas consisting of a blouse and trousers for CD patients and AD patients.[19-22]

The paper presents the results of the investigation (carried out by SEM, FTIR and elemental analysis) of cellulose grafted with MCT-β-CD, as a support for the formation of an inclusion compound for the physical absorption of AP, and reveals the moisture diffusion through the anti-allergic textile fabrics, by the method of liquid moisture management properties (moisture management tester – MMT).

EXPERIMENTAL

Materials

A tubular knitted fabric with an interlock structure, 100% cotton yarn with the fineness of Nm = 60/1, was used as a textile support for designing the anti-allergic articles. After a previous preparation, the knitted fabric was grafted with MCT-β-CD (CAVATEX W7 MCT, purchased from Wacker Chemie), and then the AP were included into the grafted fabric.

VTH represents a plant gathered from the spontaneous flora of Romania, used in the study as aqueous solution (25%); Ml was purchased from the Sigma Aldrich Co., and Pr was collected from Danesti region, in the East of Romania. The last two were used as solutions in alcohol (30% w/v).

The textile support for the patients with dermatological diseases must realize a system for the controlled release of a drug or an active principle with anti-allergic action. Another condition is to have an antibacterial influence, in order to provide the main anti-allergic curative action and to avoid synergy between the bacterial flora present on the surface of the skin and the allergic eruptive evolution.

According to the manufacturing protocol, the VTH, Ml or Pr were fixed on certain stripes of finished knitted fabric prepared in accordance with the preparation and treatment technology,[19,20] which are then assembled on the inside of the vestimentary article (pyjamas), following the distribution and the surface size of the lesions indicated by the clinical observations of the dermatologist.[19]

Methods

The conditions that need to be satisfied by the cellulose textile supports that come in direct contact with the skin are as follows: to be anti-allergic, bactericide, anti-viral, bacteriostatic, fungistatic, non-toxic, highly absorbing, breathable and biocompatible. With this aim in view, the following operations for the preparation of the cellulose support were carried out: alkaline boiling treatment (at 100 °C) in order to remove natural impurities, and bleaching with hydrogen peroxide in order to obtain an advanced sterility, soft handle and adequate wettability. After the preparation stage, the knitted fabric was grafted with MCT-β-CD through padding and thermal treatment at 160 °C; then the AP were applied on the textile surface by a spraying technique, followed by a heating treatment for 4 hours at 50 °C. The procedures of cellulose support preparation, grafting treatment with MCT-β-CD and application of AP are presented in detail in the works of Radu *et al.*[18-21]

The confirmation of grafting and formation of inclusion compounds (in our case with AP) on the cellulose support grafted with MCT-β-CD was obtained by nitrogen elemental analysis (carried out at the University of Montpellier 2, France, by courtesy of Prof. V. Hulea) and by the FTIR spectroscopic method. SEM analysis was used to render evident the AP presence on the surface of knitted fabrics grafted with MCT-β-CD. The SEM and FTIR investigations were carried out at the National Research Centre (CNR-ISMAC) from Biella, Italy (by courtesy of Mr. A. Varesano). The tests of the liquid moisture management properties were performed at the Polytechnic University of Turin, Advanced Technology Textile Laboratory, from Biella, Italy.

The nitrogen elemental analysis was carried out using a CHNS-O Analyzer Flash EA 1112 Elemental Analyzer, using the combustion method combined with gas chromatography by means of a TC detector.

The FTIR spectroscopic analysis was performed with a Thermo Nicolet Nexus spectrometer, by attenuated total reflection (ATR) technique with a Smart Endurance accessory, in the range of 1650 to 550 cm^{-1}. ATR is a powerful tool for investigating evenness, thickness and coating degree, because the infrared beam is able to analyze the thin layers of the fiber surface, the work procedure was according to Varesano.[23]

SEM investigation was performed with a LEO (Leica Electron Optics) 435 VP SEM Scanning electron microscope, at an acceleration voltage of 15 kV and a 22 mm working distance (between the sample and the scanning head). The fabrics were mounted on aluminum specimen stubs with double-sided adhesive tape. In order to improve the quality of SEM images, the samples were sputter-coated with a thin gold layer in rarefied argon atmosphere (20 Pa) using an Emitech K550 Sputter Coater, with a current of 20 mA for 80 seconds, according to the work procedure described by Varesano.[23]

The measurement, evaluation and classification of moisture management testing of the cellulose fabrics, untreated and treated with active principles, were performed according to AATCC Test Method 195-2009[24] on Moisture Management Tester M290 SDL (MMT) Atlas (England). This equipment has two horizontal (upper and lower) electrical sensors, each with seven concentric pins so that the fabric sample can be placed between the two sensors. The upper sensor can be brought down during the experiments. The measurement principle of MMT instrument consists in dripping a pre-established amount of test solution (in this case, 0.9% sodium chloride solution and distilled water) from the upper sensor onto the center of the textile fabric that comes in touch with skin (called “inner surface”). Thus the testing solution was transferred onto the surface of cellulose fabric in three directions: (i) the radial spreading toward the top surface of the material; (ii) the movement through the textile material from the inner surface toward the outer surface of the material (the surface that does not come in direct contact with the skin); (iii) the radial spreading toward the outer surface of the textile sample. The MMT instrument is conceived so as to be able to measure and record the behavior of a liquid transport in these three directions. The cellulose samples subjected to experimental tests were cut into 8 x 8 cm squares and steam ironed (covered with another fabric) to remove any wrinkles appeared on the textile samples. Then, the samples were conditioned for 48 hours under standard atmospheric conditions. Two measurements were performed for each sample and then the arithmetic mean was calculated. The time for pumping the test solution was of 20 seconds, while the total measurement time was of 120 seconds. At the end of the measuring time, the special MMT system 4.0 software automatically stopped the test, delivering the indices necessary for the experiment.

Five knitted cotton fabric samples were subjected to experimental investigation, numbered according to Table 1.

Table 1
Variants of cellulosic fabrics for experimental investigation

Nr	Indication of cellulosic fabric variant
I	Cellulosic fabric with alkaline boiling and bleaching stages (control)
II	Cellulosic fabric with alkaline boiling, bleaching and grafted with MCT-β-CD
III	Cellulosic fabric with alkaline boiling, bleaching, grafted with MCT-β-CD and treated with VTH
IV	Cellulosic fabric with alkaline boiling, bleaching, grafted with MCT-β-CD and treated with Ml
V	Cellulosic fabric with alkaline boiling, bleaching, grafted with MCT-β-CD and treated with Pr

RESULTS AND DISCUSSION

Elemental analysis

The content of nitrogen (in percent) present in the structure of the cyclodextrin product grafted on the textile support was established through elemental analysis. The cellulosic fabric that underwent alkaline boiling and bleaching stages (sample I) was investigated and compared with the knitted cellulosic fabric that was subjected to alkaline boiling, bleaching and grafting with MCT-β-CD (sample II). Two measurements were performed for each sample and the mean value was calculated. The total nitrogen content for sample II was 0.57%, as compared to sample I, where the nitrogen content was 0%. Taking these into account, the nitrogen presence in the grafted sample shows that the cyclodextrin grafting operation on the textile material was successful.

FT-IR analysis

The fact that a certain inclusion compound was obtained was confirmed by comparing the FTIR spectra recorded for: the cellulose support subjected to alkaline and bleaching treatments (I), for the grafted sample (II) and for the samples grafted with the reactive product and treated with AP (III, IV and V).

The presence of the bands characteristic of the host species (MCT-β-CD) or guest species (AP) in the spectrum of the samples treated with AP, which are usually shifted with respect to the bands of the non-grafted and grafted samples, can be considered as a proof of the formation of the inclusion compound. The reciprocal is not always valid, since the absence of a band specific to the guest molecule does not necessarily mean that the corresponding inclusion compound was not formed. Sometimes, the host and the guest bands can overlap.[25]

Figure 1 shows the absorption spectra obtained within the range 1650-550 cm^{-1} for the cellulose supports, according to the I ÷ V variants. Figure 2 presents the absorption spectra for the control ungrafted cellulose support (I) and for MCT-β-CD grafted cellulose support (II). The IR absorption bands characteristic of the MCT-β-CD grafted knitting are: 1570 cm^{-1}, 1431 cm^{-1}, 876 cm^{-1} (characteristic of the triazinyl band). Due to the reaction between MCT-β-CD and cellulose, the chlorine atom was replaced by the O-Cel group. The presence of the absorption bands shows that the knitted sample was indeed grafted with the MCT-β-CD.

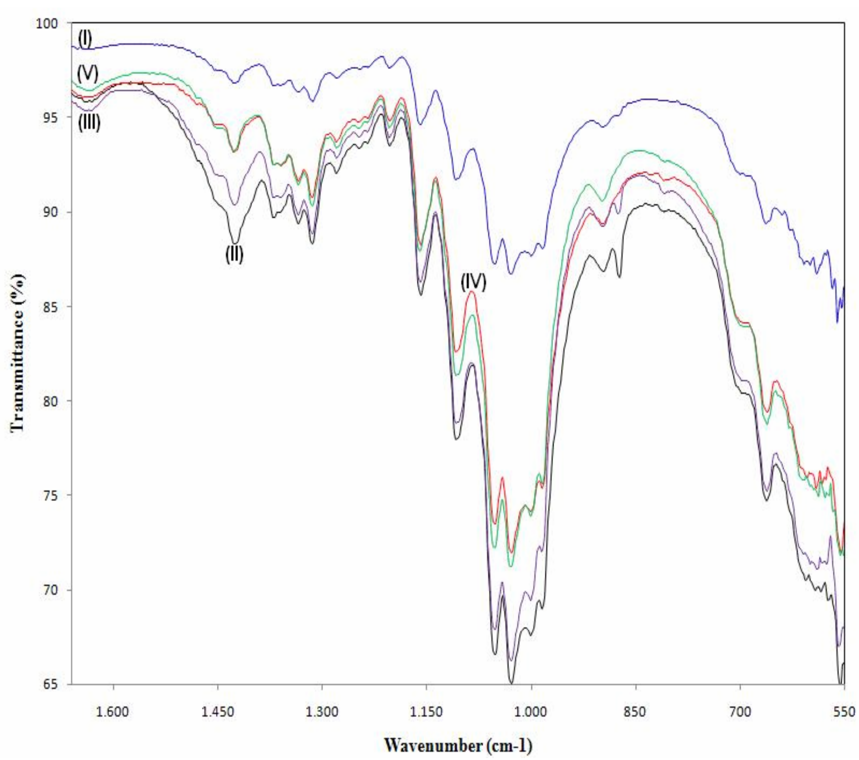

Figure 1: FTIR spectra of I, II, III, IV, V variants of cellulosic samples

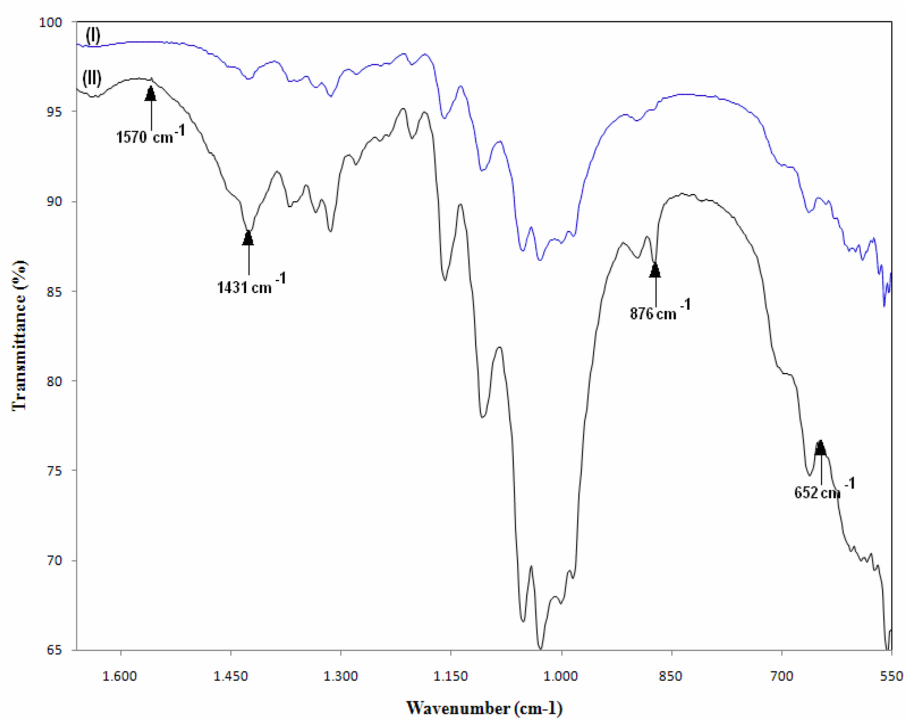

Figure 2: FTIR spectra of I and II variants of cellulosic samples

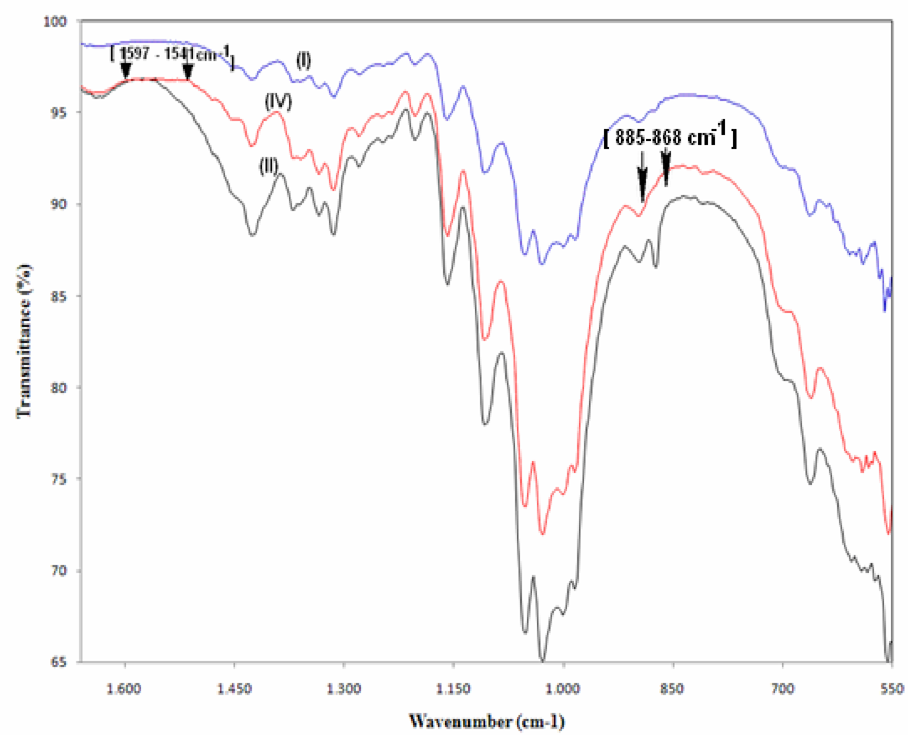

Figure 3: FTIR spectra of I, II and IV variants of cellulosic samples

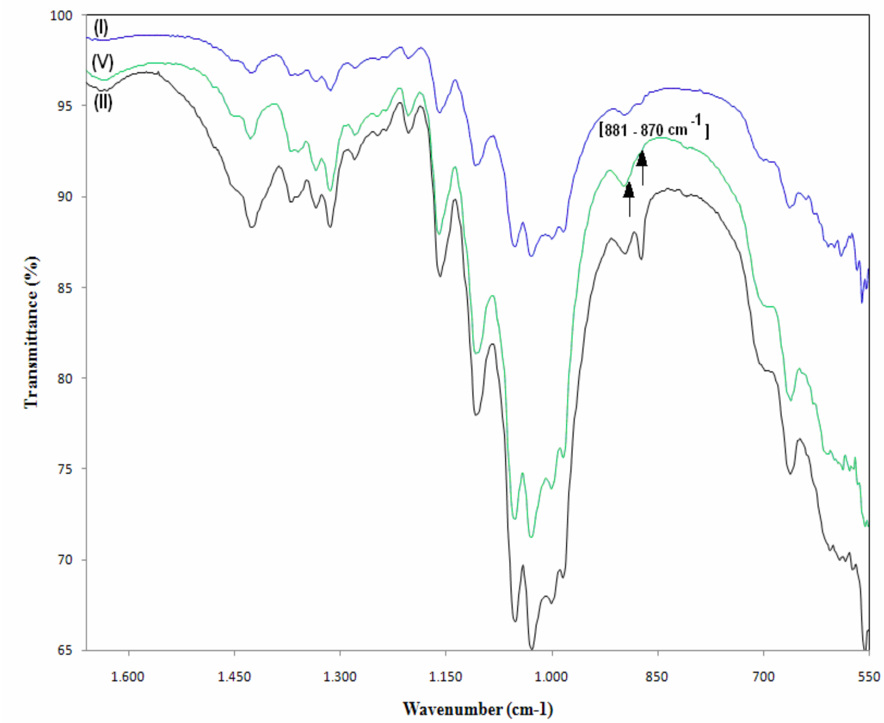

Figure 4: FTIR spectra of I, II and V variants of cellulosic samples

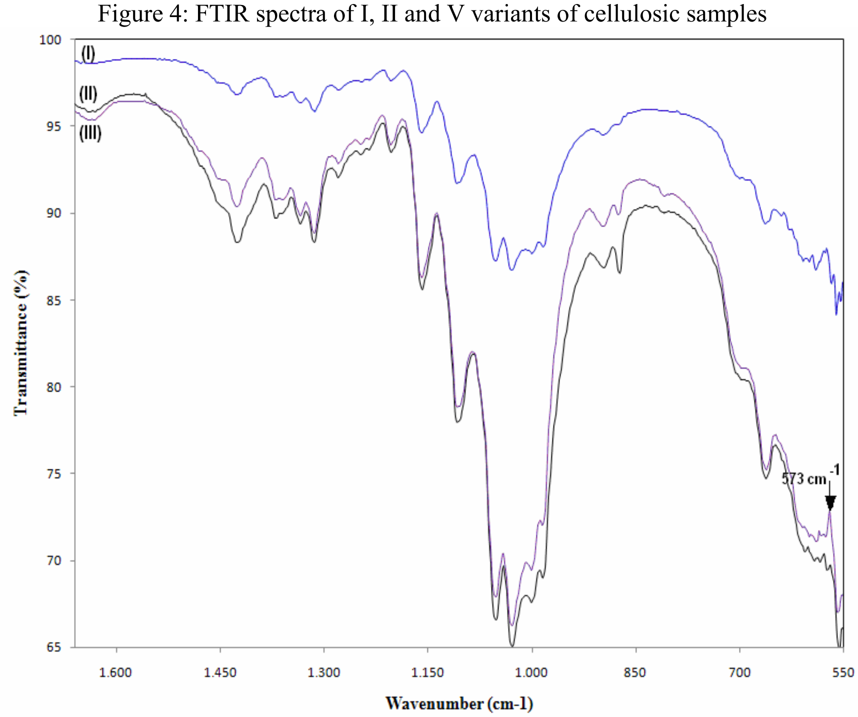

Figure 5: FTIR spectra of I, II and III variants of cellulosic samples

The spectrum of the MCT-β-CD grafted cellulose sample and treated with Ml is almost similar to the spectrum of the grafted cellulose sample (Fig. 3). The small shifts characteristic of the C-H and O-H bonds (existing in the menthol structure) appear within the range 1597-1541 cm^{-1}. Shifts characteristic of the C-C and C-O groups can also be noticed within the range 885-868 cm^{-1}. Generally, the significant peaks of the O-H group characteristic of menthol appear within the range 3500-3200 cm^{-1}.[26]

For the cellulose substrate treated with Pr, small shifts specific to the C-H bond (present in the structure of flavonoids entering the propolis composition) are rendered evident within the range 881-870 cm^{-1} (Fig. 4). For the sample grafted and treated with VTH, one can notice modifications in the absorption spectrum at 573 cm^{-1} (Fig. 5).

Scanning Electron Microscopy analysis

According to the SEM analysis, one can notice from Figure 6 that there is no micro-deposit on the surface of the ungrafted cellulose support, as compared to the surface of the grafted cellulose support.

From Figure 7, the presence of micro-deposits (spherical) is obvious on the surface of the cellulose support, due to MCT-β-CD grafting. One can notice that after fixation treatment at 160 °C during the grafting operation, the fibrils were not thermally degraded, preserving their shape and size.

It is worth noting that the surfaces of the cellulose supports grafted and treated with the three APs appear to be much cleaner (Figs. 8-10). The possible explanation may consist in the process of complexing the APs, which were successfully included in the cyclodextrin cavity. At the same time, one can notice that the cellulose supports treated with MI and Pr, used as alcohol solutions, are the cleanest, due to ethanol, which can clean the cellulose surface.

Moisture management tester

Beside the pharmacological action of the AP, the textile materials with anti-allergic properties also need to offer a high comfort at the cutaneous level. Therefore, it is necessary to evaluate the capacity to transfer liquids (water or perspiration) from the epidermis to the environment in the case of the cellulose samples, treated or not with APs. The results on the liquid transport properties for the cellulose materials obtained after our experiments are briefly presented in Table 2.

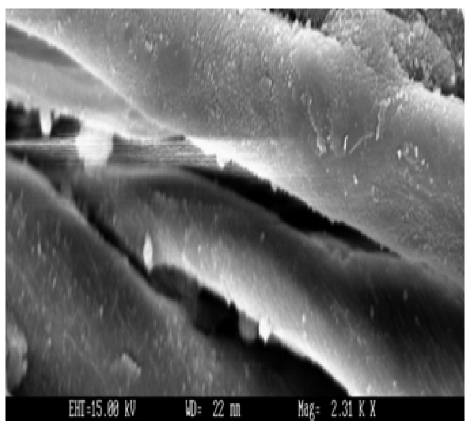

Figure 6: SEM image of ungrafted cellulosic support

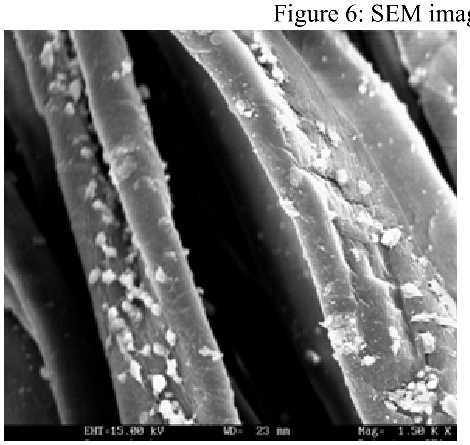

Figure 7: SEM image of grafted cellulosic support

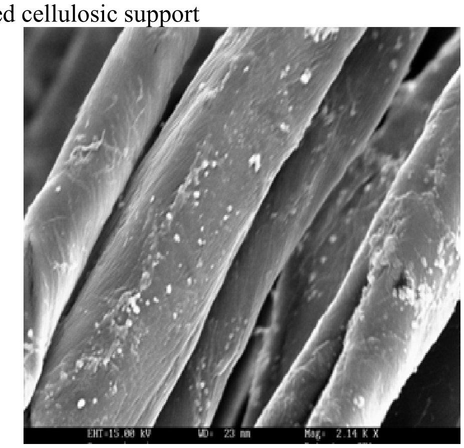

Figure 8: SEM image of grafted cellulosic support, treated with VTH

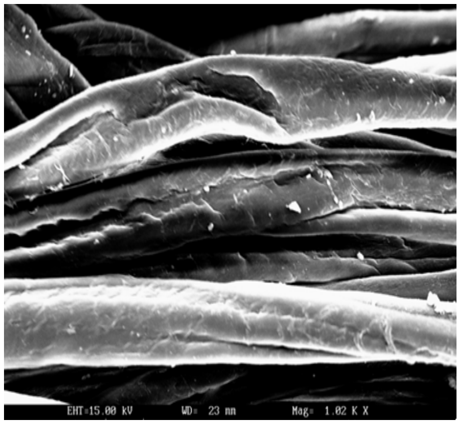

Figure 9: SEM image of grafted cellulosic support, treated with Ml

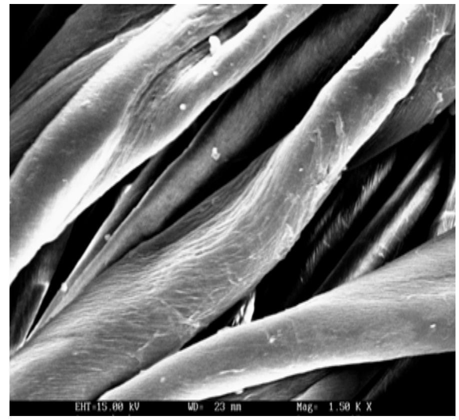

Figure 10: SEM image of grafted cellulosic support, treated with Pr

Table 2
Moisture management properties of cellulosic samples untreated and treated with anti-allergic active principles

Sample nr	Moisture management properties of cellulosic samples											
	WT [s]		AR [%/s]		MWR [mm]		SS [mm/s]		R [%]		OMMC	
	Int.S.	Ext.S.	Int.S.	Ext.S.	Int.S.	Ext.S.	Int.S.	Ext.S.	Int.S.	Ext.S.	Int.S.	Ext.S.
I	2.719	3.094	53.4436	40.7573	25	25	5.2508	4.5473	778.7609		0.8354	
II	2.8595	2.719	42.3936	35.9953	25	25	5.2289	4.8573	898.8011		0.8222	
III	2.859	2.8125	45.6179	39.3627	25	17.5	5.2434	3.8521	856.3655		0.7983	
IV	7.3125	3.422	65.2033	102.8266	20	17.5	2.7485	4.7444	864.3737		0.911	
V	120	1.2655	0	100.8481	0	15	0	8.1273	2220.0371		0.8934	

where: Int.S. – interior surface, represents the side of the fabric that would come in direct contact with the skin; Ext.S. – exterior surface, the side of the fabric that would be the outer surface when the piece of clothing is worn; WT – wetting time, the period time in seconds during the wetting process from outer to inner surfaces of the specimen after the test is strarted; AR – absorption rate, the average speed of liquid moisture absorption for the interior and exterior surfaces of the sample during the initial change of water content during period of testing; MWR – maximum wetted radius, the greatest ring radius measured on the exterior and interior surfaces; SS – spreading speed, the accumulated rate of surface wetting from the center of the specimen where the test solution is dropped to the maximum wetted radius; R – accumulative one-way transport capability, the difference between the area of the liquid moisture content curves of the exterior and interior surfaces of a specimen with respect to time; OMMC – overall liquid moisture management capability

The high liquid overall moisture management capacity (OMCC) and the high one-way transport capacity (R) show that liquid/sweat can be easily and quickly transferred from the skin to the outer surface of the cellulose fabrics.[27] From Table 2, one can notice that the sample treated with MCT-β-CD and Ml (IV) and the sample treated with MCT-β-CD and Pr (V) present the highest OMMC values (OMCC = 0.911 for sample IV, and OMCC = 0.8934 for sample V), while sample V also has the highest R value (R = 2220%).

The mean absorption rates (AR between 35-53%/s), spreading speeds (SS = 2-5.2 mm/s), maximum wetted radii (MWR = 17.5-25 mm) and fast wetting time (WT = 2.7-3.0 s) of cellulose fabrics I, II and III indicate that they are fast absorbing and slow drying fabrics.

It is worth noting that for the grafted knitted sample treated with propolis (V), the surface that comes in direct contact with skin (Int.S.) presents the lowest values of the absorption rate, which indicates that, under these testing conditions, the material has good properties of liquid transport from one zone (coetaneous level) to another (environment).[27] Therefore, the difference should be made between hydrophilic material, which has good properties of humidity sorption, and a material that facilitates the humidity transfer between two interfaces. In the latter case, the structure and the manner of the textile support realization (thickness, texture and yarn fineness) are important, while the material wettability is no longer necessary. It is compensated for by the hydrophobic character of propolis, which diminishes the liquid sorption capacity of the textile material.

CONCLUSION

For anti-allergic medical textiles, knitted fabrics are the most adequate, given their capacity to follow the body shape, their good flexibility and advanced permeability, as compared to woven or unwoven materials.

Among the textiles that satisfy the imposed medical requirements, we can name natural silk and cotton. We preferred cotton out of the two, since it is a low-cost fiber, with remarkable hygienic-physiological indices, good grafting capacity of a controlled release system and a good technological accessibility. Also, it is well-known that the use of wool or synthetic fibres may causes worsening of cutaneous diseases.

The studies carried out by elemental analysis, FTIR and SEM have proved that the cellulose supports used to obtain anti-allergic textile were grafted with the reactive monochlorotriazinyl-beta-cyclodextrin. At the same time, we have successfully managed to include the anti-allergic principles (VTH, Ml and Pr) in the cavity of the cyclodextrine product grafted on the cellulose support. Another obvious conclusion is that the SEM scanning method can have certain limitations, such as masking of the AP included in the cellulose fibers.

The knitted fabrics analyzed from the point of view of humidity diffusion (ungrafted, grafted, treated with VTH or treated with Ml) show quite high absorbance and slow drying, which causes a moisture sensation. The wet compresses have the role of hydration, cleaning, calming and anti-itchiness. Accordingly, the cellulose materials treated with Ml that also have a calming effect, can be beneficial in the treatment of AD. On the other side, the advanced wettability of the textile materials, such as cotton textiles, can help attenuate the skin dryness or atopic dermatitis.[28]

The decrease of wettability of fabric grafted and treated with Pr is correlated with the hydrophobic behaviour of Pr, which covers the surface of the cellulosic fabric, making it more hydrophobic. The moisture management method does not measure drying performance directly. Drying performance is inferred based on the area of liquid moisture spreading.[24] The fabrics with high moisture management performance keep the skin dry and provide the maximum comfort to the wearer.[29] In our case, the moisture management properties of the cellulosic fabrics are not critical in keeping the skin dry (because a higher wettability of the cellulosic fabrics could help manage dry or atopic skin).

The utilization of the three APs can be a solution in designing textiles with anti-allergic properties. Thus, the AP evaluation through *in vivo* experiments[21] confirmed that VTH shows the strongest anti-allergic action, followed by Ml and Pr. From the point of view of the comfort at the skin level, Ml can be the most indicated, due to its cooling character. Pr can offer the most significant antibacterial action, but for the patients who have an immunoglobulin level over 100 units (lgE > 100 units), it is not advisable to follow a treatment with Pr.[30]

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